

Laser powder bed fusion of M789 on N709 steel substrate: characterization of mechanical and microstructural properties

by

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Abstract

This study investigates the feasibility of employing laser powder bed fusion (LPBF) of a new commercial alloy, M789 steel, on a wrought N709 steel substrate, potentially enhancing the manufacturing of complex plastic injection moulds and die inserts with improved strength and reduced cycle times. Utilizing an EOS M290 LPBF machine at voestalpine Additive Manufacturing Centre (vAMC) in Mississauga, Ontario, hybrid samples were printed and subsequently subjected to direct aging treatment. This treatment facilitated dislocation annihilation and the formation of the η -phase ($\text{Ni}_3(\text{Ti},\text{Al})$) in the M789 section. The electron backscatter diffraction (EBSD) technique revealed random orientations of martensitic grains (in both as-printed and heat-treated states) with traces of reverted austenite that formed after heat treatment. Nanoindentation results (hardness profile across the interface) showed that the post-processing treatment increased the mechanical properties of M789 steel with hardness values comparable to wrought N709 steel. This assessment was validated through tensile testing, where the as-printed material failed in the M789 section, while the heat-treated sample fractured in the N709 section. Failure was detected away from the interface in both conditions, indicating the formation of a strong metallurgical bond.

To further understand the properties of the interface between M789 and N709 steels, thermodynamic and kinetic simulations were performed after the aging treatment, and the results revealed that different sizes and amounts of η - $\text{Ni}_3(\text{Ti},\text{Al})$ strengthening precipitates exist at the interface, with a maximum volume fraction and radius of about 5% and 5 nm, respectively (near the M789 region). Moreover, the volume fraction of precipitates significantly increases with Ti additions of up to 0.6 wt.% (i.e., 100 μm from the N709

region); however, beyond this threshold, the increase in the amount of precipitation became gradual.

Finally, since M789 steel is a relatively new commercial alloy in the additive manufacturing community, it has been characterized further to determine its high temperature mechanical properties utilizing a Gleeble 563 thermomechanical simulator. By employing the experimental high temperature flow curves, the application of Sellars and Tegart hyperbolic sine model, the Kocks-Mecking model and the Johnson-Mehl-Avrami-Kolmogorov equation facilitated the development of physical material models that accurately depicted the hot deformation behavior of LPBF-fabricated M789 steel. Traditional models, such as the modified Johnson-Cook model, were found inadequate to capture the complexities and metallurgical phenomena associated with high-temperature deformation. The microstructural analysis revealed that dynamic recrystallization (DRX) occurs more readily at higher strain rates and deformation temperatures, whereas at lower temperatures, a lower strain rate was preferred to generate a higher fraction of DRX grains.

Dedication

Dedicated to my family.

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1. Introduction

1.1 Background

Advanced manufacturing techniques, notably additive manufacturing (AM), have revolutionized the metal production industry over the past several decades [1–3]. This transformative technology offers inherent advantages, such as the ability to produce sophisticated and specialized components with shortened lead times, albeit typically in relatively low quantities [4]. The adoption of AM has been primarily driven by the modern manufacturing demands for better production efficiency, minimized energy consumption and lowered carbon footprints [5]. From one of the earliest reports of AM use in metallic material [6,7], the advancement of this technology has paved further innovation in designing high performance metals and alloys that are suited for the tool, automotive and aerospace industries [3,8]. Of particular interest to this work is the application of laser powder bed fusion (LPBF) AM technology in the hybrid production of a novel M789 maraging steel alloy with mechanical and corrosion properties similar to M250 and PH17-4, respectively [9,10]. The LPBF process involves a powder layering approach with intermittent laser fusion that produces highly dense near net shape components [11]. Its use in the production of maraging steels has facilitated the development of complex and topologically functional geometries such as those in die inserts, and injection moulds with conformal cooling channels that reduce mould cycle times [12]. In comparison with conventional/traditional manufacturing techniques, LPBF supports the development of conformal cooling channels in maraging steels that can be positioned near a tool's surface profile, enabling efficient melt cooling with minimized internal stresses [13,14].

Maraging steels, a commonly used alloy for AM processes, have excellent weldability due to their low carbon content. Moreover, high-strength properties can be achieved by means of post-processing aging [1]. Aging is critical in influencing the formation of strengthening precipitates within maraging steels, and this is achieved by the addition of alloying elements which form strong (Ti, Be), moderate (Mn, Si, V, Al, Ta), and weak (Cu, Co, Zr) intermetallics [12,15]. Recent research efforts have confirmed great potential to produce maraging steels with microstructure and mechanical properties comparable to or superior to conventionally processed steels [16,17].

Despite the material and process efficiency of LPBF, certain drawbacks limit its large-scale adoption compared to conventional techniques, particularly the high production costs and the relatively low building rate and build size capacities [18,19]. Furthermore, poor surface finishes that exhibit rough and stair-cased surfaces have been reported [20], often requiring post-processing to rectify. Non-uniform thermal deformations often result in localized residual stresses during cooling, resulting in part dimensional distortions [20]. These mechanical constraints limit a part's ability to meet structural design fittings and ultimately lead to premature failure in service conditions [8,21]. Other shortcomings in LPBF production are the lack of fusion and keyhole porosities, which act as stress concentrators and precursors to premature failure [22]. Other metallurgical defects, such as elemental loss and segregation, in conjunction with corrosive service conditions, have also been known to play a significant role in lowering the fatigue endurance of service components [8,23].

As highlighted, AM processes have the potential for improvement from a processing and cost-of-production standpoint. The current status quo is not sustainable for the tool and die industries' needs, which often require large cost-effective build sizes for plastic injection moulds and die inserts. A proposed way to achieve a big build stock at a possibly lowered cost is through hybrid AM [24]. The functional upper cavities of tooling components (conformal cooling channels) can be fabricated by the LPBF process on top of a conventionally made base material, which is cast or wrought. The hybrid/dual metal has the potential to meet the needs of industry by providing more efficient functionality with mechanical and physical properties that are derived from the joined metals [12]. Inevitably, complexities arise in these hybrid solutions as a result of complex thermal histories, which involve directional heat extraction, varying coefficients of thermal expansion (CTE), repeated melting, and fast localized solidification at the interface [14].

1.2 Research Statement and Objectives

Laser powder bed fusion is currently a costly process compared to conventional processing techniques (casting, forging, rolling, etc.), and its application is limited to high-end components and small build volumes of less than 0.064 m^3 [5]. The system productivity rate of the LPBF process is estimated to be lower than 70g/h, while that of conventional methods is estimated to exceed 100kg/h [25]. As such, an attempt to print bigger builds has high overhead costs that limit LPBF application in mass production operations. For tooling applications, the slow and expensive LPBF process is a hindrance to creating large-volume builds. Thus, the goal of this research is to study the feasibility of combining LPBF technology with conventional processes, employing the newly developed M789 maraging steel, designed and developed by voestalpine Additive Manufacturing Centre, and

conventional wrought N709 alloy. Furthermore, it is in the interest of this work to understand the microstructural and mechanical properties of M789 steel in this hybrid application employing optimized process conditions. Thus, the objectives of the present study are as follows:

- To fabricate the M789-N709 hybrid alloy by printing M789 steel powders on top of wrought N709 steel using the LPBF process.
- To assess the mechanical properties of the as-printed and heat-treated M789-N709 hybrid alloy, including its microstructural evolution during heat treatment.
- To analyze and understand the mechanical and microstructural properties of the interface between the M789 and N709 steel.
- To establish material models associated with M789 steels to assist the manufacturers in modelling the mechanical behaviour of M789 steel.

1.3 Thesis structure

In the interest of this study, M789, a novel corrosion-resistant steel with strength properties comparable to W722 and M250, is investigated, particularly its feasibility to be joined with a 13-8 Cr-Mo-based alloy, N709 steel. The current research explores fabricating an LPBF-M789–wrought-N709 hybrid alloy by evaluating its microstructure, texture, and mechanical behaviour after printing and heat treatment. The findings of this work have the potential to validate and expand the use of this hybrid material to meet the evolving needs of the industry.

This thesis follows a manuscript-based structure and consists of 7 chapters. **Chapter 1** introduces the background, motivation, and objectives of the research. **Chapter 2** provides

an extensive literature review on the recent progress in hybrid additive manufacturing, encompassing the definition of hybrid additive manufacturing and detailed case studies of challenges associated with the interfaces of hybrid alloys, such as alloy compatibility, interface reaction products, and thermal conductivity mismatches. Additionally, the research gap in literature related to LPBF-M789-N709 hybrid steel is identified.

Chapter 3 focuses on the evaluation of the microstructure, texture and mechanical properties of hybrid M789/N709 steel with the application of a non-conventional heat treatment protocol. The strength of the interface is assessed, and key microstructure and mechanical properties are identified.

The precipitation behaviour at the interface of M789/N709 hybrid alloy, as well as the strengthening mechanism, is assessed in **Chapter 4** with an in-depth analysis of the effects of dilution at the interface. **Chapter 5** presents the high temperature behaviour of LPBF-M789 steel, focusing on its dynamic recrystallization behaviour and the prediction of its hot deformation characteristics using physical-based models. A method is developed for accurate data input before developing the material models. Moreover, an innovative quantitative analysis combining parent grain reconstruction and shape factor measurements is presented to identify the occurrence of dynamic recrystallization. The observed dynamic recrystallization phenomenon is then correlated with material physical modelling, facilitating a comprehensive understanding of microstructural evolution. Finally, a summary of the important findings of this work as well as the contributions to original knowledge are given in **Chapters 6** and **7**, respectively.

1.4 References

- [1] C. Turk, H. Zunko, C. Aumayr, H. Leitner, and M. Kapp, ‘Advances in Maraging Steels for Additive Manufacturing’, BHM Berg- Hüttenmänn. Monatshefte, vol. 164, no. 3, pp. 112–116, Mar. 2019, doi: 10.1007/s00501-019-0835-z.
- [2] J. Pasco, Z. Lei, and C. Aranas, ‘Additive Manufacturing in Off-Site Construction: Review and Future Directions’, Buildings, vol. 12, no. 1, p. 53, Jan. 2022, doi: 10.3390/buildings12010053.
- [3] S. Cooke, ‘Metal additive manufacturing Technology, metallurgy and modelling’, J. Manuf. Process., 2020.
- [4] P. Bajaj, A. Hariharan, A. Kini, P. Kürnsteiner, D. Raabe, and E. A. Jägle, ‘Steels in additive manufacturing: A review of their microstructure and properties’, Mater. Sci. Eng. A, vol. 772, p. 138633, Jan. 2020, doi: 10.1016/j.msea.2019.138633.
- [5] B. Blakey-Milner, ‘Metal additive manufacturing in aerospace: A review’, p. 33, 2021.
- [6] R. Merz, F. B. Prinz, K. Ramaswami, M. Turk, and L. E. Weiss, ‘Shape Deposition Manufacturing’, 1994.
- [7] M. Cima and J. Cornie, ‘Three-Dimensional Printing: Rapid Tooling and Prototypes Directly from a CAD Model’, 1990.
- [8] T. DebRoy et al., ‘Additive manufacturing of metallic components – Process, structure and properties’, Prog. Mater. Sci., vol. 92, pp. 112–224, Mar. 2018, doi: 10.1016/j.pmatsci.2017.10.001.

- [9] Y. Tian, R. Palad, and C. Aranas, ‘Microstructural evolution and mechanical properties of a newly designed steel fabricated by laser powder bed fusion’, *Addit. Manuf.*, vol. 36, p. 101495, Dec. 2020, doi: 10.1016/j.addma.2020.101495.
- [10] Z. Brytan et al., ‘Microstructural and Mechanical Properties of Novel Co-Free Maraging Steel M789 Prepared by Additive Manufacturing’, *Materials*, vol. 15, no. 5, p. 1734, Feb. 2022, doi: 10.3390/ma15051734.
- [11] F. Klocke, K. Arntz, M. Teli, K. Winands, M. Wegener, and S. Oliari, ‘State-of-the-art Laser Additive Manufacturing for Hot-work Tool Steels’, *Procedia CIRP*, vol. 63, pp. 58–63, 2017, doi: 10.1016/j.procir.2017.03.073.
- [12] A. Kirchheim, Y. Katrodiya, L. Zumofen, F. Ehrig, and C. Wick, ‘Dynamic conformal cooling improves injection moulding: Hybrid moulds manufactured by laser powder bed fusion’, *Int. J. Adv. Manuf. Technol.*, vol. 114, no. 1–2, pp. 107–116, May 2021, doi: 10.1007/s00170-021-06794-0.
- [13] K. Gupta, N. K. Jain, and R. F. Laubscher, ‘Overview of Hybrid Machining Processes’, in *Hybrid Machining Processes: Perspectives on Machining and Finishing*, K. Gupta, N. K. Jain, and R. F. Laubscher, Eds., in *SpringerBriefs in Applied Sciences and Technology*, Cham: Springer International Publishing, 2016, pp. 1–7. doi: 10.1007/978-3-319-25922-2_1.
- [14] A. Seidel et al., ‘Added value by hybrid additive manufacturing and advanced manufacturing approaches’, *J. Laser Appl.*, vol. 30, no. 3, p. 032301, Aug. 2018, doi: 10.2351/1.5040632.

- [15] R. E. Smallman and A. H. W. Ngan, Physical metallurgy and advanced materials., 7th ed. /., 1 online resource (xxi, 650 pages): illustrations vols. Amsterdam; Butterworth Heinemann, 2007. [Online]. Available: <http://www.books24x7.com/marc.asp?bookid=37245>
- [16] Y. Tian, K. Nyamuchiwa, K. Chadha, Y. He, and C. Aranas, ‘Laser powder bed fusion of M789 maraging steel on Cr–Mo N709 steel: Microstructure, texture, and mechanical properties’, Mater. Sci. Eng. A, vol. 839, p. 142827, Apr. 2022, doi: 10.1016/j.msea.2022.142827.
- [17] J. Samei et al., ‘A hybrid additively manufactured martensitic-maraging stainless steel with superior strength and corrosion resistance for plastic injection moulding dies’, Addit. Manuf., vol. 45, p. 102068, Sep. 2021, doi: 10.1016/j.addma.2021.102068.
- [18] K. S. Prakash, T. Nancharaih, and V. V. S. Rao, ‘Additive Manufacturing Techniques in Manufacturing -An Overview’, Mater. Today Proc., vol. 5, no. 2, pp. 3873–3882, 2018, doi: 10.1016/j.matpr.2017.11.642.
- [19] V. V. Popov and A. Fleisher, ‘Hybrid additive manufacturing of steels and alloys’, Manuf. Rev., vol. 7, p. 6, 2020, doi: 10.1051/mfreview/2020005.
- [20] S. Jayanath and A. Achuthan, ‘A computationally efficient hybrid model for simulating the additive manufacturing process of metals’, Int. J. Mech. Sci., vol. 160, pp. 255–269, Sep. 2019, doi: 10.1016/j.ijmecsci.2019.06.007.
- [21] A. Yadollahi and N. Shamsaei, ‘Additive manufacturing of fatigue resistant materials: Challenges and opportunities’, Int. J. Fatigue, vol. 98, pp. 14–31, May 2017, doi: 10.1016/j.ijfatigue.2017.01.001.

- [22] K. Darvish, Z. W. Chen, and T. Pasang, ‘Reducing lack of fusion during selective laser melting of CoCrMo alloy: Effect of laser power on geometrical features of tracks’, *Mater. Des.*, vol. 112, pp. 357–366, Dec. 2016, doi: 10.1016/j.matdes.2016.09.086.
- [23] A. J. Sterling, B. Torries, N. Shamsaei, S. M. Thompson, and D. W. Seely, ‘Fatigue behaviour and failure mechanisms of direct laser deposited Ti–6Al–4V’, *Mater. Sci. Eng. A*, vol. 655, pp. 100–112, Feb. 2016, doi: 10.1016/j.msea.2015.12.026.
- [24] H. Azizi et al., ‘Metallurgical and mechanical assessment of hybrid additively-manufactured maraging tool steels via selective laser melting’, *Addit. Manuf.*, vol. 27, pp. 389–397, May 2019, doi: 10.1016/j.addma.2019.03.025.
- [25] M. Baumann, P. Dickens, C. Tuck, and R. Hague, ‘The cost of additive manufacturing: machine productivity, economies of scale and technology-push’, *Technol. Forecast. Soc. Change*, vol. 102, pp. 193–201, Jan. 2016, doi: 10.1016/j.techfore.2015.02.015.

2. Recent Progress in Hybrid Additive Manufacturing of Metallic Materials

This chapter presents a comprehensive literature review focusing on the progress of hybrid additive manufacturing of maraging steels, with a particular emphasis on the challenges associated with achieving robust metallurgical bonds at the interface.

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*Kudakwashe Nyamuchiwa led the investigation of this review and bore primary responsibility for this article. Co-authors contributed to reviewing and finalizing the manuscript.

2.1 Abstract

Additive Manufacturing (AM) is an advanced technology that has been primarily driven by the demand for production efficiency, minimized energy consumption, and reduced carbon footprints. This process involves layer-by-layer material deposition based on a Computer-Aided Design (CAD) model. Compared to traditional manufacturing methods, AM has enabled the development of complex and topologically functional geometries for various service parts in record time. However, there are limitations to mass production, the building rate, the build size, and the surface quality when using metal additive manufacturing. To overcome these limitations, the combination of additive manufacturing with traditional techniques such as milling and casting holds the potential to provide novel manufacturing solutions, enabling mass production, improved geometrical features, enhanced accuracy, and damage repair through net-shape construction. This amalgamation is commonly referred to as hybrid manufacturing or multi-material additive manufacturing. This review paper aimed to explore the processes and complexities in hybrid materials, and joining techniques, with a focus on maraging steels. The discussion is based on existing literature and focuses on three distinct joining methods: direct joining, gradient path joining, and intermediate section joining. Additionally, current challenges for the development of the ideal heat treatment for hybrid metals are discussed, and prospects of hybrid additive manufacturing are also covered.

2.2 Introduction

Additive Manufacturing (AM), an advanced technology, has gained prominence due to its focus on enhancing production efficiency by reducing energy consumption and minimizing carbon footprints [1,2]. This process utilizes layer-by-layer material deposition in

conjunction with a Computer-Aided Design (CAD) model to create 3D objects. Additive manufacturing has been applied in the fabrication of polymeric, ceramic, composite, and metallic materials [3]. Multiple techniques have been developed for AM [4,5], as shown in **Fig. 2.1**. Vat photo-polymerization, material extrusion, and material jetting are mainly used for producing polymer-based products with UV-curable polymers, thermoplastic polymers, and jetted polymeric material. Techniques labelled M (in red) in **Fig. 2.1** are used for Metal Additive Manufacturing (M-AM) or metal printing. In contrast to traditional manufacturing methods, M-AM has enabled the rapid development of intricate and functionally diverse geometries in metallic components used in various service parts [6,7]. In M-AM processes, various material building approaches are employed. For instance, in the sheet lamination process, metal sheets are accurately cut and then bonded together using metallurgical techniques such as laser welding, diffusion bonding, and resistance welding to create a three-dimensional product. In binder jetting, a binder deposited onto the metal powder consolidates, sinters, or infiltrates another metal into the printed product. Techniques such as Direct Energy Deposition (DED) and Laser Powder Bed Fusion (LPBF) utilize a focused energy source, such as high-energy electrons or laser, to precisely sinter or melt a layer of powders. Among these techniques, LPBF is commonly preferred in the AM community in the production of nickel, titanium, copper-based alloys, and maraging steel components because of its smoother surface finish compared to DED. Moreover, the relative density of parts fabricated by means of LPBF is comparable to that of conventionally produced parts [8].



Figure 2.1. The classification of additive manufacturing technologies [5,9].

Metal additive manufacturing processes offer design freedom, which is limited in conventional processes [3]. Complex designs such as topologically optimized cooling channels and consolidated multiple components enable reduced lead and cycle times. Consolidated multiple components refers to the merging of multiple components into one build system and enables the removal of joining surfaces and structures. Consolidating multiple components in a single build has the potential to reduce fabrication and joining costs. In addition, the design freedom offered by AM allows for optimized material distribution and lightweighting whilst maintaining the minimum mechanical requirements and performance requirements [10]. Metal AM also allows the efficient repair of worn-out parts and saving inventory storage space. Parts can be prepared as per the need or in situ, allowing for an extended component life and efficient servicing of heavy and expensive components such as turbine blades [11].

Despite the novelty of M-AM processes, they have limitations when compared to conventional techniques, specifically in terms of mass production, building rate, build size,

and surface quality, as highlighted by Prakash et al. [12] and Popov et al. [13]. Service parts produced by the M-AM and surface quality, as highlighted by Prakash et al. [12] and Popov et al. [13]. Service parts produced by the M-AM process often have rough and stair-cased surface finishes, in addition to internal residual stresses and part distortion. Jayanath et al. [14] reported that non-uniform thermal deformations cause localized stresses in M-AM products, resulting in non-uniform elastic distortions during cooling to room temperature. These mechanical constraints limit a part's ability to meet structural design fittings, leading to premature failure during fatigue loading due to accumulating and propagating dislocations [15,16]. The presence of lack of fusion defects was found by Darvish et al. [17] to negatively impact the fatigue life of service components. Sterling et al. [18] and Johnson et al. [19] performed studies on Ti-6Al-4V and IN718 using the DED-Laser process, and both affirmed that metallurgical defects act as stress concentrators, which lower fatigue endurance. For the LPBF process, internal pore defects are categorized into Lack of Fusion (LOF) and gas defects. Lack of fusion defects are often found in the vicinity of spatter particles in between the laser track and powder layers. This is associated with insufficient volumetric energy density and poor powder spreading [20,21]. On the other hand, gas pores include keyhole pores, which are caused by instable melt pools, powder feedstock, and hydrogen pores, which arise from trapped gas and precipitating hydrogen during processing, respectively [21]. Moreover, studies by Zhao et al. [22] showed the importance of surface quality for mechanical properties. In situ laser polishing of the IN718 alloy produced by selective laser melting reduced the surface roughness by about 34%. Laser polishing has the capability of reducing the surface cracks of alloys, fuse surface pores, and melt surface residual powder. The scalability and mass production of

these M-AM continue to be a challenge, and these limitations for M-AM pose significant drawbacks in its adoption application and expansion.

Combining traditional manufacturing techniques (milling, casting, etc.) and M-AM could potentially provide new manufacturing solutions relevant to mass production, improved geometrical collaboration, accuracy, and damage repair using net-shape construction [4,13]. From this point forward, the fusion of traditional manufacturing and M-AM will be referred to as hybrid manufacturing or Multimaterial Additive Manufacturing (MMAM) throughout the document. Hybrid manufacturing is relatively in its infancy, and its governing mechanisms are still subjects of great interest. The demand for multi-service superior alloys with multiple properties is rising to suit lightweight functionality, corrosive environments, high-temperature applications, and prolonged exposure to radiation [23]. Additive manufacturing shows great promise as an alternative to traditional joining techniques for different materials.

Inevitably, complexities arise in these hybrid solutions because of incompatibilities, which may arise due to complex thermal histories, which involve directional heat extraction, varying coefficients of thermal expansion (CTE), repeated melting, and fast localized solidification for the considered metals [7]. In addition, the mechanical behaviour of the interface is crucial to developing sound MMAM to avoid premature failure of such components. Components manufactured by metal joining processes are prone to premature failure due to the presence of joints and interfaces between different materials [24]. This behaviour can be attributed to the nature of the compositional gradient at the interface.

This review paper explored the types of processes and complexities involved in hybrid manufacturing encompassing joining techniques and combinations with an emphasis on

the utilization of AM as a potential joining method for dissimilar metals, in particular, maraging steels. Notably, there has been limited research on joining dissimilar materials, especially of this material type. The discussion will also cover other alloys including stainless steel, Cu-based alloys, and Ni-based steels. Subsequent sections will evaluate different hybrid techniques and their respective performances.

2.3 Hybrid Techniques Based on the Use of Other Manufacturing Techniques with AM

The term hybrid additive manufacturing is not limited to the use of dissimilar metals but also encompasses the use of other manufacturing technologies in conjunction with AM to achieve better performance and functionality. Despite the positive outcomes of integrating conventional manufacturing techniques with AM in superalloys and steels, hybrid techniques still have limited application in the industry. This is due to the challenges of adopting hybrid AM parameters for each specific material in the hybrid system to match the substrate or for specific geometries [13]. Earlier work by Manogharan et al. [25] explored the possibility of merging additive and subtractive techniques based on individual processes' limitations and capabilities.

This section focuses on hybrid manufacturing techniques, i.e., the use of AM in conjunction with other manufacturing technologies, in situ or post-AM. Note as well that processes such as Selected Laser Melting (SLM) and Selective Laser Sintering (SLS) according to ASTM F42 [26] refer to Powder Bed Fusion (PBF) processes. The names of the processes are given based on the way they were presented in the original manuscripts. Moreover, the discussion in the sections below is focused on the actual technique since an in-depth analysis (such as texture evaluation) of each method is currently not available.

2.3.1 Electron Beam Melting + Computer Numerically Controlled Rapid Prototyping

Additive System Integrated Subtractive (AIMS) systems are the combination of Electron Beam Melting (EBM) and Computer Numerically Controlled Rapid Prototyping (CNC-RP) (**Fig. 2.2**). The AIMS method is used for metallic components, such as the Ti-6Al-4V alloy, in which the tolerances and surface finishes are improved by digital machining post-EBM-AM. The comparative studies between the CNC-RP technique and the AIMS method revealed that the AIMS method is faster, with an estimated buy-to-fly ratio of less than 3. On the other hand, the CNC-RP process had a buy-to-fly ratio greater than 13. The buy-to-fly ratio is the measure of the efficiency of raw material use. It gives the weight ratio of the raw material to the final product [27].

The AIMS process has minimized tool wear and has better material utilization than the CNC-RP technique. However, the authors noted that the subtractive techniques require custom-made tools and fixtures, which can be a limitation in expanding this process [25]. The work conducted identified fixturing limitations when combining the two processes, necessitating the need for customized and more fixture designs tailored to different M-AM techniques.

2.3.2 Laser Metal Deposition + Computer Numerical Control

Merklein et al. [29] reviewed the Laser Metal Deposition (LMD) AM technique in conjunction with a subtractive process in a single-step procedure. The authors proposed a combination of LMD and CNC milling centres for the aerospace industry by using functional lightweight metals followed by a smooth surface finishing (**Fig. 2.3**). This technique provides a solution to maximize the production volume whilst eliminating

changeover cycles. The LMD process produces moderate thermal gradients and defect-free components. The possibility of the laser alloying of tool alloys was explored to reduce the abrasive and adhesive tool wear. According to the study, the chipping volume was significantly reduced, albeit this was not quantified. Similar to laser case hardening, the application of laser energy for heating hypothetically proves advantageous in surface hardening of components [29]. Laser metal deposition can facilitate the production of several materials in a single process, and the addition of ceramic inclusions on the surface of the metals was proposed to improve surface wear. In another study, the integration of a direct energy deposition technique with CNC machining was achieved in a process termed the “ArcHLM process”, proposed to be suited for LMD processes [30]. The uniqueness of this technique is that machining is performed after every successive layer of weld-deposited material. As a result of the process, oxide coatings are formed, leading to a rough surface finish. The surface irregularities were estimated to be less than 2 mm. Hence, to avoid a loss of accuracy along the build direction, each layer was face-milled to the required thickness. This study showed that fast deposition rates are achievable; however, there are issues such as the need for controlled heat gradients and atmosphere to minimize oxidation and phase stability. In the case of maraging steels, the cooling to martensite start is crucial to minimize the amount of retained and reverted austenite, which can increase the ductility at the expense of strength. Instead, the use of the SLM process in conjunction with CNC machining has been proposed to be a better alternative [20]. Small melt pools are produced, which allow for faster cooling and better surface quality. The process demonstrated significantly higher hardness values of about 56 HRC for the hybrid additive technique in comparison to 40 HRC for the material produced solely through printing without heat

treatment. This study showed the importance of the surface finish for the mechanical properties. The major setback of this technique is that the time interval between additive manufacturing and machining is not optimized, resulting in long lag times.

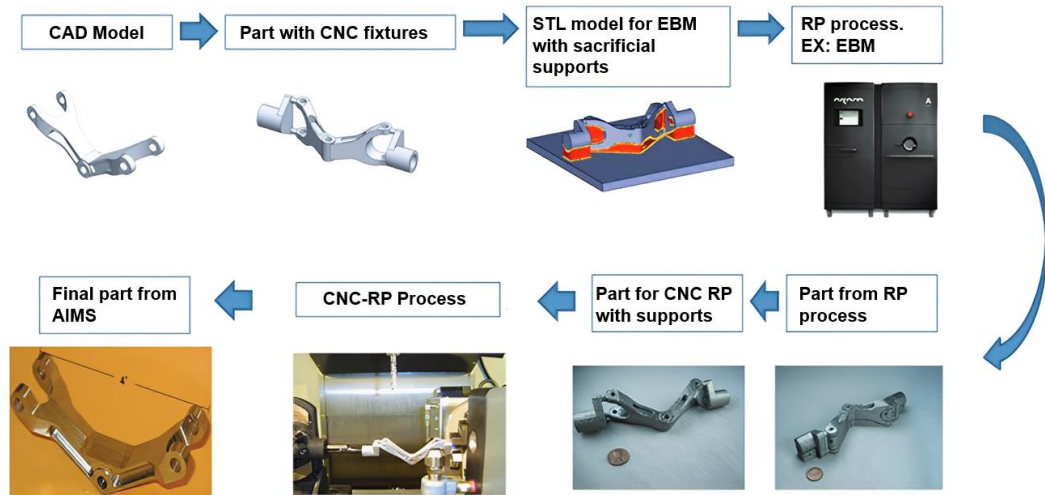


Figure 2.2. The classification of additive manufacturing technologies [28].

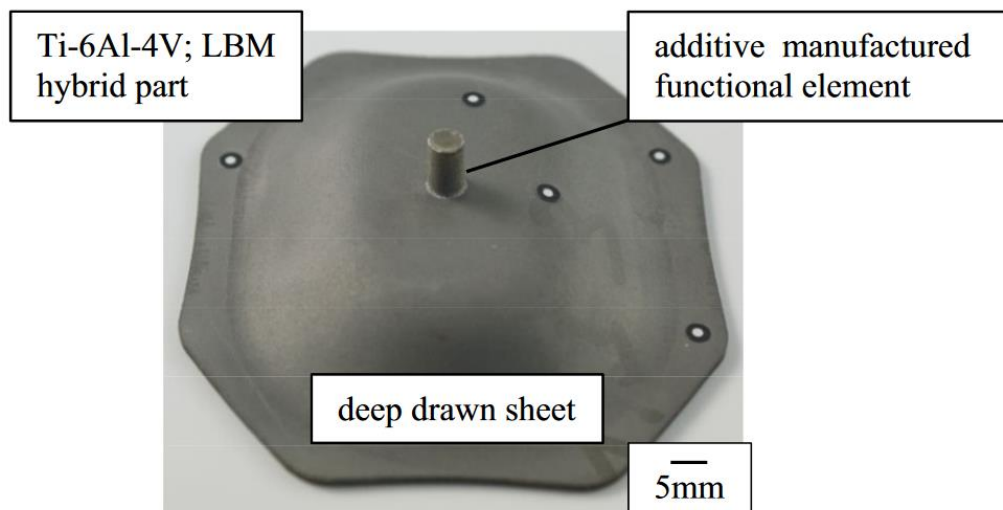


Figure 2.3. The classification of additive manufacturing technologies [29].

2.3.3 Selective Laser Sintering + Die Substrate

In another work by Oter et al. [31], a Selective Laser Sintering (SLS) technique was used to design a die matrix on an extruded hot steel die, and the resulting work produced a die that did not require post-processing. This work was motivated by the need to address the mismatch between simulation and production results due to human intervention in post-processing correction in conventionally produced extrusion dies, which results in the poor quality of the extruded profile. However, there was evidence of decarburization on one of the sections, which created a hardness variation from 381 to 451 HV [31]. The loss of carbon is detrimental to the performance of steel since carbon plays a crucial role in solid solution strengthening, which contributes to its overall strength and performance. Carbon migration is reported to cause sensitization in alloy systems that rely on high chromium contents for corrosion resistance [32].

Preferential deposition of chromium carbides along the grain boundaries increases the chances of localized stress corrosion, especially in austenitic and low-alloy-ferritic steels [33]. The distribution of other alloying elements and how they affect die strength were not explored in this work, potentially giving room for more work in understanding the behaviour of the alloying elements.

A study by Aziz et al. [34] explored the possibility of building C300 maraging steel (using MS1 powder) on top of an inexpensive conventional H-13 die structure using Selective Laser Melting (SLM). A sharp microstructural contrast was observed between the two alloys and the interface and was reported to be weak due to the microstructure and chemical composition variation at the interface. A low strain-to-fracture average of 0.27 was recorded and localized at the dissimilar joint, and porosity was observed at the interface.

Earlier work by Wang et al. [35] deposited 1Cr12Ni2WMoVNb martensitic steel on a wrought bar of a similar material using Laser Melting Deposition (LMD) to produce a sound metallurgical bond, and failure occurred in the wrought substrate zone [35]. In addition, Zhu et al. [36] had similar findings from a TC11 sample produced using LMD. The interface had sound metallurgical bonding, as evidenced by failure in the substrate material during tensile testing. These results showed that careful microstructure development is critical for good metallurgical bonding.

2.3.4 Wire and Arc Additive Manufacturing Filling + Worn-out Part

Wire Arc Additive Manufacturing (WAAM) was proposed as an innovative and cost-effective AM solution to repair worn-out parts [37]. This study revealed that the inclination angle between the groove on the worn-out surface and the deposited layer of H0.8Mn2Si was a critical parameter for efficient fusion. Each layer deposited in the cavity must satisfy a specific height to avoid incomplete fusion at the inclined interface, as shown in **Fig. 2.4**. An optimized height-to-width ratio of a single weld bead corresponded to $\tan\Theta = 1.377$, where Θ was the inclination angle used in this study. This inclination angle enabled free working space for the weld gun, and there was no evidence of weld porosity or crack formation. Despite the evidence of deformation on the repaired section, a buy-to-fly ratio of 0.921 and sufficient fabrication and material were achieved. The deformation was due to the shrinkage of the weld beads and the adjacent substrate metal during cooling [37]. These deformities could be hindrances for repair works that require strict geometrical fittings. As a result, this may ultimately lead to the high post-processing cost of the repaired parts.

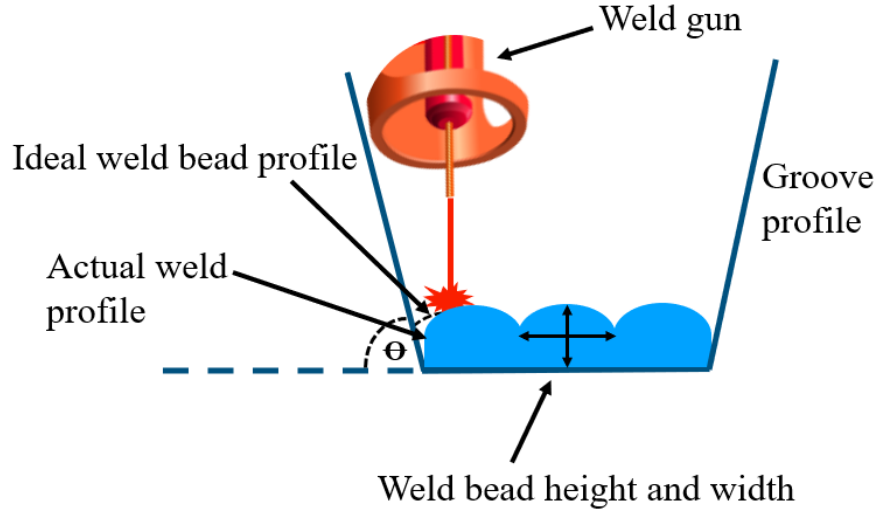


Figure 2.4. Lack of fusion at the interface due to mismatched inclination angle [37].

Earlier work on IN718 by Praniewicz et al. [38] investigated the possibility of model analysis on worn-out surfaces and optimized the AM-built weld. The basis of this work was to address the various distortions and worn-out geometries. Transformed CAD geometries were matched to the geometry of deformed samples using an iterative algorithm with a Coordinate Measurement Machine (CMM) for improved precision during adaptive repairs. This approach resulted in a maximum surface deviation of 0.1%. As a result, it was concluded that a material efficiency above 74% and efficient processing times are achievable [38].

2.3.5 Laser Powder Bed Fusion + Fused Filament Polymer

A process termed “AddJoining” by Chueh et al. [39] used Fused-Filament-Fabricated (FFF) polymer and a Laser-Based Powder Fusion (LBPF) process for dental applications [40]. Consequently, laser heating and remelting were performed on the interface to ensure the sound interface bonding strength for the different materials. From the observations made, the fracture stress in the polymer section was 22 MPa, and the interface remained

intact, indicating a sound polymer infiltration into the metal section and concluding that dissimilar material joining is possible with a thorough understanding of the structure–property relationships.

2.3.6 SLM + WAAM

Shi et al. [41] explored the feasibility of fusing the SLM and WAAM processes using a Ti-6Al-4V alloy. The SLM process has the advantage of generating fine surfaces and high-density components with good mechanical properties and geometrical flexibility. However, the process has high production costs associated with low building rates and small-scale production [42,43]. On the other hand, WAAM has a high deposition rate and production volume, but it has limited geometrical accuracy and poor surface quality. The synergy of these two processes was implemented to take advantage of each process's capability, simultaneously minimizing their constraints. The SLM-produced component was set as the substrate and was partitioned into two sections, namely the complicated and simple sections. The complicated section's fabrication was SLM processed, and the rest of the simple section was completed using WAAM. To prevent potential defect formation and maintain a high bonding strength at the interface, the SLM process was employed as the substrate-processing technique. This choice aimed to mitigate the risks associated with defect formation and ensure robust bonding at the interface. From **Fig. 2.5**, the joining of the AM processes has three zones, the SLM zone, interface zone, and WAAM zones, with an interface of about 3 mm. Each zone appears to have a distinct microstructure. The SLM zone primarily produced short β columnar grains consisting mainly of a martensitic structure. Subsequently, a complex interface with coarse grains caused by repeated heating facilitates grain growth. The martensite formation is most likely due to high cooling rates

from above the β -transit temperature of Ti-alloys. Furthermore, the progression into the WAAM zone revealed an α lamella with evident epitaxial growth. It was found that the WAAM build sections had inferior mechanical properties (yield strength: 814 MPa, tensile strength: 927 MPa, and elongation: 10.5%) compared to the SLM section (yield strength: 1020 MPa, tensile strength: 1098 MPa, and elongation: 10.2%).

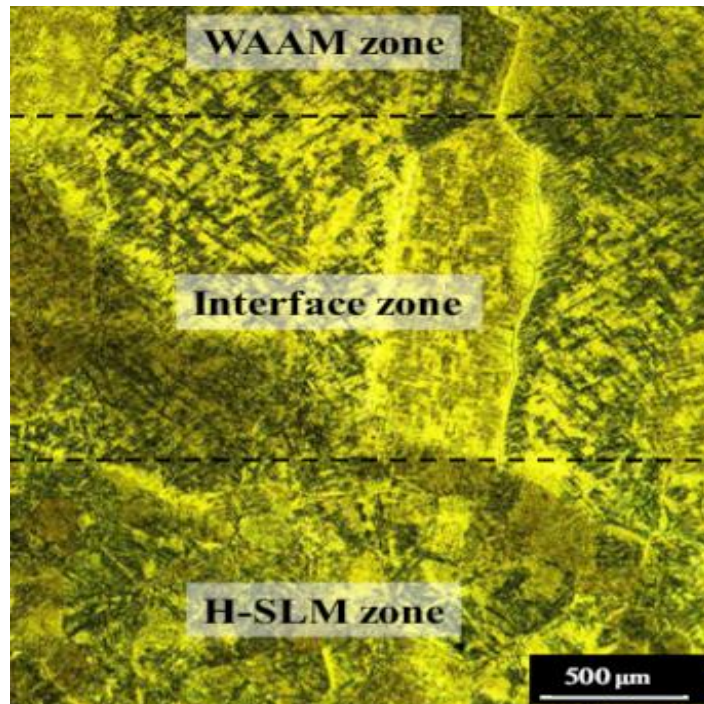


Figure 2.5. Different zones created by WAAM and SLM [41].

To confirm the effects of these varied mechanical properties, further testing was conducted on the hybrid parts parallel to the building direction, and failure was observed in the WAAM section with a yield strength of 890 MPa and a tensile strength of 905 MPa. Sections perpendicular to the build direction were evaluated to expand the applicability of this approach; however, it had low mechanical properties (yield strength: 850 MPa, tensile strength: 995 MPa). It was evident that the strength of the hybrid was higher compared to

the WAAM component, despite being lower than the SLM component. Thus, the advantages of the combined processes can be realized. In this work, a sound metallurgical bond was achieved with no identifiable defects; however, no heat treatment of the produced hybrid material was performed, which could have optimized the mechanical properties of the alloy [41].

2.3.7 Laser Metal Deposition + Turning + Milling

Yamazaki [44] developed a hybrid multi-tasking platform (Mazak Hybrid Multi-tasking), which incorporated turning and milling functionalities with an LMD process. The motivation of this study was to reduce the manufacturing and process times for part development for the offshore oil industry. In this work, IN718 was deposited onto a stainless-steel alloy (316S31) using the Mazak Hybrid Multi-tasking machine. A material failure occurred on the 316S31 substrate, which had inferior mechanical properties to IN718. According to the report, a high density was achieved (quantity not specified), and no defects were observed at the interface [44]. However, this is unlikely to occur in such processes where defects are inevitable [16]. Li et al. [45] modified a similar work by developing a strategy that utilizes a robotic arm with six degrees of freedom and with multi-changeable heads integrated into the system. The goal was to create a multi-axis AM system. The optimized 6-DOF hybrid additive–subtractive manufacturing process had an estimated production time savings of above 50%. The proposed system was material-efficient and provided better surface finishes [45]. For convenience, a summary of the selected hybrid solutions discussed in this section is presented in **Table 2.1**.

Table 2.1. A summary of hybrid solutions.

Hybrid Technique	Fe-Based Systems	Other Systems	References
AM + CNC part/CNC as post-processing	Maraging steels, tool steels, and SS316L.	-	[25,29]
AM as a repairing technique	Maraging steels, SS316L, and tool steels.	Al6061, Ti-6Al-4V; Ni-super-alloys, and brass.	[37,38]
Combined AM processes	SS316L	Ti alloys, resin, polymers (PLA, PET), and ceramics.	[37,39,41]
Hybrid machine	SS316L	Inconel, PLA, and ABS.	[44,45]
AM process on a conventionally made part	Maraging steels	-	[34,46,47]

2.4 Hybrid Additive Manufacturing as a Joining Technique and Its Challenges

To achieve a reliable component fabricated by joining processes such as welding, a thorough understanding of the interface behaviour of the joined materials is necessary. Traditional joining techniques, such as welding or fastening, have design limitations at the joints. It is well known that there can be localized stresses, corrosion, and inferior mechanical properties in the joints, compared to the parent metals. During welding, materials are heated to a suitable temperature to combine them. A melt pool is created, which solidifies at the rear of the weld pool to create a fusion zone; then, a Heat-Affected Zone (HAZ) is generated adjacent to the base metal. Both AM and welding require localized heat sources to create melt pools. In addition, both have similar behaviours in reacting to the temperature—steep thermal gradients formed at temperatures above the liquidus temperature of the alloy. The cooling behaviour of each material depends on the individual chemical composition and thermal expansion coefficient [16]. Regardless of the techniques used, the joining of dissimilar alloys inherits challenges because the areas in the

vicinity of the interface tend to produce inhomogeneous structures [48,49]. On the other hand, similar melting joining techniques also develop heterogeneous structures at their interfaces. Constricted Arc Welding (GTCAW) was used to join a maraging 250 steel [50], and the resulting microstructure varied across the similar alloy joint. The weld zone region for the maraging steel joint had fine-grained martensite adjacent to a structured dendritic interface. Further away from the weld zone, the HAZ exhibited a mixture of coarse-grained austenitic-martensitic structures. Similarly, a PH 13-8Mo stainless steel joint had a coarse weld [50], resulting in an interface similar to the parent material because of a filler material of similar composition. Such inhomogeneous structures result in various material properties, which are usually undesirable for uniformly loaded structural components [23]. Thermal conductivity differences have been observed in welding to contribute to the lack of fusion in joints. Dissipating heat moves faster in the high-conducting material than in the low-conducting material, resulting in poor fusion.

To help future studies on hybrid/MMAM and expand the usefulness of alloy compatibility, interface reaction products and thermal property mismatch needs to be well understood [23]. The following sub-sections discuss the effects of these parameters based on previous studies.

2.4.1 Alloy Compatibility

Minor alterations to an alloy's prescribed composition or thermal history significantly alter its performance and properties, such as resilience, toughness, and resistance to corrosion. Homogeneity in the microstructure is crucial for joint strength for dissimilar metals, as shown in a study by Aziz et al. [34]. Due to chemical and microstructural inhomogeneity, a hybrid built from MS1, and a wrought H-13 maraging steel substrate fractured at the

interface. Porosity also contributed to the failure, as reported in the study. A solute variation of alloying elements was observed at the interface transition. The chemical composition changed abruptly from Ni, Mo, and Co-based MS1 to H13, which is Cr-based. (**Fig. 2.6**). Chromium diffused through the interface into the MS1 section [34].

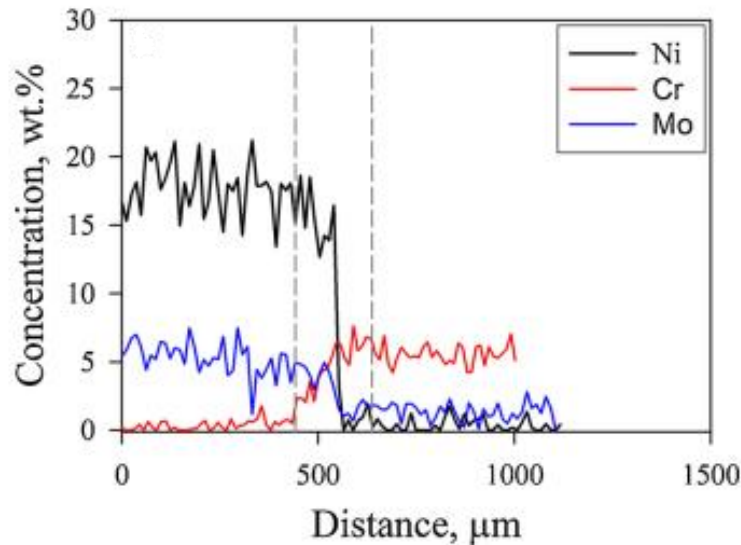


Figure 2.6. EDS analysis of elemental distribution across the interface of MS1–H13 HB [34].

In support of this, Conde et al. [51] noted that elemental diffusion caused alloying elements' depletion and micro-segregation in interdendritic regions. This is consistent with the observations of Shakerin et al. [46], who studied a similar MS1–H13 hybrid material system with some heat treatment modifications. Despite achieving a homogeneous microstructure, adequate precipitation did not reach the interface due to insufficient aging of the required precipitates [46]. Subsequently, sharp compositional interfaces acted as potential gradients, which facilitated the movement of the alloying elements and impurities; these produced various mechanical and physio-thermal properties, which exacerbated the failure.

The joining of dissimilar metals can be achieved in three ways, namely direct joining, the gradient path, and the intermediate section, as shown in **Fig. 2.7** [52]. The application of each technique is given in the following section with a focus on LPBF. A summary of these joining techniques and their outcomes is given in **Table 2.2**

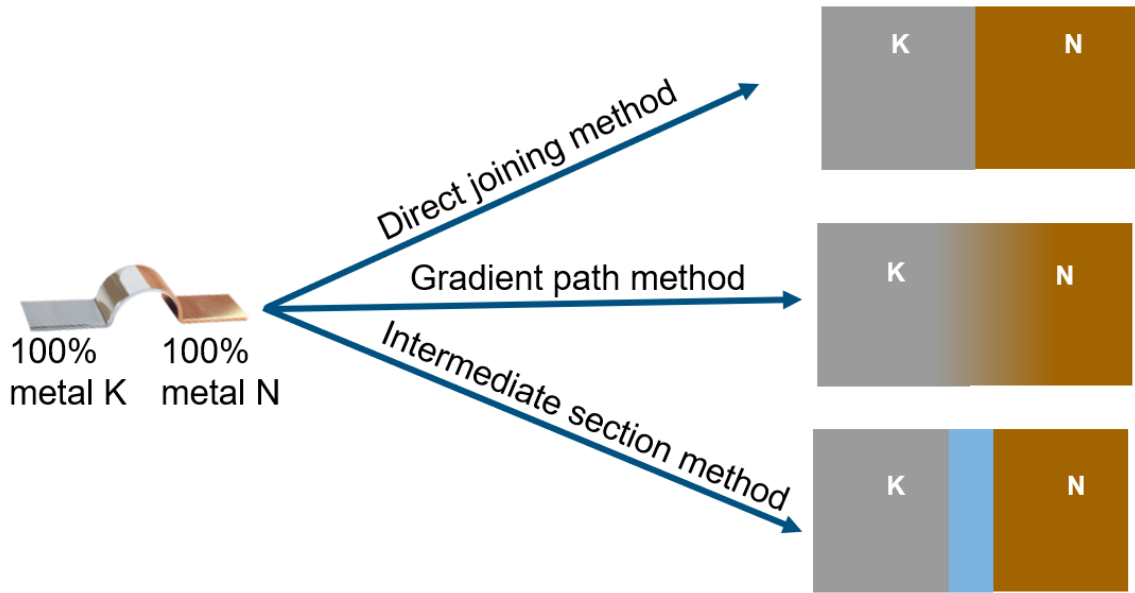


Figure 2.7. Material joining strategies [52].

2.4.1.1 Direct Joining

Liu et al. [53] applied the direct joining technique on a 316/C18400 alloy using LPBF. Cracks were observed on the stainless-steel section close to the interface because the highly conducting Cu diffused into the austenite grain boundaries, resulting in thermal stress inside the matrix. Fused Ion Beam imaging (FIB) revealed an intermix region, approximately 750 μm in length, with a mixture of fine-grained structures (**Fig. 2.8**). Inadequate melting caused substantial porosity in the Cu alloy (**Fig. 2.8c**). This was most likely due to Cu's high reflectivity and heat conductivity at a 300 W laser power. The copper alloy was melted directly on top of the stainless steel, and a large quantity of copper

was presumably present in the melt pool, causing micro-cracks to occur during cooling [53].

LPBF's slow layer-by-layer building strategy produced an excellent metallurgical bond between 316L and Cu10Sn [43]. Appropriate melt pool agitation (Marangoni convection) and equal distribution of alloying components in the heterogeneous alloy phases were acclaimed by Chen et al. [43] for the strong metallurgical bonding.

Furthermore, grain refinement and recrystallization improved the bonding performance at the interface with the following operating parameters: a laser power of 400 W and a scanning speed of 500 mm/s [43]. In another work by Hengsbach et al. [54], a 316L/H13 MMAM produced a sound metallurgical bond at the interface with a build platform temperature of 200 °C. The build temperature was chosen to minimize thermal stresses at the bonding interface. In addition, the good bonding was attributed to the efficient agitation and distribution of elements inside the melt pool. Epitaxial growth at the interface was observed and attributed to the almost similar chemical composition of the two materials. The nickel content in 316L was reported to decrease in each layer scan gradually, and no Ni was detected in the H13 layer. Moreover, the fractography revealed inclusions and pores at the fracture surface on the 316L section [54]. In this work, no post-Heat Treatment (HT) was performed under the premise that localized heat treatment occurred during laser scanning. The benefits of joining materials with similar chemistries was evidenced by a robust interface achieved for a M789-wrought N709 hybrid alloy using LPBF [55]. The interface revealed a fine microstructure with possible precipitation of the η -TiAl phase and β -NiAl phase associated with M789 and N709, respectively [47,55]. In contrast, void nucleation and coalescence were reported at the interface of a Corrax-420SS hybrid

due to mechanical incompatibilities and decohesion [56]. The conclusion from these reports indicated that, with carefully studied elemental diffusion and processing parameters, direct joining techniques are a possible utility for MMAM.

2.4.1.2 Gradient Joining

Gradient path methods offer a gradual change of composition to minimize mismatches at the interface, which could potentially cause interface cracking. To promote the bonding strength between the dissimilar materials, new chemistries can be developed between the two materials to limit the possibility of secondary brittle phase formation, which can arise from compositionally sharp gradients [52,57].

Carroll et al. [58] utilized direct laser deposition of IN625 on SS304L steel to produce a Functionally Graded Material (FGM). Thus, a diffuse gradient zone was produced, with no sharp distinctions and compositional variation. However, cracks were observed in a region containing about 79 wt.% SS304L and 21 wt.% IN625. This was due to the precipitation of a brittle secondary phase [58]. Despite introducing a steady gradient, the appearance of an undesirable phase was unavoidable. Similarly, cracks were also observed in a Ti-6Al-4V/AlSi10Mg FGM with 25% gradient increments due to the formation of brittle phases [48]. Bobbio et al. [59] also reported cracks and a significant fluctuation of elemental composition on a Ti-6Al-4V/Invar FGM. The distinct segregation of phases was observed with Ti-rich particles enclosed in an Invar matrix [59].

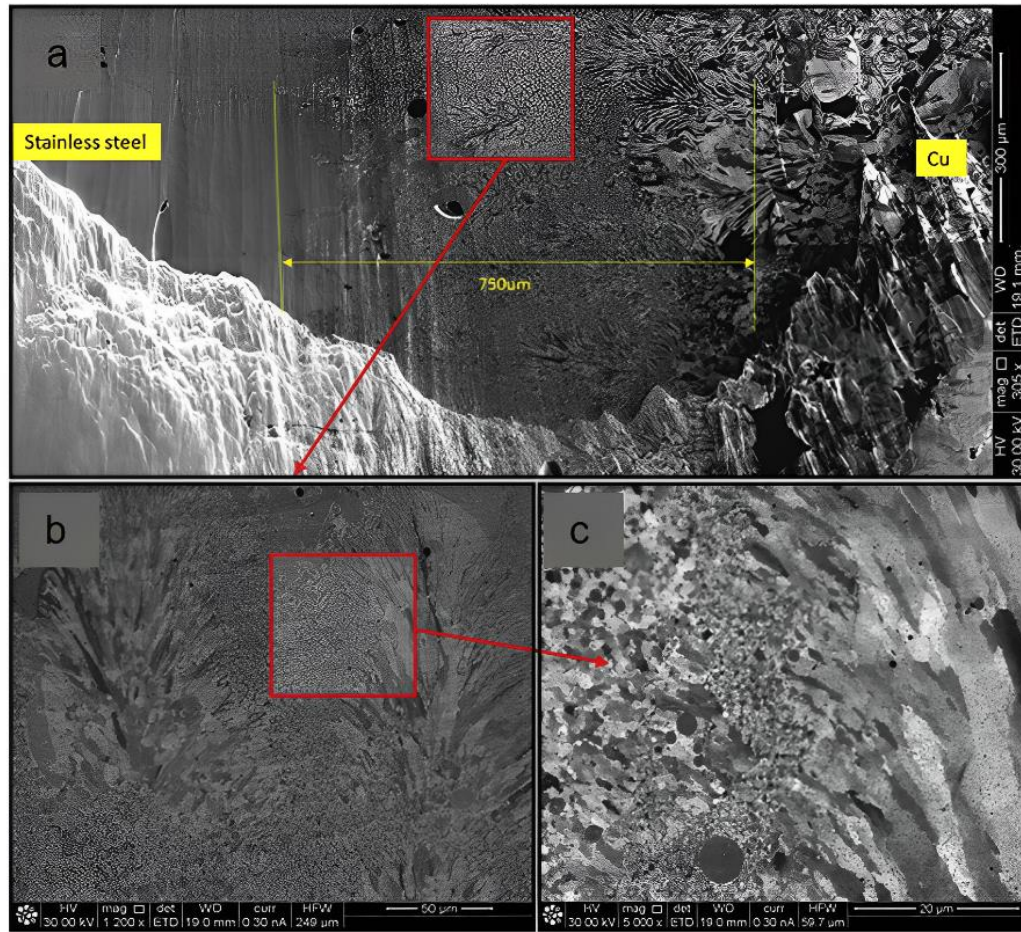


Figure 2.8. (a) Steel and Cu interface observation using FIB; (b,c) zoomed interface [53].

2.4.1.3 Intermediate Layer Joining

Researchers have proposed an intermediate layer technique commonly used in welding to eliminate the precipitation of undesirable phases. Investigations on electron and laser beam welding [60], diffusion bonding [61], and friction welding [62] have been reported to have used inter-layer metals, which form continuous solid solutions with the base materials. The filler material must have matched thermal–physical properties and sufficient thickness to avoid intermixing of the materials involved [23]. Early work by Sahasrabudhe et al. [63] investigated the use of LPBF by directly depositing Ti-6Al-4V on SS410. The resulting

delamination prompted the use of NiCr as an intermediate. This way, the precipitation of detrimental intermetallic phases (Cr_2Ti , FeTi , and Fe_2Ti) was avoided. A sound metallurgical bond was formed without delamination and cracks [63]. The choice of an intermediate is critical to prevent the formation of a detrimental phase between the intermediate section and the FGMs, as reported by Bobbio et al. [64]. The use of V as an intermediate in a Ti-6Al-4V/SS340L FGM resulted in the formation of FeTi and FeV phases, which promoted failure at the interface [64]. Li et al. [65] modified the Ti-6Al-4V/SS316L FGM system and used a V/Cr multi-material intermediate instead. This achieved good solubility between the Fe and Ti-containing base materials [65]. Later work by Onuike et al. [66] also reported successful bonding of a Ti-6Al-4V/Inconel 718 FGM with a VC compositional bond layer, which minimized the formation of the Ni_2Ti and Ni_3Ti brittle phases [66]. Not every intermetallic phase was considered detrimental such as the TiAl intermetallic phase, which has reported favorable lightweight characteristics [66]. Clearly, it is necessary to understand the design of intermediate FGM materials and how they enhance the properties of a joint. However, this inevitably results in more costs in production. Optimization studies and models for intermediate joining techniques must be developed to predict the resultant phases and determine suitable intermediate materials for joining incompatible metals [52].

Table 2.2 identifies the discussed findings in the literature on the use of LPBF to combine dissimilar metals using the direct sharp interface, gradient, and interlayer methods. The observations of the interface performances are highlighted. Based on these observations, it can be concluded that, despite the joining technique employed, the performance of each hybrid is unique based on the material used.

Table 2.2. Summary of the behaviour of dissimilar joints.

Joining Method	Materials	Observations	References
Direct sharp interface	316L/C18400	Sound metallurgical bonding at the interface; however, cracks were evident because Cu diffused into the austenitic grain boundaries. As a result, low ultimate strength was achieved.	[42]
	316L/Cu10Sn	Crack-free interface with a strong metallurgical bond.	[43]
	18Ni300/CuSn	Al ₂ Cu intermetallic phases were observed together with cracks due to non-uniform thermal expansion.	[67]
	1.2709 steel/CuCr1Zr	A 20 mm powder overlap was achieved, but cracks and pores were unavoidable.	[68]
	316 L/In718	Cross-finger interface produced improved the horizontal bond strength.	[69]
	Al12Si/Al3.5Cu1.5Mg1Si	Some Al12Si powders were unmelted, and Al ₂ Cu intermetallic phase was precipitated.	[70]
	Ti5Al2.5Sn/Ti6Al4V	A good metallurgical bond was achieved regardless of HT. The interfacial bond strength was higher than the Ti-5Al-2.5Sn section.	[71]
	AlSi10Mg/C18400	Brittle intermetallic phase Al ₂ Cu was produced with crack formation due to non-uniform CTE.	[42]
	316 l/H13	Fair metallurgical bonding at the interface with improved material intermixing inside the melt pool.	[54]
	MS1/H13	HT potentially removed microstructural inhomogeneity but	[46]

		did not achieve microstructural evolution at the interface.	
	M789/N709	Failure was away from the interface for both as-printed and heat-treated conditions.	[47,72]
	MS1/wrought C300	Fracture at the built AM layer was caused by void expansion.	[34]
	MS1/wrought H13	Fracture at the interface due to chemical and microstructural inhomogeneity.	[34]
Gradient	316L/MS1	Good intermixing between the materials with no visible crack formation.	[73]
	Ti6Al4V/IN718	Composition of above 20% IN718 promoted the formation of brittle Ti ₂ Ni intermetallic phase.	[74]
	316 L/Cu10Sn	Fair metallurgical bonding between the two materials with the presence of unmelted 316L powder.	[75]
	Fe/Al12Si	Materials were immiscible in most sections, and macro-cracks were observed.	[76]
	IN718/Cu10Sn	Good metallurgical bonding due to improved scan strategy and reduced hatch distance, which improved the melting of IN718 powders.	[77]
	Invar36/Cu10Sn	Significant portions of unmelted Invar36 due to low laser absorptivity and high thermal conductivity of Cu alloy.	[78]
Intermediate section	Ti6Al4V/316L	Copper alloy interlayer insert avoided the intermetallic phase formation between Ti6Al4V/316.	[79]

A high degree of thermal compatibility is crucial for robust interfaces. For instance, for the direct joining technique of 316/C18400, cracking occurred due to the differences in the thermal conductivity between Fe and Cu [42]. Copper has four-times greater thermal conductivity than iron, and this difference yields different cooling rates, resulting in higher thermal stresses within the hybrid [78]. The same was observed for the 1.2709/CuCr1Zr [68], 18Ni300/CuSn [67] combinations. Surprisingly, for 316L/Cu10Sn, no cracking was reported [43] post-printing, and this was attributed to recrystallization refinement, better melt pool dynamics, and heterogeneity in the alloy phases. Considering Al₁₂Si/Al_{3.5}Cu_{1.5}Mg₁Si, poor powder melting was reported [70], possibly because of a low volumetric energy density together with the presence of copper, which has a high reflectivity. The presence of Al₂Cu precipitates interrupted the continuous cellular microstructure, ultimately producing low hardness values (<125 HV_{0.05}) in these regions compared with the Al₂Cu-precipitate-free zones (>130 HV_{0.05}) [70]. The use of the gradient method becomes relevant in such cases to avoid the precipitation of undesirable phases by gradually adjusting the composition of one more element (316L/MS1) [73]. However, as observed from Fe/Al₁₂Si, macro-cracks were observed due to the formation of immiscible regions [76]. For specific compositions, miscibility gaps are formed as a result of spinodal decomposition, promoting segregation and mechanical mixtures [80]. Moreover, the gradient method is not suited for most LPBF processes because of limited powder delivery techniques. The use of filler material is an alternative to avoid detrimental phases; however, the design of the filler material is a costly process [79].

The importance of chemical similarity can be observed in gradient joining in M789/N709. These two steels are both precipitation-hardening steels with comparable thermal

conductivities and compositions. The interface formed was robust, and failure was located away from its interface in both the as-printed and heat-treated state [55,72]. The importance of post-heat treatment is discussed in Section 2.6 with an emphasis on precipitation-hardening steels produced by LPBF.

2.4.2 Limited Solubility and Intermetallic Phase Formation

As previously described in [51,61–64,66,72], the precipitation of brittle phases is a significant setback in joining different metals. Most metal systems have limited solid solubilities, which often precipitate intermetallic phases and other related stoichiometric products. On the other hand, the precipitation of intermetallic phases does not always lead to detrimental effects [46]. Other intermetallic products (Ni_3Al , Fe_3Al , and FeAl) improve high-temperature functionality and strength [81]. However, in some cases, the intermetallic formed at the joints in AM fails to accommodate the heat and residual stresses accumulated during processing, which then eventually leads to a loss of toughness and ductility [23].

2.5. Microstructural Characteristics, Mechanical Behavior, and Properties of Hybrid Maraging Steels

The microstructure of an alloy plays a crucial role in determining its functional properties, which can have a significant impact on the material's overall performance. In additive manufacturing, the anisotropy of mechanical properties can be a challenge for applications that experience stresses from multiple directions; thus, making a uniform structure is desirable to achieve isotropic mechanical properties. The contrasting material properties of AM metals compared to conventional metals are mainly due to differences in microstructure characteristics [16]. In the AM process, solidification is influenced by the previously deposited layer and the thermal gradient from the melt pool interior and

boundaries, which can impact grain development and, ultimately, the texture of the AM material [16,82]. Moreover, studies by Murr et al. [83] and Rafi et al. [84] using LPBF demonstrated the potential of AM to produce large quantities of retained austenite ranging up to 75%, as evidenced by the mechanical behaviours of the precipitation-hardening steels studied. Such high quantities of retained austenite are not suitable for die and tool applications [47].

Based on the literature data, summarized in **Table 2.3**, as-built AM alloys generally possess inferior mechanical properties (i.e., yield strength, ultimate tensile strength, and hardness) compared with their conventional counterparts; the data are associated with the properties of 17-4PH, including the effect of the processing parameters. The data suggest that, for the PBF process, high laser power values result in inferior mechanical properties, largely because of the retention of a significant amount of austenite, leading to higher ductility at the expense of strength [16]. LeBrun et al. [85] established that the application of post-heat treatment on 17-4PH can lead to optimum mechanical properties by transforming the retained austenite to martensite. In their work, they showed that the quantity of retained austenite can decrease from 36% to below 3%.

More complex scenarios are presented for dissimilar metals. One such example is the MS1–H13 hybrid produced by Laser Powder Bed Fusion (LPBF) [86], which features a microstructure comprising a large equiaxed grained matrix, oval precipitates on the H13 tool steel side, and ultra-fine martensite grains on the MS1 (LPBF) side. The H13 side of the MS1–H13 hybrid material exhibited a constant hardness value of 232 HVN throughout the section, whereas the hardness on the MS1 side was reported to peak at 644 HVN just after the interface (**Fig. 2.9**), mainly due to the Hall–Petch strengthening mechanism. This

increase in hardness is likely attributed to the presence of fine martensite grains, which provide a greater number of grain boundaries and dislocations, which resist the motion of dislocations and enhance the material's strength. In its as-built state, the MS1–H13 material exhibited a lower ultimate tensile strength of 644 MPa, which is 462 MPa less than that of the MS1 side. The failure occurred specifically at the H13 section with a dimple rupture. The lower strength in the H13 counterpart is attributed to the soft annealing heat treatment performed on the conventional H13 steel prior to the material's use in AM [86].

Table 2.3. Mechanical properties of additively manufactured 17-4PH.

Process-P (W)	V (mm/s)	H (J/mm)	Orientation	E (GPa)	σ_y (MPa)	σ_{uts} (MPa)	Elongation (%)	HVN
DED—350 [86]	8.3	36.1	Absent	40 ± 10	400 ± 100	900 ± 200	5 ± 3	441
PBF—190 [85]	787	0.24	Longitudinal	Absent	661 ± 24	1255 ± 3	16.2 ± 2.5	333 ± 2
PBF—195 [82]	800	0.24	Absent	Transverse	570 ± 13	944 ± 35	50 ± 1	Absent
PBF—95 [87]	350	0.27	Longitudinal	Absent	610 ± 10	1050 ± 20	11 ± 0	Absent
			Transverse	Absent	610 ± 10	910 ± 10	3.5 ± 1.5	Absent
Conventional processing N/A [88]	Wrought, solution annealed and aged	N/A	N/A	199	992	1018	13.4	430

Note: P = laser power, v = scanning speed, H = linear heat input, E = Young's modulus, σ_y = yield strength, and σ_{uts} = tensile strength; HVN is the Vickers Hardness Number.

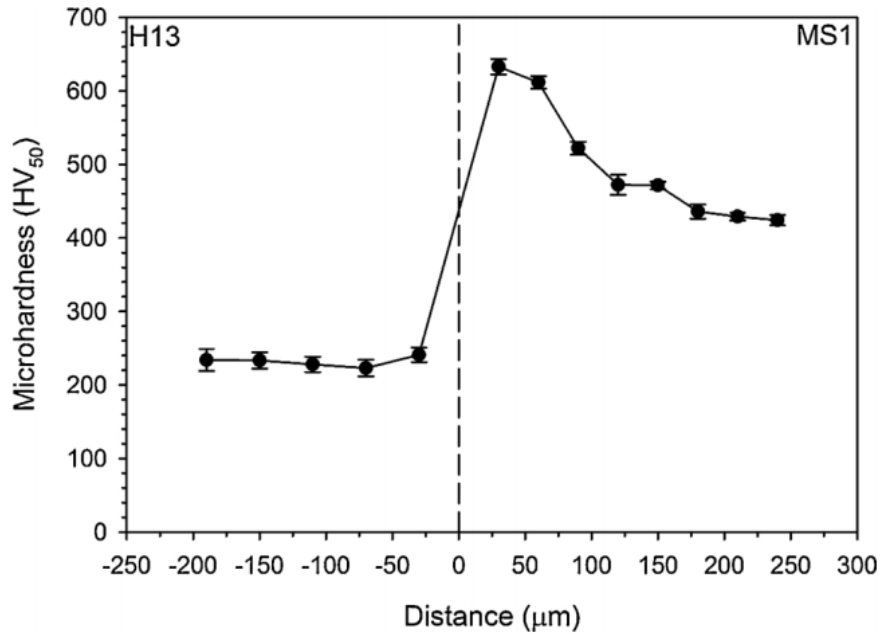


Figure 2.9. Microhardness changes across the MS1–H13 interface [86].

An extension to the previous work [86] involved modifying a similar hybrid material through a heat treatment regime that included Solution Treatment (ST) at 815 °C and 915 °C for 15 min each, followed by aging at 490 °C for 360 min [46]. The resulting material exhibited a uniform hardness averaging 600 HVN across the entire hybrid structure, which was attributed to the coarsening of lath martensite in the MS1 section and solid solution hardening in the H13 steel section [46]. The solution treatment temperatures used minimized the alloy segregation and produced hard martensite after quenching. The material's yield strength increased to around 1860 MPa, but with a lower fracture strain of 5% compared to 23% in the as-built state. Interestingly, the fracture occurred near the interface, which was scarcely distinguishable from the rest of the hybrid material. Direct aging of the hybrid material produced mechanical properties almost similar to the as-built alloy, with fracture occurring in the H13 steel section [46]. These results differed from

those reported by Cyr et al. [90], in which fracture occurred at the interface. In their study, the hybrid material was produced using LPBF and subjected to ST at 815 °C, followed by a 982 °C preheating for 1 h before aging at 490 °C for 6 h, based on the manufacturer's specifications. This heat treatment procedure resulted in a peak strength of approximately 1700 MPa, owing to Orowan loop strengthening with the help of Co and Mo precipitates and the grain refining effect of Ti [91,92]. Non-conventional heat treatment protocols have also been adopted [47,55], whereby direct aging is used to avoid the loss of strength in a peak-aged hybrid base. An intact interface was obtained in both the as-printed and aged states. In the as-printed state, failure occurred in the printed material M789 at about 1100 MPa, and after aging, failure occurred in the N709 at approximately a 1600 MPa base plate. The effects of direct aging improved the strength of M789 by precipitation of the η -phase, at the same time minimizing the loss of strength in the hybrid base.

Based on the literature, developing an optimized heat-treatment schedule is crucial for achieving the desired mechanical behaviour in a hybrid material system, regardless of the additive manufacturing technique used. It has been noted that individual heat treatment methods for each material may not provide the best combination of strength and ductility, as observed by Cyr et al. [90]. This is particularly relevant for AM maraging steels and wrought medium carbon steels, which can benefit from a well-designed heat treatment. The ideal heat treatment procedure should balance the strength and ductility of the hybrid material while eliminating microstructural inhomogeneities, which may vary depending on the specific hybrid material system. A systematic approach is required to arrive at an optimized heat treatment schedule based on the intended application.

Aziz et al. [34] employed a combination of solution treatment and aging to modify the properties of a similar MS1–H13 hybrid material, as demonstrated in **Fig. 2.10**. The resulting microstructure consisted of a coarse-grained H13 substrate and a fine-grained MS1 layer (**Fig. 2.11**). The solution treatment involved heat treatment up to 1030 °C, followed by quenching, which caused the H13 alloy to revert to austenite and form a hard martensitic structure upon cooling. The MS1 layer, on the other hand, experienced a reduction in hardness due to austenite formation. Aging at 650 °C for 60 min further enhanced precipitate formation and resulted in a maximum hardness of 420 HVN for MS1 and 520 HVN for H13, respectively. In the as-built state, the H13 layer had a lower hardness of 220 HVN than MS1, which averaged 420 HVN. The solution treatment improved the hardness of H13 to about 600 HVN and lowered the hardness of the AM layer, which is in agreement with the findings of Shakerin et al. [46] and LeBrun et al. [85]. Interestingly, this finding contradicts previous reports suggesting the formation of austenite, which typically has lower mechanical properties [16]. In the case of the H13 alloy, it contained approximately 0.40% C, which resulted in the formation of a hard and brittle martensitic structure after undergoing solution treatment. During tensile testing of the MS1–H13 hybrid material, the fracture occurred at a stress and fracture strain of approximately 1100 MPa and 11%, respectively. Before the fracture, a considerable amount of strain was concentrated in the dissimilar joint. The inhomogeneous microstructure resulted in strain concentration in the dissimilar joint. In conclusion, the impact of solution treatment on the two alloys varied. It caused H13 to revert to austenite and form a hard martensitic structure upon cooling. After solution treatment, MS1

experienced a reduction in hardness due to austenite formation while enhancing precipitate formation (**Fig. 2.12**) [34].

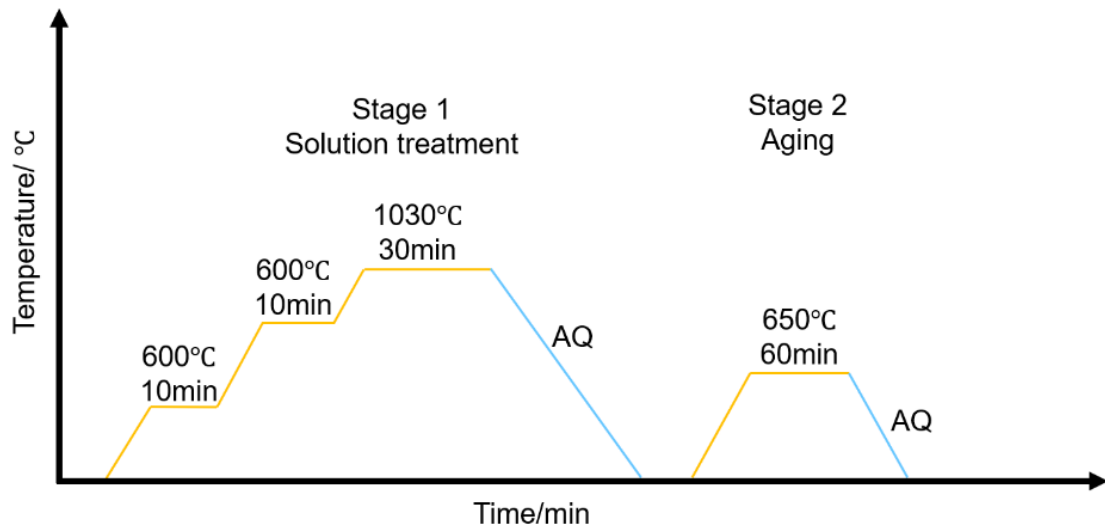


Figure 2.10. Heat treatment profile for MS1–H13 hybrid. AQ: Quench [34].

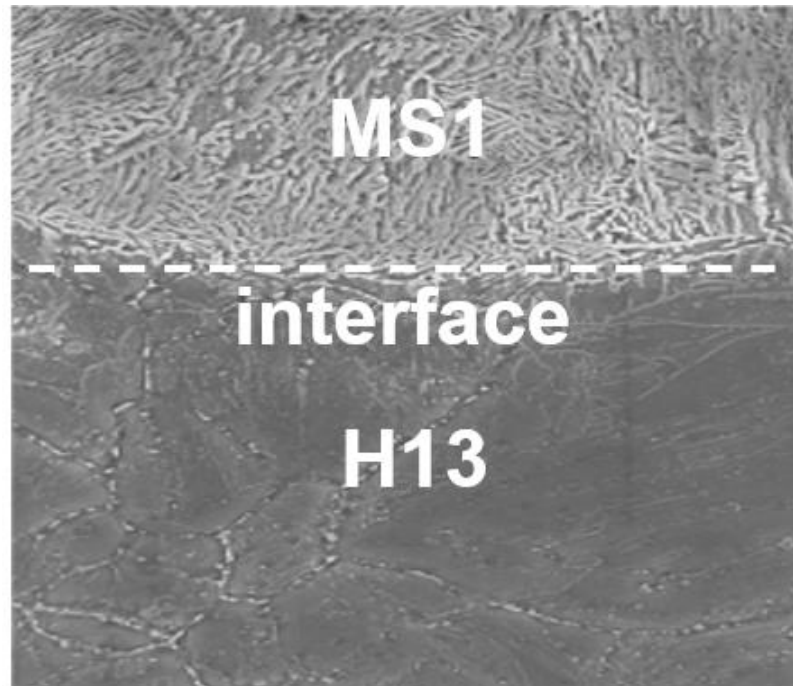


Figure 2.11. Microstructure variation between wrought H13 and printed MS1 [34].

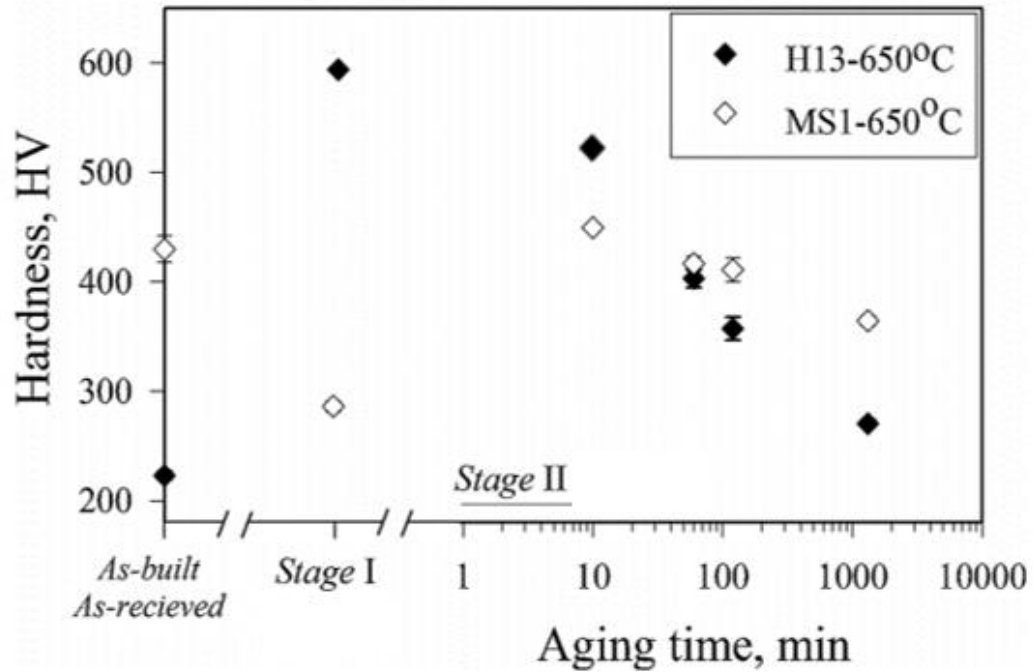


Figure 2.12. Hardness variation at different conditions [34].

The properties of an MS1–C300 hybrid material were compared in a similar study [34], where the C300 substrate was employed and the MS1 was deposited using LPBF. The individual hardness values of the MS1 and C300 after aging were found to be similar to the MS1–C300 hybrid material, with a peak value of 450 HVN achieved after solution treatment at 850 °C for 90 min, followed by aging at 650 °C for 10 min. During aging at 490 °C, it was observed that the hardness value continued to increase steadily beyond 67 h of aging time. The peak hardness was not determined, indicating that no over-aging had occurred. Mechanical testing of the MS1–C300 material after aging at 650 °C revealed a fracture in the MS1 portion with an ultimate tensile strength of 1200 MPa, which was 900 MPa higher than that of the individual C300 alloy aged at 490 °C. The optimal temperature for good mechanical properties was found to be 650 °C, which increased the hybrid material’s ductility to 10% due to a loss of strength at higher temperatures. The failure in

the MS1 section was attributed to about a 10% lower flow stress and initial porosity, which facilitated void expansion and coalescence. Pore defects acted as stress concentration points, which exacerbated failure [21]. The strain was localized in the MS1 region after the peak strength was reached [34].

The studies mentioned above have shown that the primary objective of hybrid materials is to attain maximum hardness after adequate aging of the printed alloy and tempering of the conventional material to achieve uniform properties. However, there are still challenges involved in achieving the desired properties due to the different responses of various materials.

2.6 Effect of Heat Treatments in AM Processed Maraging Steels

The strength of maraging steels relies primarily on the presence and distribution of strengthening precipitates. The impact of these precipitates and how they affect the final strength of maraging steels are discussed.

2.6.1. Strengthening Mechanisms' Models

The strengthening mechanism for maraging steels includes solid solution strengthening, dislocation hardening, and precipitation strengthening. In addition, the effect of these mechanisms may be utilized to evaluate the Vickers hardness and yield strength of maraging steels according to Equation (2.1) [92–94].

$$H_v = \frac{1}{3}\sigma_y = \frac{1}{3}(\sigma_{ss} + \sigma_{mart} + \sigma_p) \quad (2.1)$$

where σ_{mart} is the effective martensite strength, σ_p is the precipitation strengthening, and σ_{ss} is the contribution of solid solution strengthening.

2.6.1.1 Solid-Solution-Strengthening Model

The Fleisher equation is used to determine the critical resolved shear stress due to the substitution of solute elements by other alloying elements involved:

$$\sigma_{ss} = \sum \left((\beta_i^2 x_{i,\alpha})^{\frac{1}{2}} \right) \quad (2.2)$$

where $x_{i,\alpha}$ is the atomic fraction of the element in consideration (x_i) in the matrix and β_i is the constant strengthening value that describes the lattice and modulus misfit of the element concerning the major element [95], which in this case is iron. **Table 2.4** shows the iron values of several solution-strengthening constants for alloys studied by Nava et al. [92]. These values assume infinite solid solubility in iron. According to the values in **Table 2.4**, Mo and Ti give the strongest solid solution strengthening. With increasing particle volume fraction, the effect of the precipitate forming elements (Mn, Ni, Ti, Mo) decreases. The changes in $x_{i,\alpha}$ are determined by Equation (2.3).

$$x_{i,\alpha} = \frac{x_i - \sum x_{ij} f_j}{1 - \sum f_j} \quad (2.3)$$

where f_j represents the volume fraction of particle j and x_{ij} is the volume fraction of element i in j [92].

Table 2.4. Constants for solid solution strengthening [92].

Element	β_i (MPa/at)
Ni	708
Mn	540
Cr	622
Al	196
Ti	2628
Mo	2368

2.6.1.2 Effective Martensitic Strength

Grain boundary area and dislocation density are contributors to the adequate strength of the martensite matrix in maraging steels [96]. The block size d_{block} (**Fig. 2.13**) is considered the practical grain dimension and formulated in the form of the Hall–Petch relationship and the Taylor equation, which resolves the strength effect to the dislocation density [95].

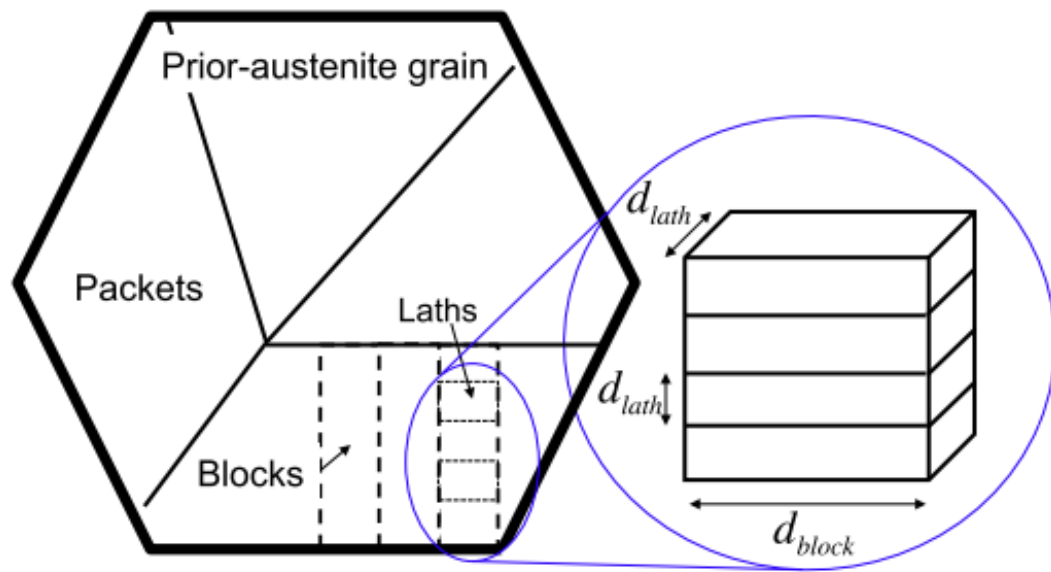


Figure 2.13. Sub-structure sketch of a typical martensite grain [92].

The combined formulation is presented in Equation (2.4).

$$\sigma_{\text{mart}} = \frac{300}{\sqrt{d_{\text{block}}}} + 0.25M\mu b\sqrt{\rho} \quad (2.4)$$

where μ is the shear modulus, ρ is the dislocation density after solution treatment, b is Burger's vector, and M represents the Taylor orientation factor. However, for the simplicity

of calculations, d_{block} is assumed to be constant, despite reports mentioning the existence of small variations in the sizes even in aged conditions [96,97]. The low distortions are due to low lattice distortions and the high solubility of solute elements [95].

2.6.1.3 Precipitation Strengthening

The presence of precipitates hinders dislocation gliding. They act as obstacles, which reduce their mobility, hence reducing slip and grain boundary sliding. Based on the size of the precipitate and its coherency inside the matrix, dislocations have two main ways of passing these obstacles, viz. bowing (Orowan–Ashby model) [99] and shearing (Friedel model) [100], as described in Equations (2.5) and (2.6), respectively.

$$\sigma_p = 0.26 \frac{\mu b}{r_p} f^{\frac{1}{2}} \ln \left(\frac{r_p}{b} \right) \quad (2.5)$$

$$\sigma_p = \frac{1}{b} \frac{2S}{\sqrt{\pi} w_q} \left(\frac{\gamma w_r r_p}{S} \right)^{\frac{3}{2}} \sqrt{f} \quad (2.6)$$

where r_p is the particle radius (this model assumes that particles are spherical), S is the dislocation line tension ($\mu b^2/2$), w_r and w_q are particle statistical constants [100], γ is the antiphase boundary energy associated with the sheared precipitates, and f is the fractional volume of precipitates. In the case of maraging steels, the pile-up model by Ansell and Lenel assumes that yielding will occur after a critical number of dislocations' pile up exceeding the maximum stress [101] (Equation (2.7)). The Orowan–Ashby model and the modified Orowan–Ashby model [102] (Equation (2.8)) are considered to be the most-relevant, according to Niu et al. [95]. In contrast to Schnitzer et al. [97], the Orowan and

the Friedel models had good fitting results with experimental results based on a PH 13-8Mo stainless steel under aging and over aging conditions.

$$\sigma_p = 2 \frac{\mu'}{4C} \left(\frac{f^{\frac{1}{3}}}{0.82 - f^{\frac{1}{2}}} \right) \quad (2.7)$$

$$\sigma_p = \frac{2\mu b \phi \ln \left(\frac{d - 2r_p}{2b} \right)}{4\pi(d - r_p)} \quad (2.8)$$

where μ' is the shear modulus of the precipitates, C is a constant, d is the inter-particle spacing, and Φ is related to the Poisson ratio (ν) by the following relationship: $\Phi = [1 + 1/(1 - \nu)]/2$. The previously mentioned scenarios assume that only one form of precipitate exists, which is not the case as purported by Nava et al. [92]. Ni-Ti-rich clusters were associated with Mo-rich phases. According to Ardel [103], the total precipitate strength is the summation of each particle's contribution, represented by Equation (2.9).

$$\sigma_p = \left(\sum_j \sigma_j^2 \right)^{\frac{1}{2}} \quad (2.9)$$

where σ_j is the strength increase due to precipitate j with f and r_p , following Equations (2.5) to (2.8).

There are limitations in using these precipitation models (Equations (2.2) – (2.9)) [104], as their exact contribution of precipitation to strength is difficult to determine because their exact interplanar spacing is not accounted for from the volume and fraction sizes and the lattice misfits between the matrix and precipitates. For example, it was shown that the

Orowan model outputs different magnitudes with a factor of 10 in some instances based on the type of distributions used [95]. In the case where several mechanisms are expected to proceed with different precipitate sizes, Schnitzer et al. [105] suggested that no single model can account for the peak hardness. Hence, based on the precipitate sizes, strength can be estimated by the shearing model below a critical size value and the Orowan model above this value. The previously mentioned Ansell and Lenel model [101] can be used to describe the contribution of fine precipitates; however, the model is appropriate for a constant volume fraction. To accommodate varying precipitate sizes during different aging times, a new formulation (Equations (2.10) – (2.12)) is better suited.

$$\Delta\sigma_{Oro} = \frac{0.81M_T G b \ln \frac{2r_s}{b}}{4\pi r_s \left[\left(\frac{\pi}{4\Phi} \right)^{0.5} - 1 \right] (1-\nu)^{0.5}} \quad (2.10)$$

$$\Delta\sigma_\beta = \min(\Omega_{\beta,Oro} | \kappa_{\beta, ratio} \Omega_{\beta, sh}) \quad (2.11)$$

$$\Delta\sigma_{\beta, Oro} = (\Phi)^{0.5} \frac{0.81M_T G b \ln \frac{2r_s}{b}}{4\pi r_s \left[\left(\frac{\pi}{4\Phi} \right)^{0.5} - 1 \right] (1-\nu)^{0.5}} \quad \Delta\sigma_{\beta, Sh} = (\Phi)^{0.5} \frac{2M_T S}{br(\pi r \omega_q)^{0.5}} \left(\frac{\pi r \omega_r Y}{S} \right)^{0.5} \quad (2.12)$$

where $r_s = \left(\frac{2}{3} \right)^{0.5} r$, M_T is the Taylor factor, G is the matrix shear modulus, b is the magnitude of the dislocations of Burger's vector, ν is Poisson's ratio, ω , q , and r are constants derived from particle statistics, S is the dislocation line tension given by $S = Gb^2/2$, and r and Φ are the particle radius and volume fraction, respectively.

This was used to determine the strength contribution of NiAl precipitates in a 9922 alloy (maraging steel) undergoing shearing and Orowan looping [104]. The precipitate sizes were normalized with a standard deviation of one under the assumption that the overall strength is a combination of the two mechanisms. In addition to precipitate strengthening,

the increase in the strength of maraging steels involves contributions from the lath structure (σ_l), the intrinsic strength of iron (σ_{Fe}), and Solid Solution strengthening (SS). As a limitation of most models, the contribution of the SS is not well defined. Various researchers have identified different relationships between the composition (χ) of solute elements and the resulting strengthening in maraging steels. These relationships often exhibit varying values of the composition exponent, with commonly used values being 1 or 1.5. Additionally, different proportionality constants are used [106,107]. The strengthening varies in a complex way with changes in the elemental concentrations.

The contribution of these factors is represented in Equations (2.13) and (2.14). The hardness of the lath structure and Fe matrix is also included.

$$\Delta\sigma_{ss}(HV) = 14.5x'_{Al} + 14.5x'_{Mo} - 4.5x'_{Ni} + 14.5x'_w \quad (2.13)$$

$$\sigma_0 = \sigma_{Fe} + \sigma_l \quad (2.14)$$

For the 9922 alloy, the size and distribution of precipitates was determined using Atomic Probe Topography (APT) and Small-Angle Neutron Scattering (SANS), and the overall strength can be represented as shown in Equation (2.15).

$$\sigma_Y = \sigma_0 + \Delta\sigma_{ss} + \kappa L \Delta\sigma_{Oro} + \kappa\beta \Delta\sigma_{\beta} \quad (2.15)$$

Where κL , $\kappa\beta$, and $\kappa\beta$ ratio are fitting variables.

The results of this model (**Fig. 2.14**) showed that the strengthening via the Orowan model decreases with extended aging time because of precipitate coarsening. A noticeable jump in the NiAl precipitate was observed based on the SANS measurements. The estimated strength contribution of the Laves phase was difficult to determine as evidenced by the

small-sized precipitates reporting a similar hardness with the precipitates with a large size fraction. Solid solution strengthening decreased with the aging time because of the diffusion of strengthening elements towards the Laves phases. This suggests that the growth of the Laves phases is faster at higher temperatures, and hence, the contribution of the solid solution to the overall hardness is expected to diminish at higher temperatures. In contrast, at low aging times, the NiAl precipitates, along with other intrinsic strengthening contributions such as solid solution, lath structure, and matrix effects, play a dominant role. However, at extended aging times exceeding 50 h, the dominance shifts to the formation of Laves phases. Some discrepancies can be observed in this model as it shows that the peak strength at 560 °C is lower (475 HV) than the experimental value (520 HV) at a total aging time of about 50 h. Conversely, the predicted hardness is higher than the actual value at 520 °C. The model failed to predict the complete behaviour, potentially due to errors from the acquired SANS data.

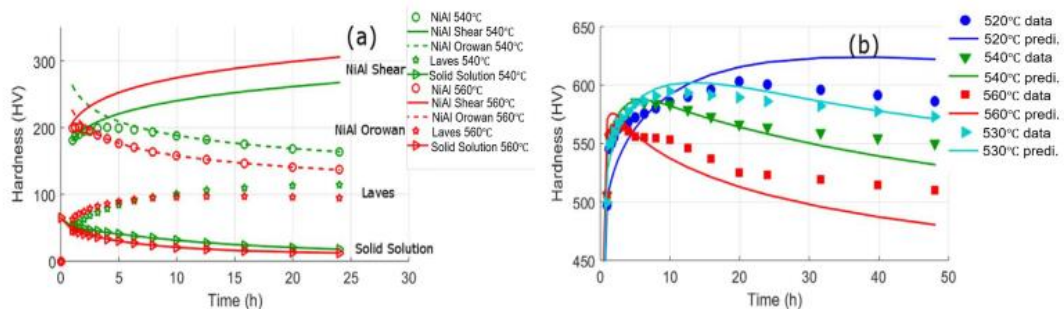


Figure 2.14. (a) Precipitate strength contribution of the NiAl and Laves phases and (b) total strength [104].

Better predictability can be achieved by isolating the phases and studying the dominant strengthening mechanism for each phase at different aging temperatures and how it relates to the volume size and fraction. To isolate the strengthening effects of different phases, [108] used APT to calculate the spacings between the precipitates and to determine

the contribution of the β and η strengthening phases in Fe-12Ni. In this study, two models were used. Model 1 included the contribution of the grain size, dislocation density, and solid solution strengthening. This was later used to validate the results from Model 2, which incorporates the mean free radius and distance and the surface-to-surface distance of precipitates as measured from the APT data. It was found that the precipitation strengthening of the combined β and η phases was close to 1000 MPa at an aging temperature of 510 °C for 16 h and that the relative strengthening effect of each phase was sensitive to the Ti and Al concentration. Low titanium concentrations (approximately 1 wt.%) and high Al concentrations (approximately 1.6 wt.%) in the β phase contributed more to the overall strength compared to high Ti concentrations (approximately 1.5 wt.%) and Al concentrations (0.9 wt.%).

In the case of more complex microstructures, such as those observed in LPBF–Corrax [109], precipitation strengthening was found to be the main contributor to strength via the shearing mechanism. Three factors can be considered to contribute to the overall strength according to Equation (2.16) [110].

$$\Delta\sigma = \Delta\sigma_o + \Delta\sigma_c + \Delta\sigma_m \quad (2.16)$$

where $\Delta\sigma_o$ is the order strengthening, $\Delta\sigma_c$ is the coherency strengthening, and $\Delta\sigma_m$ is the modulus mismatch strengthening. Order strengthening arises from the antiphase boundaries during dislocation shear through the ordered β -NiAl, and it is expressed as:

$$\Delta\sigma_o = 0.4M \frac{\gamma_{APB}}{b} \left(\frac{3\pi f}{8} \right)^{0.5} \quad (2.17)$$

where γ_{APB} is the antiphase boundary energy of the precipitates. M is the Taylor factor, and f is the volume fraction of precipitates.

Coherency strengthening, which arises from the mismatch strain between the coherent precipitates and the matrix, is given by:

$$\Delta\sigma_c = 2.6M(\varepsilon G)^{1.5} \left(\frac{2rf}{Gb} \right)^{0.5} \quad (2.18)$$

where r is the precipitate mean radius and ε is the lattice mismatch parameter.

Lastly, the modulus mismatch strengthening arises from the variation in the dislocation energy as dislocations approach a precipitate with a different shear modulus to the matrix:

$$\Delta\sigma_m = 0.0055M(\Delta G)^{1.5} \left(\frac{2rf}{(Gb)^{0.5}} \right)^{0.5} b \left(\frac{r}{b} \right)^{1.5m-1} \quad (2.19)$$

where ΔG is the shear modulus mismatch between the precipitate and matrix (G_p and G , respectively) and m is a constant (0.85).

The estimation of the final yield strength was estimated to be about 1530 MPa as a contribution of the hierarchical structure of the microstructure. However, based on [104,105] previously discussed, the volume and size fractions need to be considered over the aging times for a more-accurate representation, in addition to the contribution of the high dislocation densities in AM parts.

Maraging steels are highly valued for their combination of strength, toughness, ductility, and weldability. Heat-treatment procedures can be used to optimize their properties by controlling the precipitation of intermetallic phases within the low-carbon Fe-Ni/Mn martensitic matrix. Additive manufacturing offers a unique platform for producing

maraging steels with tailored microstructures and mechanical properties [4,111,112]. One common approach is to homogenize the material by solution treatment, followed by aging at a lower temperature to promote the precipitation of nanoscale intermetallic phases such as $\text{Ni}_3(\text{Mo}, \text{Mn}, \text{Al})$ and Ni_3Ti [113,114]. The choice of alloying elements plays a critical role in determining the mechanical properties by influencing the type of intermetallic phases formed. The nucleation and growth of precipitates are dependent on the segregation energy and the degree of diffusion of the solute atoms involved. The interaction energy associated with the alloying elements is expressed by Equation (2.20). This formula can be used to determine the tendency of elemental segregation.

$$\Delta E_{AB} = E(A + B, N - 2) + E(N) + E(A, N - 1) + E(B, N - 1) \quad (2.20)$$

where $E(A + B, N - 2)$ is the supercell energy for two different solutes located in different nearest neighbour positions and $N - 2$ is the major alloying element atoms (Fe in the case of maraging steels). $E(N)$ is the supercell energy with N number of Fe atoms. $E(A, N - 1)$ and $E(B, N - 1)$ are the supercell energies associated with A and B solute atoms and $(N - 1)$ Fe atoms. As demonstrated in **Fig. 2.15**, the research of Niu et al. [95] showed the variance of the interaction energy between different solute combinations. The interaction energy between elements that are further away in the periodic table is lower compared to elements in the vicinity. As a result, the interaction energy between Ni and Co at the second nearest neighbour is negative (**Fig. 2.15**), implying that Ni will be attracted to Co at the second nearest neighbour position. The interaction energy between Ti and Co is also negative at the first nearest neighbour position (Co), resulting in the mutual attraction of Ti and Co during aging. In the case of the Ti-Mo and Ni-Mo systems, the nearest atom interaction energies are positive, indicating a tendency for Mo repulsion from the Ni-Ti-

rich clusters. Additionally, the third nearest neighbour energy for Ti-Mo is negative, suggesting that Mo atoms will be excluded from the Ni-Ti-rich clusters and, instead, form a separate Mo-rich phase. Due to the relatively low aging temperatures employed in the study (480 °C), the diffusion of Mo is slow, which explains the absence of a Mo-rich phase in the early stages of aging. However, as the aging process advances, it was observed that a Mo-rich phase started to nucleate at the interface between Ni₃Ti and the matrix.

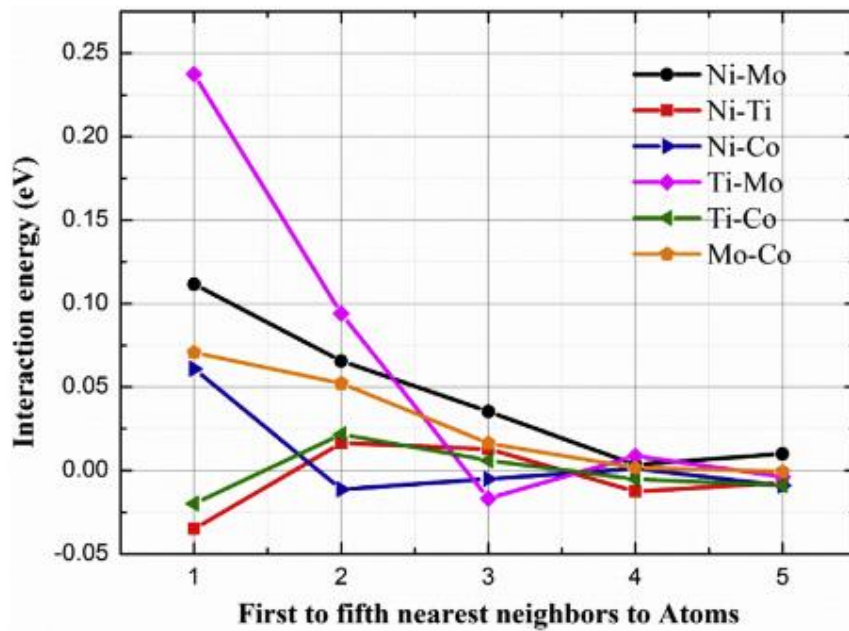


Figure 2.15. Interaction energies of different element combinations [95].

Due to the low interaction energies between Ni, Ti, and Co, these elements are closely associated with each other. The combination of these elements during aging can be classified into three potential categories (refer to **Fig. 2.16**):

- (a) All three elements form a cluster distribution (cd);
- (b) Ni and Ti combine to form a cluster distribution in the iron-rich region (cd), resulting in Co being dispersed (dd);
- (c) Co, in turn, forms clusters with Ni and Ti.

The stability of each of these possibilities may be compared to the dispersed distribution of alloying elements, which can be expressed using Equation (2.21).

$$\Delta E(X - dd_{Co} - dd_{Ni} - dd_{Ti}) = (E_X - E_{Fe-Co} - E_{Fe-Ni} - E_{Fe-Ti}) \quad (2.21)$$

where E_X is the distribution energy of the involved elements in this case (Ni, Co, and Ti), E_{Fe-Co} , E_{Fe-Co} , and E_{Fe-Ti} are the energies associated with the dispersed distribution. The lowest energy state is when all three elements form a cluster together. Since Ni and Ti have the lowest interaction energy, they tend to nucleate in the form of Ni_3Ti with an association of Co and Fe atoms with formation energies similar to the Ni and Ti sites. The formation energy can be represented by Equation (2.22).

$$E^f = nE_{Ni_3Ti-Fe/Co} - \left(\left(\frac{3}{4}n \right) \text{ or } \left(\frac{3}{4}n - 1 \right) \right) E_{Ni} - \left(\left(\frac{1}{4}n - 1 \right) \text{ or } \left(\frac{1}{4}n \right) \right) E_{Ti-E_{Co}/E_{Fe}} \quad (2.22)$$

where $nE_{Ni_3Ti-Fe/Co}$ is the energy of the supercell with one Co or Fe atom, $3/4n$ or $(3/4n - 1)$ is the bulk energy of Ni atoms, and $(1/4n - 1)$ or $1/4n$ is the bulk energy of one Co or Fe atom. **Fig. 2.16** shows that, regardless of Co's and Fe's occupied Ni and Ti sites, the formation energy of Ni-Ti clusters is less endothermic, and as aging progresses, Co and Fe will be rejected from Ni_3Ti .

For example, in the early work by Lang et al. [115], high-Ni-content-based maraging steels with Ti led to the precipitation of Ni_3Ti phases. Partial substitution of the Ni by Fe and Co in some sublattices was also reported, together with the formation of Fe_2Mo phases at extended aging treatments [112,115]. This is consistent with the results of Tian et al. [93] and Bai et al. [116] on the Heatvar alloy and 18Ni-300, respectively.

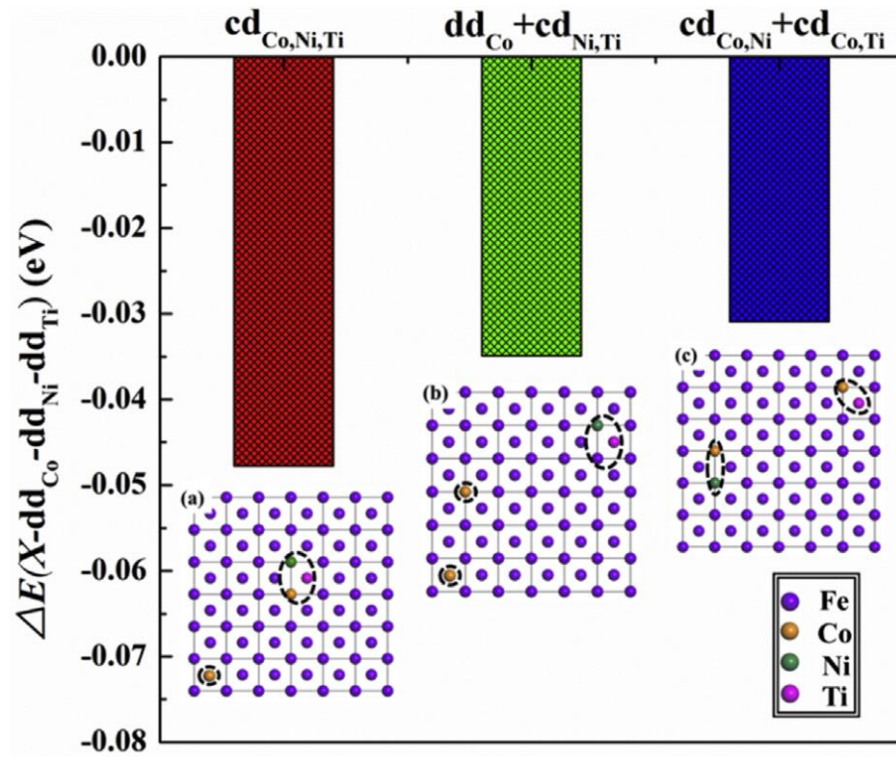


Figure 2.16. Clustering of (a) Co, Ni, and Ti, (b) Ni, and Ti, and (c) Co distributions relative to their dispersion in BCC Fe [95].

In the latter study [116], it was observed that heat treatment led to variations in both mechanical and microstructural properties. Surprisingly, Directly Aged (DAG) samples and Solution treatment followed by Aging samples (SAG) displayed a comparable hardness (654 HVN) and tensile strength (2126 MPa), despite their distinct microstructures. Austenitization above 900 °C resulted in the growth of austenite grains, producing large lath martensite, and solution treatment facilitated the migration of segregated Mo and Ti into the grain, causing the Mo- and Ti-rich white grain boundaries to disappear. Previous studies, by Aboulkhair et al. [117], also observed the disappearance of alloy-rich grain boundaries after solution treatment. However, white borders with high Mo and Ti content were seen in the grain boundaries compared to the DAG samples. This

suggests that the maximum temperature used for aging (560 °C for 12 h) did not provide sufficient energy to diffuse Mo and Ti from the grain boundaries into the grain interiors. For maraging steels generated by LPBF, solution treatment may be unnecessary, and a direct aging strategy can be utilized due to the rapid cooling (103 to 108 K/s [16,118]) during processing, which produces lath martensite. While employing DAG may be a cost-effective option for post-heat treatment of maraging steels generated by additive manufacturing, this may not always be true [116,119]. Solution treatment is known to improve ductility and reduce micro-segregation, and achieving a good balance of ductility and strength is crucial for practical applications. However, at extended periods, it can cause the reversion of martensite into austenite [85]. Additionally, residual stress from additive manufacturing processes can adversely affect mechanical performance [14]; therefore, solution treatment is deemed a necessary step. It is worth noting that this study failed to clearly explain the response of martensite laths' size to different heat treatments, which is a crucial factor in establishing the effectiveness of each heat treatment procedure.

In a previous study by Jäggle et al. [112] on 18Ni-300 steel, it was found that localized heating during the AM process did not result in phase transformations due to the rapid heating/cooling. However, after aging at 480 °C for 5 h, three main phase precipitates were observed, namely $(\text{Fe,Ni,Co})_3(\text{Ti,Mo})$, $(\text{Fe,Ni,Co})_3(\text{Mo,Ti})$, and $(\text{Fe,Ni,Co})_7\text{Mo}_6$ (μ -phase). Atom probe tomography results identified the existence of two Ni_3X phases, with one phase having a lean Ti concentration compared to Mo in a ratio of 1:3 and the other phase having a lean concentration of Mo compared to Ti in a ratio of 1:3. Reverted austenite was suggested to be present in the Ni-rich patches detected in the precipitate-free zone. The equilibrium concentration of austenite at 480 °C was calculated using thermodynamic

calculations and was confirmed through experimental validation. The study observed theoretical and empirical deviations in Ni concentrations for the martensite, with 4 at. % and 10 at. %, respectively. The cause of this discrepancy was not clear, as it could have been due to the precipitation of the new intermetallic or slow diffusion of Ni [112].

Allam et al. [120] focused on increasing the number density of intermetallic precipitates to improve the homogeneity of 300 maraging steel produced using LPBF. The absence of nano-precipitates in the as-printed state was confirmed by the earlier findings by Jägle et al. [112]. The authors suggested that Ni segregation under direct aging at 510 °C for 6 h possibly reduced the potential active sites for nucleation, favouring growth over nucleation. On the other hand, solution aging (900 °C for 1 h + 510 °C for 6 h) promoted fine precipitates with significant density, limiting austenite reversion [120]. These findings can expand the ability to control the different mechanical properties of materials by adjusting the volume density of precipitates. In contrast to the findings of Bai et al. [116] and Thorsteinsdóttir et al. [119], the results obtained in this study demonstrated that solution treatment is an essential step for effectively managing and controlling precipitation, as well as achieving the desired mechanical properties.

Tian et al. [93] conducted a study on the Heatvar alloy, comparing the effects of solution and DAG on the precipitates' evolution in the as-printed state. The study found that the alloy exhibited a Laves phase during solidification along the melt pool boundaries where Mo was segregated. At ambient conditions, this phase was found to possess brittleness, making it prone to act as crack nucleation sites [121]. A theoretical equilibrium partition coefficient of 0.34 and a solid front velocity solidification averaging 0.5 m/s confirmed this observation together with early reports on Fe-Mo systems [111]. To address this issue,

solution treatment was conducted, which simultaneously dissolved the eutectic phase and improved the alloy's elongation from approximately 4% to 15%. The solution treatment was carried out at an elevated temperature of 1233 K for 30 min, which ensured the redistribution of alloying elements inside the grain interior.

M_6C and MC carbides were identified, consistent with Liu et al.'s work [122], who confirmed the observations. These carbides provide sufficient strength and ductility to the alloy. A comparison of the as-printed and solution-treated samples showed a good balance of the strength and ductility in the solution-treated samples, making the heat treatment suitable for low-strength, high-ductility applications. Direct aging at 903 K for 240 min resulted in the highest strength value, averaging 1980 MPa. During the aging of maraging steels, Fe_2Mo precipitates with a size of approximately 36 nm were formed [122], which were confirmed by TEM analysis. The disappearance of previously observed melt pool boundaries was also observed, similar to the solution-treated samples [93]. The most-complex material was produced with a distribution of around 20% precipitates and a hardness of over 50 HRC. However, there was a considerable loss of ductility of 13% EL compared to the ST samples due to the production of hard precipitates, as shown in **Table 2.5**. Formation of discontinuous precipitates may lead to failure through intergranular brittleness [121] and at prolonged aging lead to decomposition especially in C containing alloys [122].

To gain a better understanding of the effect of aging temperature on strength, aging at temperatures of 883 K, 903 K, and 923 K, based on the Lifshitz–Slyozov–Wagner (LSW) model, was simulated. Unlike other alloying systems, aging was found to cause severe negative drops in the mechanical properties. However, the Heatvar alloy showed a minimal

reduction of only 2 HRC after being aged, indicating excellent high-temperature properties. In contrast to other LPBF alloy systems previously studied, such as MS1, which produces a soft austenite phase at elevated aging temperatures due to over-aging, Heatvar exhibited superior mechanical stability at a temperature of 893 K for 100 h (**Fig. 2.17**). Beyond this optimal temperature, Orowan looping became more pronounced as the precipitates coarsened. Previous studies on wrought H13 and MS1 showed significant drops in the HRC values [93], demonstrating the Heatvar alloy's superior mechanical properties. However, this study did not address the combined method of applying solution treatment and DAG to optimize the alloy's ductility-versus-strength ratio. Further research is needed to analyze the effect of solution treatment before aging and the results of aging time at different temperatures, as in the previous work by Bai et al. [116]. To maximize the mechanical and functional qualities of alloys, the development of an optimal HT method is crucial. Different heat treatment procedures with the same chemical composition may produce different reactions under similar chemical compositions.

Table 2.5. Change of mechanical properties with heat treatment [93].

Sample Treatment	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
As-printed	1165 ± 12	1190 ± 16	4 ± 1
Aged	1660 ± 18	1980 ± 20	2 ± 1
Solution-treated	1156 ± 12	1369 ± 18	15 ± 1

An LPBF-EOS SS CX alloy and a wrought alloy belonging to the PH13-8Mo was reported [123] to possess a martensitic structure with a distribution of austenite because of austenite stabilizing elements (Mo, Ni, and Mn). Moreover, these elements decreased the martensite finish temperature. The fraction of retained austenite in the LPBF and the wrought alloy was 6% and 3%, respectively. Due to limited energy and time for the

austenite-stabilizing elements to diffuse out of the austenite phase during the cooling process, significant elemental segregation was observed [120].

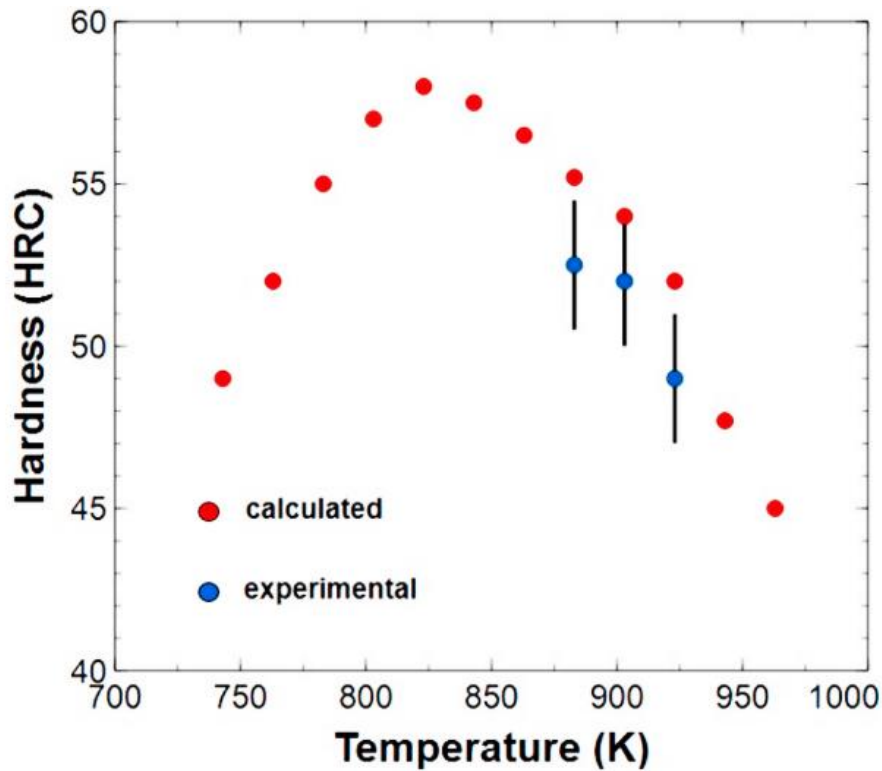


Figure 2.17. Change of hardness with aging time [93].

A similar solution treatment protocol was applied to the hybrid materials [123], consisting of heating at 850 °C for 30 min (**Fig. 2.18**). A peak hardness of 50 HRC was achieved for both alloys after 2 h of aging. In this study, the LPBF alloy precipitated a larger (100 nm) NiAl phase and less reverted austenite (1%) compared to the wrought counterpart, which had a smaller (<40 nm) NiAl phase and approximately 5% retained austenite. This presence reverted of reverted austenite could be attributed to the influence of nitrogen gas during powder atomization, as reported by Meridith et al. [124] in their work on PH17-4. The LPBF alloy exhibited a superior yield strength of 1700 GPa compared to the wrought alloy's yield strength of 1600 GPa. In contrast, the wrought alloy exhibited reduced

ductility of approximately 4% compared to 10% for the LPBF alloy. This highlights the significance of investigating the substantial retainment of austenite, as suggested by Troitter et al. [123], to better understand the underlying factors contributing to the mechanical properties of these alloys.

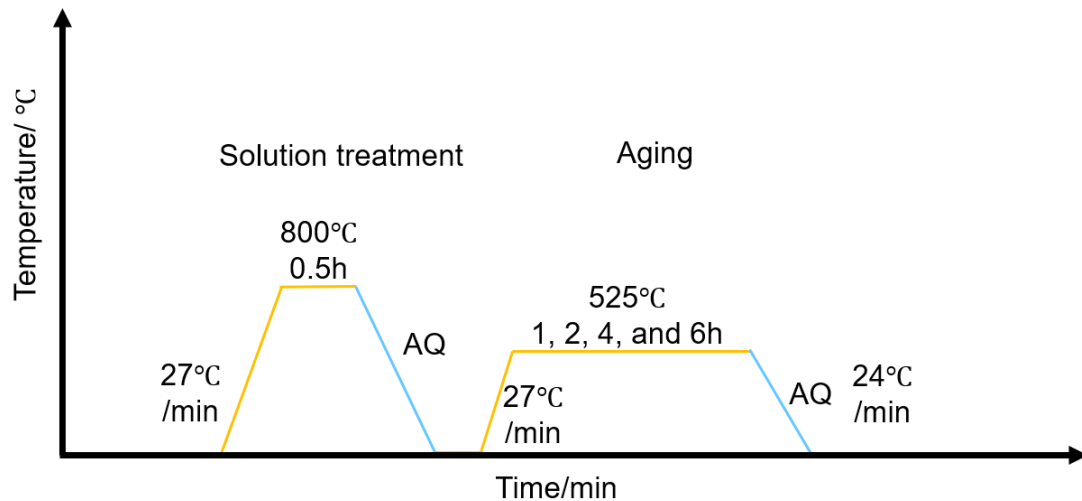


Figure 2.18. Heat treatment schedule for LPBF-EOS SS CX and PH13-8 wrought alloy [123].

LPBF metals produce fine grain structures that strengthen metals due to a high grain boundary following the Hall–Petch mechanism, hence a higher yield strength [16]. Based on these observations, an ideal balance of precipitate size, reverted austenite, temperature, and aging time requires more understanding to tailor specific properties. Both alloys exhibited different microstructures and sensitivities to the applied heat treatments despite having similar compositions. The joining of dissimilar metals poses a significant challenge in the design of heat treatment schedules, as eluded earlier [34,46]. Dissimilar joints of maraging steel (250) and PH 13-8Mo studied by Murthy et al. [50] exhibited the best

strength of 1528 MPa, after a post-weld HT of 510 °C for 4 h followed by air cooling, attributed to the presence of Ni₃Ti, Ni₃Mo, and β-NiAl precipitates.

The presence of precipitates in the maraging steel was not systematically quantified but inferred from the observed improvement in hardness after heat treatment, which peaked at 600HVN. The PH 13-8 Mo HAZ (where the failure occurred) exhibited a mixture of martensite and reverted austenite, which lacked NiAl precipitates. The absence of precipitates enhanced the failure through eased dislocation motion [97]. The low hardness (450 HV) and potential elemental segregation were identified as leading factors contributing to the challenge of joining dissimilar metals. A similar alloy system was modified by performing different welding techniques, such as GTCAW and Laser Beam Welding (LBW), which have similarities to LPBF and Electron Beam Welding (EBW) [125]. All joints using the different welding techniques demonstrated high tensile strengths above 1500 MPa after heat treatment [125]. The materials yielded the greatest strength of 1649 MPa and 1656 MPa for LBW and EBW, respectively, due to the presence of uniformly distributed Ni₃Ti, Ni₃Mo, and -NiAl precipitates (510 °C for 4 h). In contrast, a lower heat treatment of 485 °C for 3.5 h (with air cooling) resulted in lower strength measurements of 1490 MPa (GTCA), 1600 MPa (LBW), and 1610 MPa (EBW). The E/LBW failure occurred at the weld zone, with a fine dendritic structure. The failure was attributed to Ti and Mo elemental segregation near the interdendritic borders [120,125]. According to this study, an increase in aging time and temperature increases the mechanical characteristics of the parent alloys, despite failure in the joined section. This work did not determine the upper heat treatment limit to obtain the highest mechanical properties. In

addition, solution treatment can be applied post-welding to minimize elemental segregation at the interface.

2.7. Conclusions

In this review, we presented the use of hybrid additive manufacturing and its progress over the past decade. The term hybrid additive manufacturing refers to processes that are used in conjunction with an additive manufacturing process in situ or post-printing to improve the quality of the final product or the production process. This review identified several processes as given in the literature with a focus on the use of AM as a joining technique for metals. Direct joining, gradient joining, and inter-layer joining were identified as the main joining techniques that produce interfaces with unique chemistry and mechanical properties. Poor bonding at the interface was a major concern, and the robustness of the interface was found to be governed by alloy compatibility, interface reaction products, and thermal conductivity. Solubility between metals is crucial to avoid segregation. Furthermore, mismatched thermal coefficients and chemical compositions result in accumulated stresses and detrimental phases at the interface, respectively. These parameters contribute to the lack of fusion in joints and premature failure. Hybrid AM was identified as an alternative low-cost production option that can be achieved in the AM of maraging steels by printing on a low-cost substrate material. This reduces the quantity of printable material. In addition, dissimilar metal AM offers customized functionality to meet specific design requirements. However, careful design of the post-processing heat treatment is necessary to ensure good metallurgical bonding at the interface, as well as for the individual materials. Based on the literature surveyed, there is a lack of a systematic way of optimizing the heat treatments of the dissimilar metals and their interfaces. The heat

treatments are specific for individual materials and often compromise the strength of the interface. The growth of hybrid additive manufacturing for steels depends on the development of competent and specific heat treatment protocols, which are to meet the growing demand for multimaterials in industry. It is evident that multimaterials are crucial for productivity and efficiency, and hybrid AM is a promising field. There is a need for more understanding of the potential afforded by hybrid additive manufacturing.

2.8. Summary of Literature Review

In this review, the term ‘hybrid’ has been thoroughly explored, drawing from existing literature to establish a clear definition. Specifically, within the context of this work, hybrid manufacturing is characterized by the integration of additive manufacturing with traditional manufacturing processes. This approach utilizes AM as a cost-effective joining technique, particularly advantageous when applied to low-cost substrates.

The review explores the complexities associated with joining dissimilar materials, addressing issues such as alloy compatibility, the formation of interface reaction products, and the challenge of managing thermal conductivity disparities at the joining interface. To ensure a robust interface, it is essential to develop a comprehensive understanding of the behaviour of the materials being joined, aiming to minimize defects arising from factors like porosity and undesirable phase transformations.

Furthermore, the review highlights the role of solute variations across the interface, which can lead to weak metallurgical bonding due to elemental segregation. This phenomenon is attributed to the high cooling rates during deposition, which can alter the primary solidification phase. Additionally, micro segregation during solidification contributes to chemical inhomogeneity, leading to locally varying phase stabilities. Thermal conductivity

mismatches also pose a significant hurdle in hybrid joining processes, as heat dissipation occurs at different rates in materials with varying conductivities. This imbalance can result in insufficient fusion at the interface and the accumulation of residual stresses, ultimately compromising the integrity of the joint.

Despite the promise of hybrid AM as a viable low-cost production technique, a proper understanding of phase evolutions and stability at the interface is crucial in achieving sound metallurgical bonds. The literature review highlights that many attempts to produce hybrid materials have yielded interfaces unsuitable for industrial applications, even after undergoing post-heat treatment modifications. While heat treatments have been employed to address interface issues, this review reveals that a substantial number of failures persist, highlighting the ongoing challenges in achieving reliable hybrid joints and emphasizing the need to address these challenges effectively.

Given the groundwork laid out regarding hybrid manufacturing, the focus of this study now shifts towards exploring its application to LPBF-M789, a novel maraging steel tailor-made for additive manufacturing. Developed by the voestalpine group, M789 steel is uniquely formulated for employment in the LPBF AM process [4]. It has superior mechanical properties, with an ultimate tensile strength of approximately 1800 MPa (after post-heat treatment) [4,126]. To the best of the author's knowledge, no prior research has been conducted on the application of M789 in hybrid manufacturing, despite some earlier investigations into the properties of LPBF-M789. Comparative studies between M789 and W722 steels revealed that post-heat treatment of M789 (1000°C for 1hr, aging at 500°C for 3 hrs) and W722 (960°C for 5 hrs, aging at 820°C for 2hrs) resulted in a comparable hardness of above 52 HRC for both alloys. However, M789 exhibited a lower tensile

strength of approximately 1800 MPa compared to 2000 MPa for W722 steel. Toughness for the new alloy, as measured by the Charpy test, was reported to be 17 J, i.e., 3 J more than W722. The corrosion resistance of M789 was measured against N700 steel (also known as PH17-4), and it was reported that its pitting potential had a high value of 750 mV compared to 450mV for N700 due to the presence of chromium (12.2wt.%) [4]. These results revealed that this new alloy has potential use in conditions that require the mechanical properties of W722 and corrosion resistance of N700 steel. A typical use would be material for plastic injection molds and die inserts where extended tool life is a priority.

As highlighted in the literature review, different attempts have been made to form hybrid materials via additive manufacturing, with detailed discussions on the metallurgical challenges they present. However, according to the literature reviewed, no previous work has explored the use of M789 in hybrid combinations. Thus, this study represents the first attempt to integrate LPBF-M789 steel into a hybrid system with conventional N709. N709 has an inherent combination of high strength of 1482 MPa, and a toughness of 41 J (Charpy V notch) at H1000 conditions [127], and stress corrosion resistance (up to 300°C), which makes it useful for applications in die inserts, high strength bolts, and aircraft landing gears [128,129]. The presence of Ni and Al in this steel grade has been reported to promote the formation of β -NiAl precipitates enclosed in lath martensite at aging temperatures between 450°C-575°C [130]. Even at temperatures exceeding 575°C for 4 hours of aging, the alloy has been reported to maintain a coherent phase with some reverted austenite [130], which is an indication of good resistance to over-aging [131,132]. Guided by past work on AM hybrid, N709 is used. The similarities in properties between M789

and N709 steels make them promising candidates for hybrid fabrication, as they mitigate alloy incompatibilities and thermal conductivity disparities.

Central to this study is the necessity for a comprehensive understanding of the interactions between M789 and N709 steels, with the possibility of modifying hybrid properties through heat treatment. **Table 2.6** summarizes the individual properties of M789 and N709.

The M789 alloy represents a novel material whose properties are yet to be fully explored, especially in the context of its application within a hybrid manufacturing process. There exists a significant opportunity to achieve cost-effective and efficient production of M789, aligning with the industry's demands. Leveraging the geometrical capabilities of LPBF, together with the utilization of a relatively inexpensive base material N709, offers a viable and cost-effective pathway for production.

This study undergoes an in-depth study of the first application of M789-N709 in a hybrid process with a particular emphasis on establishing the mechanical properties and chemistry of M789. The characteristics of the interface are explored for this emerging new hybrid providing an opportunity for lowered cost of production and its expanded use in industry. Furthermore, this study investigates an in-depth exploration of the properties of LPBF-M789 under conditions of high temperature and dynamic straining, aiming to enhance understanding and predictability for real-world applications.

Knowledge obtained from the M789-N709 hybrid research contributes to understanding the processing-structure-property relationship that arises due to the complex solidification and chemical changes at the interface. This will aid in the development of suitable heat treatment procedures, material models, and process design of M789 hybrid materials. Moreover, this study contributes to validating the viability of this novel hybrid material,

thereby broadening the use of M789 across diverse industries such as tooling, aerospace, automotive, and energy.

Table 2.6. Properties of M789 and N709 steels.

	Alloy - M789	Alloy - N709
Description	Mechanical properties are similar to M300 and corrosion resistance of PH 17-4	PH 13-8 Cr-Mo family
Alloying Elements	< 0.02 C, 12.2 Cr, 1.00 Mo, 10.00 Ni, 1.00 Ti, and 0.60 Al. (all in wt.%)	0.03 C, 12.7 Cr, 2.2 Mo, 8.1 Ni, 1.1 Al, < = 0.08 Si, and Mn. (all in wt.%)
Processing technique	LPBF	Wrought, and hardened
Peak Tensile Strength	1840 MPa	1541 MPa
Peak Yield Strength	1714 MPa	1603 MPa
Elongation	11 %	13%
Toughness	13 J	42 J
Hardness	52 HRC	45 HRC
Heat Treatment	ST at 1000 °C for 1hour, and Aging at 500 °C for 2 hours	ST at 925 °C for 1 hour, and Aging at 500 °C for 4 hours
Strengthening Mechanism	Precipitation Hardening	Precipitation Hardening
PREN value	175.5	149.56
Melting Point	1457.59	1482.41
Density (r.t.p)	7.78g/cm ³	7.75g/cm ³
Crystal Structure (r.t.p)	BCT	BCT
Thermal conductivity (r.t.p)	41.71W/(m*K)	47.73 W/(m*K)

2.9 References

- [1] P. Cardullo and R. Kitchin, ‘Smart urbanism and smart citizenship: The neoliberal logic of “citizen-focused” smart cities in Europe’, *Environ. Plan. C Polit. Space*, vol. 37, no. 5, pp. 813–830, Aug. 2019, doi: 10.1177/0263774X18806508.
- [2] F. Klocke, K. Arntz, M. Teli, K. Winands, M. Wegener, and S. Oliari, ‘State-of-the-art Laser Additive Manufacturing for Hot-work Tool Steels’, *Procedia CIRP*, vol. 63, pp. 58–63, 2017, doi: 10.1016/j.procir.2017.03.073.

- [3] S. Parupelli and S. Desai, 'A Comprehensive Review of Additive Manufacturing (3D Printing): Processes, Applications and Future Potential', *Am. J. Appl. Sci.*, vol. 16, no. 8, Sep. 2019, Accessed: Jun. 08, 2021. [Online]. Available: <https://par.nsf.gov/biblio/10124832-comprehensive-review-additive-manufacturing-printing-processes-applications-future-potential>
- [4] Y. Tian, R. Palad, and C. Aranas, 'Microstructural evolution and mechanical properties of a newly designed steel fabricated by laser powder bed fusion', *Addit. Manuf.*, vol. 36, p. 101495, Dec. 2020, doi: 10.1016/j.addma.2020.101495.
- [5] M. Rafiee, R. D. Farahani, and D. Therriault, 'Multi-Material 3D and 4D Printing: A Survey', *Adv. Sci.*, vol. 7, no. 12, p. 1902307, Jun. 2020, doi: 10.1002/advs.201902307.
- [6] K. Gupta, N. K. Jain, and R. F. Laubscher, 'Overview of Hybrid Machining Processes', in *Hybrid Machining Processes: Perspectives on Machining and Finishing*, K. Gupta, N. K. Jain, and R. F. Laubscher, Eds., in *SpringerBriefs in Applied Sciences and Technology.*, Cham: Springer International Publishing, 2016, pp. 1–7. doi: 10.1007/978-3-319-25922-2_1.
- [7] A. Seidel et al., 'Added value by hybrid additive manufacturing and advanced manufacturing approaches', *J. Laser Appl.*, vol. 30, no. 3, p. 032301, Aug. 2018, doi: 10.2351/1.5040632.
- [8] S. Luisa, N. Contuzzi, A. Angelastro, and A. Domenico, 'Capabilities and Performances of the Selective Laser Melting Process', in *New Trends in*

- Technologies: Devices, Computer, Communication and Industrial Systems, M. Joo, Ed., Sciyo, 2010. doi: 10.5772/10432.
- [9] ‘Introduction to FDM 3D printing’, Hubs. Accessed: Jun. 08, 2021. [Online]. Available: <https://www.hubs.com/knowledge-base/introduction-fdm-3d-printing/>
- [10] B. Blakey-Milner, ‘Metal additive manufacturing in aerospace: A review’, p. 33, 2021.
- [11] T. Petrat, B. Graf, A. Gumenyuk, and M. Rethmeier, ‘Laser Metal Deposition as Repair Technology for a Gas Turbine Burner Made of Inconel 718’, *Phys. Procedia*, vol. 83, pp. 761–768, 2016, doi: 10.1016/j.phpro.2016.08.078.
- [12] K. S. Prakash, T. Nancharaih, and V. V. S. Rao, ‘Additive Manufacturing Techniques in Manufacturing -An Overview’, *Mater. Today Proc.*, vol. 5, no. 2, pp. 3873–3882, 2018, doi: 10.1016/j.matpr.2017.11.642.
- [13] V. V. Popov and A. Fleisher, ‘Hybrid additive manufacturing of steels and alloys’, *Manuf. Rev.*, vol. 7, p. 6, 2020, doi: 10.1051/mfreview/2020005.
- [14] S. Jayanath and A. Achuthan, ‘A computationally efficient hybrid model for simulating the additive manufacturing process of metals’, *Int. J. Mech. Sci.*, vol. 160, pp. 255–269, Sep. 2019, doi: 10.1016/j.ijmecsci.2019.06.007.
- [15] A. Yadollahi and N. Shamsaei, ‘Additive manufacturing of fatigue resistant materials: Challenges and opportunities’, *Int. J. Fatigue*, vol. 98, pp. 14–31, May 2017, doi: 10.1016/j.ijfatigue.2017.01.001.

- [16] T. DebRoy et al., ‘Additive manufacturing of metallic components – Process, structure and properties’, *Prog. Mater. Sci.*, vol. 92, pp. 112–224, Mar. 2018, doi: 10.1016/j.pmatsci.2017.10.001.
- [17] K. Darvish, Z. W. Chen, and T. Pasang, ‘Reducing lack of fusion during selective laser melting of CoCrMo alloy: Effect of laser power on geometrical features of tracks’, *Mater. Des.*, vol. 112, pp. 357–366, Dec. 2016, doi: 10.1016/j.matdes.2016.09.086.
- [18] A. J. Sterling, B. Torries, N. Shamsaei, S. M. Thompson, and D. W. Seely, ‘Fatigue behavior and failure mechanisms of direct laser deposited Ti–6Al–4V’, *Mater. Sci. Eng. A*, vol. 655, pp. 100–112, Feb. 2016, doi: 10.1016/j.msea.2015.12.026.
- [19] A. S. Johnson, S. Shuai, N. Shamsaei, S. M. Thompson, and L. Bian, ‘Fatigue Behavior and Failure Mechanisms of Direct Laser Deposited Inconel 718’, p. 13.
- [20] W. Du, Q. Bai, and B. Zhang, ‘A Novel Method for Additive/Subtractive Hybrid Manufacturing of Metallic Parts’, *Procedia Manuf.*, vol. 5, pp. 1018–1030, 2016, doi: 10.1016/j.promfg.2016.08.067.
- [21] C. Du et al., ‘Pore defects in Laser Powder Bed Fusion: Formation mechanism, control method, and perspectives’, *J. Alloys Compd.*, vol. 944, p. 169215, May 2023, doi: 10.1016/j.jallcom.2023.169215.
- [22] Y. Zhao, C. Du, P. Wang, W. Meng, and C. Li, ‘The Mechanism of In-Situ Laser Polishing and Its Effect on the Surface Quality of Nickel-Based Alloy Fabricated by Selective Laser Melting’, *Metals*, vol. 12, no. 5, p. 778, Apr. 2022, doi: 10.3390/met12050778.

- [23] A. Reichardt et al., ‘Advances in additive manufacturing of metal-based functionally graded materials’, *Int. Mater. Rev.*, vol. 66, no. 1, pp. 1–29, Jan. 2021, doi: 10.1080/09506608.2019.1709354.
- [24] I. Tomashchuk, D. Grevey, and P. Sallamand, ‘Dissimilar laser welding of AISI 316L stainless steel to Ti6–Al4–6V alloy via pure vanadium interlayer’, *Mater. Sci. Eng. A*, vol. 622, pp. 37–45, Jan. 2015, doi: 10.1016/j.msea.2014.10.084.
- [25] G. Manogharan, R. A. Wysk, and O. L. A. Harrysson, ‘Additive manufacturing–integrated hybrid manufacturing and subtractive processes: economic model and analysis’, *Int. J. Comput. Integr. Manuf.*, vol. 29, no. 5, pp. 473–488, May 2016, doi: 10.1080/0951192X.2015.1067920.
- [26] ‘Committee F42 on Additive Manufacturing Technologies’. Accessed: Jun. 05, 2023.[Online].Available:<https://www.astm.org/get-involved/technical-committees/committee-f42>
- [27] F. Caiazzo et al., ‘Laser Beam Welding of a Ti–6Al–4V Support Flange for Buy-to-Fly Reduction’, *Metals*, vol. 7, no. 5, p. 183, May 2017, doi: 10.3390/met7050183.
- [28] M. Merklein, D. Junker, A. Schaub, and F. Neubauer, ‘Hybrid Additive Manufacturing Technologies – An Analysis Regarding Potentials and Applications’, *Phys. Procedia*, vol. 83, pp. 549–559, 2016, doi: 10.1016/j.phpro.2016.08.057.
- [29] K. P. Karunakaran, S. Suryakumar, V. Pushpa, and S. Akula, ‘Low cost integration of additive and subtractive processes for hybrid layered manufacturing’, *Robot. Comput.-Integr. Manuf.*, vol. 26, no. 5, pp. 490–499, Oct. 2010, doi: 10.1016/j.rcim.2010.03.008.

- [30] G. Manogharan, R. Wysk, O. Harrysson, and R. Aman, 'AIMS – A Metal Additive-hybrid Manufacturing System: System Architecture and Attributes', *Procedia Manuf.*, vol. 1, pp. 273–286, 2015, doi: 10.1016/j.promfg.2015.09.021.
- [31] Z. C. Oter et al., 'Benefits of laser beam based additive manufacturing in die production', *Optik*, vol. 176, pp. 175–184, Jan. 2019, doi: 10.1016/j.ijleo.2018.09.079.
- [32] R. L. Klueh and J. F. King, 'Austenitic Stainless Steel-Ferritic Steel Weld Joint Failures', p. 10.
- [33] T.-K. Yeh, G.-R. Huang, M.-Y. Wang, and C.-H. Tsai, 'Stress corrosion cracking in dissimilar metal welds with 304L stainless steel and Alloy 82 in high temperature water', *Prog. Nucl. Energy*, vol. 63, pp. 7–11, Mar. 2013, doi: 10.1016/j.pnucene.2012.10.001.
- [34] H. Azizi et al., 'Metallurgical and mechanical assessment of hybrid additively-manufactured maraging tool steels via selective laser melting', *Addit. Manuf.*, vol. 27, pp. 389–397, May 2019, doi: 10.1016/j.addma.2019.03.025.
- [35] Y. Wang, H. Tang, Y. Fang, and H. Wang, 'Microstructure and mechanical properties of hybrid fabricated 1Cr12Ni2WMoVNB steel by laser melting deposition', *Chin. J. Aeronaut.*, vol. 26, no. 2, pp. 481–486, Apr. 2013, doi: 10.1016/j.cja.2013.02.027.
- [36] Y. Zhu, J. Li, X. Tian, H. Wang, and D. Liu, 'Microstructure and mechanical properties of hybrid fabricated Ti–6.5Al–3.5Mo–1.5Zr–0.3Si titanium alloy by laser

- additive manufacturing', *Mater. Sci. Eng. A*, vol. 607, pp. 427–434, Jun. 2014, doi: 10.1016/j.msea.2014.04.019.
- [37] Y. Li, Q. Han, I. Horváth, and G. Zhang, 'Repairing surface defects of metal parts by groove machining and wire + arc based filling', *J. Mater. Process. Technol.*, vol. 274, p. 116268, Dec. 2019, doi: 10.1016/j.jmatprotec.2019.116268.
- [38] M. Pranieicz, T. Kurfess, and C. Saldana, 'Adaptive geometry transformation and repair for hybrid manufacturing', *Procedia Manuf.*, vol. 26, pp. 228–236, 2018, doi: 10.1016/j.promfg.2018.07.031.
- [39] Y.-H. Chueh, C. Wei, X. Zhang, and L. Li, 'Integrated laser-based powder bed fusion and fused filament fabrication for three-dimensional printing of hybrid metal/polymer objects', *Addit. Manuf.*, vol. 31, p. 100928, Jan. 2020, doi: 10.1016/j.addma.2019.100928.
- [40] R. Falck, S. M. Goushegir, J. F. dos Santos, and S. T. Amancio-Filho, 'AddJoining: A novel additive manufacturing approach for layered metal-polymer hybrid structures', *Mater. Lett.*, vol. 217, pp. 211–214, Apr. 2018, doi: 10.1016/j.matlet.2018.01.021.
- [41] X. Shi et al., 'Selective laser melting-wire arc additive manufacturing hybrid fabrication of Ti-6Al-4V alloy: Microstructure and mechanical properties', *Mater. Sci. Eng. A*, vol. 684, pp. 196–204, Jan. 2017, doi: 10.1016/j.msea.2016.12.065.
- [42] S. L. Sing, L. P. Lam, D. Q. Zhang, Z. H. Liu, and C. K. Chua, 'Interfacial characterization of SLM parts in multi-material processing: Intermetallic phase

- formation between AlSi10Mg and C18400 copper alloy’, *Mater. Charact.*, vol. 107, pp. 220–227, Sep. 2015, doi: 10.1016/j.matchar.2015.07.007.
- [43] K. Chen et al., ‘Selective laser melting 316L/CuSn10 multi-materials: Processing optimization, interfacial characterization and mechanical property’, *J. Mater. Process. Technol.*, vol. 283, p. 116701, Sep. 2020, doi: 10.1016/j.jmatprotec.2020.116701.
- [44] T. Yamazaki, ‘Development of A Hybrid Multi-tasking Machine Tool: Integration of Additive Manufacturing Technology with CNC Machining’, *Procedia CIRP*, vol. 42, pp. 81–86, 2016, doi: 10.1016/j.procir.2016.02.193.
- [45] L. Li, A. Haghighi, and Y. Yang, ‘A novel 6-axis hybrid additive-subtractive manufacturing process: Design and case studies’, *J. Manuf. Process.*, vol. 33, pp. 150–160, Jun. 2018, doi: 10.1016/j.jmapro.2018.05.008.
- [46] S. Shakerin, M. Sanjari, B. S. Amirkhiz, and M. Mohammadi, ‘Interface engineering of additively manufactured maraging steel-H13 bimetallic structures’, *Mater. Charact.*, vol. 170, p. 110728, Dec. 2020, doi: 10.1016/j.matchar.2020.110728.
- [47] Y. Tian, K. Nyamuchiwa, K. Chadha, Y. He, and C. Aranas, ‘Laser powder bed fusion of M789 maraging steel on Cr–Mo N709 steel: Microstructure, texture, and mechanical properties’, *Mater. Sci. Eng. A*, vol. 839, p. 142827, Apr. 2022, doi: 10.1016/j.msea.2022.142827.
- [48] H.-C. Chen, A. J. Pinkerton, L. Li, Z. Liu, and A. T. Mistry, ‘Gap-free fibre laser welding of Zn-coated steel on Al alloy for light-weight automotive applications’,

- Mater. Des., vol. 32, no. 2, pp. 495–504, Feb. 2011, doi: 10.1016/j.matdes.2010.08.034.
- [49] H.-C. Chen, A. J. Pinkerton, and L. Li, ‘Fibre laser welding of dissimilar alloys of Ti-6Al-4V and Inconel 718 for aerospace applications’, *Int. J. Adv. Manuf. Technol.*, vol. 52, no. 9–12, pp. 977–987, Feb. 2011, doi: 10.1007/s00170-010-2791-3.
- [50] C. V. S. Murthy, A. Gopala Krishna, and G. M. Reddy, ‘Microstructure and mechanical properties of similar and dissimilar metal gas tungsten constricted arc welds: Maraging steel to 13-8 Mo stainless steel’, *Def. Technol.*, vol. 15, no. 1, pp. 111–121, Feb. 2019, doi: 10.1016/j.dt.2018.04.005.
- [51] F. F. Conde et al., ‘Effect of thermal cycling and aging stages on the microstructure and bending strength of a selective laser melted 300-grade maraging steel’, *Mater. Sci. Eng. A*, vol. 758, pp. 192–201, Jun. 2019, doi: 10.1016/j.msea.2019.03.129.
- [52] L. Yan, Y. Chen, and F. Liou, ‘Additive manufacturing of functionally graded metallic materials using laser metal deposition’, *Addit. Manuf.*, vol. 31, p. 100901, Jan. 2020, doi: 10.1016/j.addma.2019.100901.
- [53] Z. H. Liu, D. Q. Zhang, S. L. Sing, C. K. Chua, and L. E. Loh, ‘Interfacial characterization of SLM parts in multi-material processing: Metallurgical diffusion between 316L stainless steel and C18400 copper alloy’, *Mater. Charact.*, vol. 94, pp. 116–125, Aug. 2014, doi: 10.1016/j.matchar.2014.05.001.
- [54] F. Hengsbach et al., ‘Inline additively manufactured functionally graded multi-materials: microstructural and mechanical characterization of 316L parts with H13

- layers', *Prog. Addit. Manuf.*, vol. 3, no. 4, pp. 221–231, Dec. 2018, doi: 10.1007/s40964-018-0044-4.
- [55] K. Nyamuchiwa, Y. Tian, K. Chadha, and C. Aranas, 'Laser Powder Bed Fusion of M789 Steel on Wrought N709 Steel Substrate', in *Proceedings of the 61st Conference of Metallurgists, COM 2022*, Cham: Springer International Publishing, 2023, pp. 9–14.
- [56] J. Samei et al., 'A hybrid additively manufactured martensitic-maraging stainless steel with superior strength and corrosion resistance for plastic injection molding dies', *Addit. Manuf.*, vol. 45, p. 102068, Sep. 2021, doi: 10.1016/j.addma.2021.102068.
- [57] C. Wei, Z. Zhang, D. Cheng, Z. Sun, M. Zhu, and L. Li, 'An overview of laser-based multiple metallic material additive manufacturing: from macro- to micro-scales', *Int. J. Extreme Manuf.*, vol. 3, no. 1, p. 012003, Dec. 2020, doi: 10.1088/2631-7990/abce04.
- [58] B. E. Carroll et al., 'Functionally graded material of 304L stainless steel and inconel 625 fabricated by directed energy deposition: Characterization and thermodynamic modeling', *Acta Mater.*, vol. 108, pp. 46–54, Apr. 2016, doi: 10.1016/j.actamat.2016.02.019.
- [59] L. D. Bobbio et al., 'Additive manufacturing of a functionally graded material from Ti-6Al-4V to Invar: Experimental characterization and thermodynamic calculations', *Acta Mater.*, vol. 127, pp. 133–142, Apr. 2017, doi: 10.1016/j.actamat.2016.12.070.

- [60] I. Tomashchuk, P. Sallamand, N. Belyavina, and M. Pilloz, ‘Evolution of microstructures and mechanical properties during dissimilar electron beam welding of titanium alloy to stainless steel via copper interlayer’, *Mater. Sci. Eng. A*, vol. 585, pp. 114–122, Nov. 2013, doi: 10.1016/j.msea.2013.07.050.
- [61] H. Sabetghadam, A. Z. Hanzaki, and A. Araee, ‘Diffusion bonding of 410 stainless steel to copper using a nickel interlayer’, *Mater. Charact.*, vol. 61, no. 6, pp. 626–634, Jun. 2010, doi: 10.1016/j.matchar.2010.03.006.
- [62] G. Madhusudhan Reddy and P. Venkata Ramana, ‘Role of nickel as an interlayer in dissimilar metal friction welding of maraging steel to low alloy steel’, *J. Mater. Process. Technol.*, vol. 212, no. 1, pp. 66–77, Jan. 2012, doi: 10.1016/j.jmatprotec.2011.08.005.
- [63] H. Sahasrabudhe, R. Harrison, C. Carpenter, and A. Bandyopadhyay, ‘Stainless steel to titanium bimetallic structure using LENSTM’, *Addit. Manuf.*, vol. 5, pp. 1–8, Jan. 2015, doi: 10.1016/j.addma.2014.10.002.
- [64] L. D. Bobbio et al., ‘Characterization of a functionally graded material of Ti-6Al-4V to 304L stainless steel with an intermediate V section’, *J. Alloys Compd.*, vol. 742, pp. 1031–1036, Apr. 2018, doi: 10.1016/j.jallcom.2018.01.156.
- [65] W. Li et al., ‘Fabrication and characterization of a functionally graded material from Ti-6Al-4V to SS316 by laser metal deposition’, *Addit. Manuf.*, vol. 14, pp. 95–104, Mar. 2017, doi: 10.1016/j.addma.2016.12.006.

- [66] B. Onuiké and A. Bandyopadhyay, ‘Additive manufacturing of Inconel 718 – Ti6Al4V bimetallic structures’, *Addit. Manuf.*, vol. 22, pp. 844–851, Aug. 2018, doi: 10.1016/j.addma.2018.06.025.
- [67] M. Zhang, Y. Yang, D. Wang, C. Song, and J. Chen, ‘Microstructure and mechanical properties of CuSn/18Ni300 bimetallic porous structures manufactured by selective laser melting’, *Mater. Des.*, vol. 165, p. 107583, Mar. 2019, doi: 10.1016/j.matdes.2019.107583.
- [68] C. Anstaett, C. Seidel, and G. Reinhart, ‘Fabrication of 3D Multi-material Parts Using Laser-based Powder Bed Fusion’, p. 9.
- [69] C. Wei, L. Li, X. Zhang, and Y.-H. Chueh, ‘3D printing of multiple metallic materials via modified selective laser melting’, *CIRP Ann.*, vol. 67, no. 1, pp. 245–248, 2018, doi: 10.1016/j.cirp.2018.04.096.
- [70] P. Wang et al., ‘Microstructure and mechanical properties of Al-12Si and Al-3.5Cu-1.5Mg-1Si bimetal fabricated by selective laser melting’, *J. Mater. Sci. Technol.*, vol. 36, pp. 18–26, Jan. 2020, doi: 10.1016/j.jmst.2019.03.047.
- [71] K. Wei, X. Zeng, F. Li, M. Liu, and J. Deng, ‘Microstructure and Mechanical Property of Ti-5Al-2.5Sn/Ti-6Al-4V Dissimilar Titanium Alloys Integrally Fabricated by Selective Laser Melting’, *JOM*, vol. 72, no. 3, pp. 1031–1038, Mar. 2020, doi: 10.1007/s11837-019-03988-6.
- [72] K. Nyamuchiwa, Y. Tian, K. Chadha, L. Jiang, T. Dorin, and C. Aranas, ‘Precipitation Behaviour at the Interface of an Additively Manufactured M789–

- N709 Hybrid Alloy’, *Acta Metall. Sin. Engl. Lett.*, May 2023, doi: 10.1007/s40195-023-01558-z.
- [73] V. K. Nadimpalli, T. Dahmen, E. H. Valente, S. Mohanty, and D. B. Pedersen, ‘Multi-material additive manufacturing of steels using laser powder bed fusion’, p. 5.
- [74] M. G. Scaramuccia, A. G. Demir, L. Caprio, O. Tassa, and B. Previtali, ‘Development of processing strategies for multigraded selective laser melting of Ti6Al4V and IN718’, *Powder Technol.*, vol. 367, pp. 376–389, May 2020, doi: 10.1016/j.powtec.2020.04.010.
- [75] C. Wei, Z. Sun, Q. Chen, Z. Liu, and L. Li, ‘Additive Manufacturing of Horizontal and 3D Functionally Graded 316L/Cu10Sn Components via Multiple Material Selective Laser Melting’, *J. Manuf. Sci. Eng.*, vol. 141, no. 8, p. 081014, Aug. 2019, doi: 10.1115/1.4043983.
- [76] A. G. Demir and B. Previtali, ‘Multi-material selective laser melting of Fe/Al-12Si components’, *Manuf. Lett.*, vol. 11, pp. 8–11, Jan. 2017, doi: 10.1016/j.mfglet.2017.01.002.
- [77] H. Sun et al., ‘Selective Laser Melting for Joining Dissimilar Materials: Investigations of Interfacial Characteristics and In Situ Alloying’, *Metall. Mater. Trans. A*, vol. 52, no. 4, pp. 1540–1550, Apr. 2021, doi: 10.1007/s11661-021-06178-9.

- [78] C. Wei, H. Gu, Q. Li, Z. Sun, Z. Liu, and L. Li, ‘Understanding heat transfer and material behavior in additive manufacturing of functionally graded Invar36/Cu10Sn alloys using multi-materials laser powder bed fusion’, *Addit. Manuf.*, 2020.
- [79] C. F. Tey, X. Tan, S. L. Sing, and W. Y. Yeong, ‘Additive manufacturing of multiple materials by selective laser melting: Ti-alloy to stainless steel via a Cu-alloy interlayer’, *Addit. Manuf.*, vol. 31, p. 100970, Jan. 2020, doi: 10.1016/j.addma.2019.100970.
- [80] J. R. Mianroodi, P. Shanthraj, A. Kwiatkowski Da Silva, B. Svendsen, and D. Raabe, ‘Combined modeling and experimental characterization of Mn segregation and spinodal decomposition along dislocation lines in Fe–Mn alloys’, *Acta Mater.*, vol. 251, p. 118873, Jun. 2023, doi: 10.1016/j.actamat.2023.118873.
- [81] S. C. Deevi and V. K. Sikka, ‘Nickel and iron aluminides: an overview on properties, processing, and applications’, *Intermetallics*, vol. 4, no. 5, pp. 357–375, Jan. 1996, doi: 10.1016/0966-9795(95)00056-9.
- [82] A. Basak and S. Das, ‘Epitaxy and Microstructure Evolution in Metal Additive Manufacturing’, *Annu. Rev. Mater. Res.*, vol. 46, no. 1, pp. 125–149, Jul. 2016, doi: 10.1146/annurev-matsci-070115-031728.
- [83] L. E. Murr et al., ‘Microstructures and Properties of 17-4 PH Stainless Steel Fabricated by Selective Laser Melting’, *J. Mater. Res. Technol.*, vol. 1, no. 3, pp. 167–177, Oct. 2012, doi: 10.1016/S2238-7854(12)70029-7.
- [84] H. K. Rafi, D. Pal, N. Patil, T. L. Starr, and B. E. Stucker, ‘Microstructure and Mechanical Behavior of 17-4 Precipitation Hardenable Steel Processed by Selective

- Laser Melting', *J. Mater. Eng. Perform.*, vol. 23, no. 12, pp. 4421–4428, Dec. 2014, doi: 10.1007/s11665-014-1226-y.
- [85] T. LeBrun, T. Nakamoto, K. Horikawa, and H. Kobayashi, 'Effect of retained austenite on subsequent thermal processing and resultant mechanical properties of selective laser melted 17–4 PH stainless steel', *Mater. Des.*, vol. 81, pp. 44–53, Sep. 2015, doi: 10.1016/j.matdes.2015.05.026.
- [86] S. Shakerin, A. Hadadzadeh, B. S. Amirkhiz, S. Shamsdini, J. Li, and M. Mohammadi, 'Additive manufacturing of maraging steel-H13 bimetal using laser powder bed fusion technique', *Addit. Manuf.*, vol. 29, p. 100797, Oct. 2019, doi: 10.1016/j.addma.2019.100797.
- [87] F. Ning and W. Cong, 'Microstructures and mechanical properties of Fe-Cr stainless steel parts fabricated by ultrasonic vibration-assisted laser engineered net shaping process', *Mater. Lett.*, vol. 179, pp. 61–64, Sep. 2016, doi: 10.1016/j.matlet.2016.05.055.
- [88] A. Yadollahi, N. Shamsaei, S. M. Thompson, A. Elwany, and L. Bian, 'Mechanical and Microstructural Properties of Selective Laser Melted 17-4 PH Stainless Steel', in *Volume 2A: Advanced Manufacturing*, Houston, Texas, USA: American Society of Mechanical Engineers, Nov. 2015, p. V02AT02A014. doi: 10.1115/IMECE2015-52362.
- [89] J.-H. Wu and C.-K. Lin, 'Influence of high temperature exposure on the mechanical behaviour and microstructure of 17-4 PH stainless steel', p. 7.

- [90] E. Cyr, H. Asgari, S. Shamsdini, M. Purdy, K. Hosseinkhani, and M. Mohammadi, 'Fracture behaviour of additively manufactured MS1-H13 hybrid hard steels', *Mater. Lett.*, vol. 212, pp. 174–177, Feb. 2018, doi: 10.1016/j.matlet.2017.10.097.
- [91] S. Dehgahi et al., 'Texture evolution during high strain-rate compressive loading of maraging steels produced by laser powder bed fusion', *Mater. Charact.*, p. 111266, Jun. 2021, doi: 10.1016/j.matchar.2021.111266.
- [92] E. I. Galindo-Nava, W. M. Rainforth, and P. E. J. Rivera-Díaz-del-Castillo, 'Predicting microstructure and strength of maraging steels: Elemental optimization', *Acta Mater.*, vol. 117, pp. 270–285, Sep. 2016, doi: 10.1016/j.actamat.2016.07.020.
- [93] Y. Tian, K. Chadha, S. H. Kim, and C. Aranas, 'Strengthening mechanisms in a heatvar hot work tool steel fabricated by laser powder bed fusion', *Mater. Sci. Eng. A*, vol. 805, p. 140801, Feb. 2021, doi: 10.1016/j.msea.2021.140801.
- [94] E. I. Galindo-Nava and P. E. J. Rivera-Díaz-del-Castillo, 'A model for the microstructure behaviour and strength evolution in lath martensite', *Acta Mater.*, vol. 98, pp. 81–93, Oct. 2015, doi: 10.1016/j.actamat.2015.07.018.
- [95] M. Niu, G. Zhou, W. Wang, M. B. Shahzad, Y. Shan, and K. Yang, 'Precipitate evolution and strengthening behaviour during aging process in a 2.5 GPa grade maraging steel', *Acta Mater.*, vol. 179, pp. 296–307, Oct. 2019, doi: 10.1016/j.actamat.2019.08.042.
- [96] E. I. Galindo-Nava and P. E. J. Rivera-Díaz-del-Castillo, 'Understanding the factors controlling the hardness in martensitic steels', *Scr. Mater.*, vol. 110, pp. 96–100, Jan. 2016, doi: 10.1016/j.scriptamat.2015.08.010.

- [97] R. Schnitzer et al., 'Influence of reverted austenite on static and dynamic mechanical properties of a PH 13-8 Mo maraging steel', *Mater. Sci. Eng. A*, vol. 527, no. 7–8, pp. 2065–2070, Mar. 2010, doi: 10.1016/j.msea.2009.11.046.
- [98] U. K. Viswanathan, G. K. Dey, and V. Sethumadhavan, 'Effects of austenite reversion during overageing on the mechanical properties of 18 Ni (350) maraging steel', *Mater. Sci. Eng. A*, vol. 398, no. 1–2, pp. 367–372, May 2005, doi: 10.1016/j.msea.2005.03.074.
- [99] B. Kim, E. Boucard, T. Sourmail, D. San Martín, N. Gey, and P. E. J. Rivera-Díaz-del-Castillo, 'The influence of silicon in tempered martensite: Understanding the microstructure–properties relationship in 0.5–0.6wt.% C steels', *Acta Mater.*, vol. 68, pp. 169–178, Apr. 2014, doi: 10.1016/j.actamat.2014.01.039.
- [100] E. (Eckhard) Nembach, *Particle strengthening of metals and alloys*. New York: Wiley, 1997. [Online]. Available: <http://catdir.loc.gov/catdir/toc/onix04/96014932.html>
- [101] G. S. Ansell and F. V. Lenel, 'Criteria for yielding of dispersion-strengthened alloys', *Acta Metall.*, vol. 8, no. 9, pp. 612–616, Sep. 1960, doi: 10.1016/0001-6160(60)90015-8.
- [102] V. K. Vasudevan, S. J. Kim, and C. M. Wayman, 'Precipitation reactions and strengthening behaviour in 18 Wt Pct nickel maraging steels', *Metall. Trans. A*, vol. 21, no. 10, pp. 2655–2668, Oct. 1990, doi: 10.1007/BF02646061.
- [103] A. J. Ardell, 'Precipitation Hardening', *Trans A*, 1985.

- [104] T. H. Simm et al., ‘A SANS and APT study of precipitate evolution and strengthening in a maraging steel’, *Mater. Sci. Eng. A*, vol. 702, pp. 414–424, Aug. 2017, doi: 10.1016/j.msea.2017.07.013.
- [105] R. Schnitzer, S. Zinner, and H. Leitner, ‘Modeling of the yield strength of a stainless maraging steel’, *Scr. Mater.*, vol. 62, no. 5, pp. 286–289, Mar. 2010, doi: 10.1016/j.scriptamat.2009.11.020.
- [106] T. Narita, S. Ukai, S. Ohtsuka, and M. Inoue, ‘Effect of tungsten addition on microstructure and high temperature strength of 9CrODS ferritic steel’, *J. Nucl. Mater.*, vol. 417, no. 1–3, pp. 158–161, Oct. 2011, doi: 10.1016/j.jnucmat.2011.01.060.
- [107] H. Sieurin, J. Zander, and R. Sandström, ‘Modelling solid solution hardening in stainless steels’, *Mater. Sci. Eng. A*, vol. 415, no. 1–2, pp. 66–71, Jan. 2006, doi: 10.1016/j.msea.2005.09.031.
- [108] S. Zeisl, N. Van Steenberge, and R. Schnitzer, ‘Strengthening effect of NiAl and Ni₃Ti precipitates in Co-free maraging steels’, *J. Mater. Sci.*, vol. 58, no. 16, pp. 7149–7160, Apr. 2023, doi: 10.1007/s10853-023-08472-w.
- [109] A. Hadadzadeh, A. Shahriari, B. S. Amirkhiz, J. Li, and M. Mohammadi, ‘Additive manufacturing of an Fe–Cr–Ni–Al maraging stainless steel: Microstructure evolution, heat treatment, and strengthening mechanisms’, *Mater. Sci. Eng. A*, vol. 787, p. 139470, Jun. 2020, doi: 10.1016/j.msea.2020.139470.

- [110] D. N. Seidman, E. A. Marquis, and D. C. Dunand, ‘Precipitation strengthening at ambient and elevated temperatures of heat-treatable Al(Sc) alloys’, *Acta Mater.*, vol. 50, no. 16, pp. 4021–4035, Sep. 2002, doi: 10.1016/S1359-6454(02)00201-X.
- [111] J. K. Thorne, J. M. Dahl, and L. H. Vlack, ‘Partition coefficient of molybdenum in the Fe-Mo-Si-O system’, *Metall. Trans.*, vol. 1, no. 8, pp. 2125–2132, Aug. 1970, doi: 10.1007/BF02643425.
- [112] E. A. Jägle, P.-P. Choi, J. Van Humbeeck, and D. Raabe, ‘Precipitation and austenite reversion behaviour of a maraging steel produced by selective laser melting’, *J. Mater. Res.*, vol. 29, no. 17, pp. 2072–2079, Sep. 2014, doi: 10.1557/jmr.2014.204.
- [113] C. Tan, K. Zhou, M. Kuang, W. Ma, and T. Kuang, ‘Microstructural characterization and properties of selective laser melted maraging steel with different build directions’, *Sci. Technol. Adv. Mater.*, vol. 19, no. 1, pp. 746–758, Dec. 2018, doi: 10.1080/14686996.2018.1527645.
- [114] P. Bajaj, A. Hariharan, A. Kini, P. Kürnsteiner, D. Raabe, and E. A. Jägle, ‘Steels in additive manufacturing: A review of their microstructure and properties’, *Mater. Sci. Eng. A*, vol. 772, p. 138633, Jan. 2020, doi: 10.1016/j.msea.2019.138633.
- [115] F. H. Lang and N. Kenyon, ‘Welding of Maraging Steels’, p. 43.
- [116] Y. Bai, D. Wang, Y. Yang, and H. Wang, ‘Effect of heat treatment on the microstructure and mechanical properties of maraging steel by selective laser melting’, *Mater. Sci. Eng. A*, vol. 760, pp. 105–117, Jul. 2019, doi: 10.1016/j.msea.2019.05.115.

- [117] N. T. Aboulkhair, C. Tuck, I. Ashcroft, I. Maskery, and N. M. Everitt, ‘On the Precipitation Hardening of Selective Laser Melted AlSi10Mg’, *Metall. Mater. Trans. A*, vol. 46, no. 8, pp. 3337–3341, Aug. 2015, doi: 10.1007/s11661-015-2980-7.
- [118] D. D. Gu, W. Meiners, K. Wissenbach, and R. Poprawe, ‘Laser additive manufacturing of metallic components: materials, processes and mechanisms’, *Int. Mater. Rev.*, vol. 57, no. 3, pp. 133–164, May 2012, doi: 10.1179/1743280411Y.0000000014.
- [119] E. G. Thorsteinsdóttir, D. S. Primdahl, Y. B. Zhang, D. J. Jensen, and T. Yu, ‘Aging of 3D-printed maraging steel’, *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 580, p. 012047, Dec. 2019, doi: 10.1088/1757-899X/580/1/012047.
- [120] T. Allam, K. G. Pradeep, P. Köhnen, A. Marshal, J. H. Schleifenbaum, and C. Haase, ‘Tailoring the nanostructure of laser powder bed fusion additively manufactured maraging steel’, *Addit. Manuf.*, vol. 36, p. 101561, Dec. 2020, doi: 10.1016/j.addma.2020.101561.
- [121] A. Mahmoudi, M. R. Z. Ghavidel, S. H. Nedjad, A. Heidarzadeh, and M. N. Ahmadabadi, ‘Aging behaviour and mechanical properties of maraging steels in the presence of submicrocrystalline Laves phase particles’, *Mater. Charact.*, vol. 62, no. 10, pp. 976–981, Oct. 2011, doi: 10.1016/j.matchar.2011.07.012.
- [122] B. Liu, Z. Lü, W. Feng, T. Ren, and W. Fu, ‘Precipitation and decomposition behaviours of carbides in AISI M2 high-speed steel with nitrogen and mischmetal’,

- J. Cent. South Univ., vol. 24, no. 4, pp. 782–788, Apr. 2017, doi: 10.1007/s11771-017-3480-2.
- [123] W. Turnier Trottier, A. Kreitchberg, and V. Brailovski, ‘Structure and Mechanical Properties of Laser Powder Bed-Fused and Wrought PH13-8Mo-Type Precipitation Hardening Stainless Steels: Comparative Study’, *J. Manuf. Mater. Process.*, vol. 5, no. 3, p. 67, Jun. 2021, doi: 10.3390/jmmp5030067.
- [124] S. D. Meredith, J. S. Zuback, J. S. Keist, and T. A. Palmer, ‘Impact of composition on the heat treatment response of additively manufactured 17–4 PH grade stainless steel’, *Mater. Sci. Eng. A*, vol. 738, pp. 44–56, Dec. 2018, doi: 10.1016/j.msea.2018.09.066.
- [125] C. V. S. Murthy, A. G. Krishna, and G. M. Reddy, ‘Dissimilar Welding of Maraging Steel (250) and 13-8 Mo Stainless Steel by GTCAW, LBW and EBW Processes’, *Trans. Indian Inst. Met.*, vol. 72, no. 9, pp. 2433–2441, Sep. 2019, doi: 10.1007/s12666-019-01695-z.
- [126] C. Turk, H. Zunko, C. Aumayr, H. Leitner, and M. Kapp, ‘Advances in Maraging Steels for Additive Manufacturing’, *BHM Berg- Hüttenmänn. Monatshefte*, vol. 164, no. 3, pp. 112–116, Mar. 2019, doi: 10.1007/s00501-019-0835-z.
- [127] P. W. Hochanadel, G. R. Edwards, C. V. Robino, and M. J. Cieslak, ‘Heat treatment of investment cast PH 13-8 Mo stainless steel: Part I. Mechanical properties and microstructure’, *Metall. Mater. Trans. A*, vol. 25, no. 4, pp. 789–798, Apr. 1994, doi: 10.1007/BF02665455.

- [128] X. Li et al., ‘Effect of aging treatment on hydrogen embrittlement of PH 13-8 Mo martensite stainless steel’, *Mater. Sci. Eng. A*, vol. 651, pp. 474–485, Jan. 2016, doi: 10.1016/j.msea.2015.09.116.
- [129] L. W. Tsay, H. H. Chen, M. F. Chiang, and C. Chen, ‘The influence of aging treatments on sulphide stress corrosion cracking of PH 13-8 Mo steel welds’, *Corros. Sci.*, vol. 49, no. 6, pp. 2461–2473, Jun. 2007, doi: 10.1016/j.corsci.2006.12.006.
- [130] V. Seetharaman, M. Sundararaman, and R. Krishnan, ‘Precipitation hardening in a PH 13-8 Mo stainless steel’, *Mater. Sci. Eng.*, vol. 47, no. 1, pp. 1–11, Jan. 1981, doi: 10.1016/0025-5416(81)90034-3.
- [131] J. Mittra, G. K. Dey, D. Sen, A. K. Patra, S. Mazumder, and P. K. De, ‘Solution quenched structure of wrought PH 13–8 Mo stainless steel’, *Scr. Mater.*, vol. 51, no. 4, pp. 349–353, Aug. 2004, doi: 10.1016/j.scriptamat.2004.03.031.
- [132] Z. Guo, W. Sha, and D. Vaumousse, ‘Microstructural evolution in a PH13-8 stainless steel after ageing’, *Acta Mater.*, vol. 51, no. 1, pp. 101–116, Jan. 2003, doi: 10.1016/S1359-6454(02)00353-1.

3. Laser powder bed fusion of M789 maraging steel on Cr-Mo N709 steel: microstructure, texture, and mechanical properties

This chapter presents the feasibility of using N709 as a hybrid base for LPBF-M789 to minimize the build stock for M789. A modification of the interface by direct aging at 500 °C for 3 hours has been proposed for this hybrid material. The strength of the bonding is evaluated by conducting tensile and hardness tests before and after direct aging. The results of this work give valuable insight into the strength of the interface and the suitability of the applied processing parameters.

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3.1 Abstract

In this work, M789 steel was deposited (printed) onto wrought N709 steel by a laser powder bed fusion (LPBF) process to fabricate a hybrid M789-N709 alloy. The microstructure of the as-printed, new hybrid material consists of melt pool boundaries in the M789 region. Direct aging treatment was then applied, which allowed the annihilation of dislocations as well as the formation of the η -phase ($\text{Ni}_3(\text{Ti}, \text{Al})$) in the M789 section. Electron backscatter diffraction (EBSD) revealed essentially random orientations of the martensitic grains (in both as-printed and heat-treated states) with traces of reverted austenite that formed after heat treatment. Nanoindentation results, (hardness profile across the interface) showed that the post-processing treatment increased the mechanical properties of M789 steel to levels comparable to those of wrought N709 steel. This assessment was validated through tensile testing, where the as-printed material failed in the M789 section, while the heat-treated sample fractured in the N709 section. This can be attributed to the presence of the η -phase after heat treatment. Since the fracture location is in the base alloy, a strong metallurgical bond is formed between M789 and N709. Thus, the new hybrid alloy can be employed without compromised strength due to the interface.

3.2 Introduction

Additive manufacturing (AM) technology has been attracting the interest of various industries as an alternative manufacturing technique. The AM process can fabricate near-net-shape components with sophisticated designs using the layer-by-layer printing strategy [1]. The design freedom and layer-wise manufacturing process provide unique opportunities for complicated geometries and internal structures, such as lattice internal conformal cooling channels [2,3]. The near-net-shape manufacturing capability of AM has

other benefits over conventional manufacturing processes (casting, forging, rolling, etc.), for example, lowered usage of the raw materials, reduced post-machining requirement, and optimized post assembling steps by constructing a *one-of-a-kind* component [4]. Among all the AM techniques, the laser powder bed fusion (LPBF) technique has played an essential role in manufacturing metal parts by applying a laser beam as the fusing source to selectively melt the metal powder layer by layer based on a three-dimensional CAD model [5,6]. Nowadays, the LPBF technology has been widely employed in multiple industries such as aerospace, defence, automotive, oil and gas, medical, etc. [7,8].

To date, numerous variants of maraging steels are manufactured through the LPBF process for a range of applications such as high-pressure die casting, injection moulding, and other tooling applications. This is due to the excellent combination of good weldability and high strength of these steels [9]. After the LPBF process, applying an aging process is critical in influencing the formation of precipitates. The strengthening of the maraging steel depends upon the formation of precipitates in the material, which can be activated by the addition of alloying elements to form strong (Ti, Be), moderate (Al, Mn, Si, Ta, V) and weak (Co, Cu, Zr) intermetallics [10,11]. One critical issue related to traditional maraging steels is their low corrosion resistance which drastically reduces their service life, especially in severe environments [12–14]. Because of these shortcomings, a novel steel grade based on Grade 250 maraging steel, commercially known as M789 steel, was designed to maintain high printability while enhancing the corrosion resistance, which is superior to that of the 17-4PH stainless steel [12]. In previous research [15,16], the microstructure and mechanical properties of the M789 steel made by the LPBF process have been analyzed, including the relationship between the experimental and simulated results. The

precipitation behaviour has also been studied using transmission electron microscopy and atom probe tomography to explain the effect of different aging treatments on the final microstructure and mechanical properties of the M789 steel [17].

More recently, hybrid AM using LPBF technology has been increasingly used in research and industry applications [18,19], and the AM alloys have been fabricated with as-cast or wrought base metals. This hybrid LPBF technology significantly improves efficiency by reducing the machine operation time and lowering the usage of AM materials. The hybrid dual-metal could provide an advantageous option by achieving the mechanical and physical properties of two metals to meet the requirements in industries such as tooling, aerospace, automotive, oil and gas, etc. [18]. In addition, the hybrid LPBF concept might also be considered a valuable method to incorporate a metallic coating over a base metal [19].

Several recent publications presented results regarding the hybrid LPBF of maraging steels on tool steels H13 and 420SS [18,20], and non-ferrous copper alloys [21]. However, no study has been seen in the literature about hybrid LPBF-M789 steel. Since the combined mechanical and corrosion properties of the M789 steel exceed the performance of other similar alloys employed in the LPBF process, it is of interest to determine the feasibility of printing M789 steel on top of a well-known substrate (base metal). Therefore, in this work, M789 steel was deposited onto wrought N709 steel using the LPBF technique. The N709 steel belongs to the 13-8 Cr-Mo steel family and has similar chemical composition and properties to those of the M789 steel. The current research investigates the feasibility of practical use of the hybrid M789-N709 steel by evaluating the microstructure, texture, and mechanical properties of the material after fabrication and heat treatment. The findings

from this work may assist the AM community in selecting this material to manufacture hybrid parts to address the industry's evolving needs.

3.3 Experimental Procedure

3.3.1 Materials and Manufacturing

The hybrid LPBF-M789 on N709 steel was manufactured using an EOS M290 machine. The M789 powder was produced by the gas atomization process, while the N709 base metal was supplied as a wrought material. The N709 substrate was fine machined and demagnetized before printing. The chemical compositions of the M789 powder and N709 base metal are measured and listed in **Table 3.1**. The major difference between the two alloys is that the M789 steel contains 1.0 wt.% titanium, while the N709 steel does not. This alloying element can form a strong Ni_3Ti intermetallic phase that helps impede dislocation motion [17]. The powder morphology of the M789 steel was characterized by scanning electron microscopy (Hitachi SU70), as shown in **Fig. 3.1a**. The particle size distribution was measured using a CAMSIZERX2 digital image processing particle size analyzer and displayed in **Fig. 3.1b**. The M789 particles appear to be spherical and show a majority of sizes in the range of 15 to 60 μm . The flowability of the M789 powder was determined to be 15.84s/50g by using a Hall Flowmeter.

Table 3.1. The chemical compositions of the M789 powder and N709 steel (in wt.%)

Steel	C	Cr	Ni	Mo	Al	Ti	Fe
M789	<0.02	12.20	10.00	1.00	0.60	1.00	Balance
N709	0.03	12.70	8.10	2.20	1.10	-	Balance

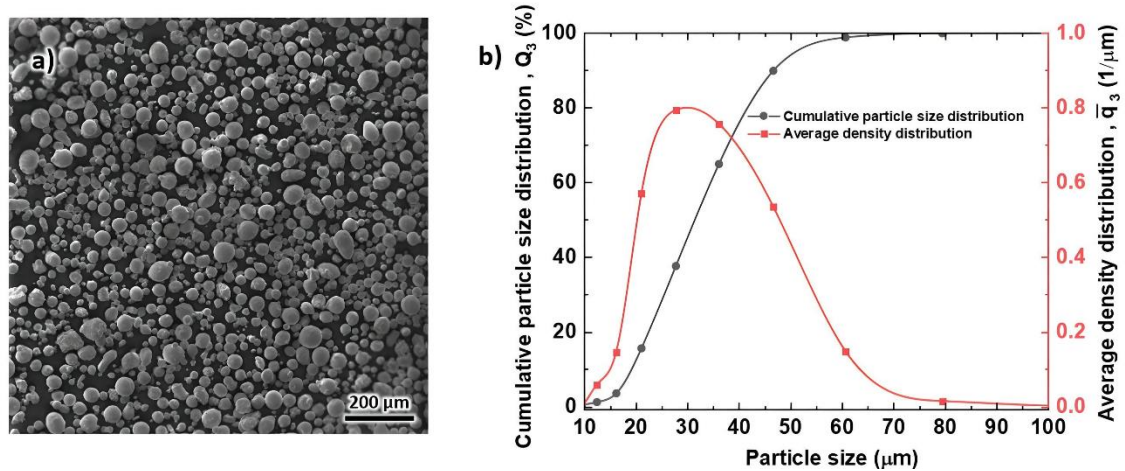


Figure 3.1. Morphology and particle size of the M789 powder: a) scanning electron microscopy (SEM) image of the M789 particles, b) particle size distribution, where Q_3 and q_3 are the cumulative distribution and average density distribution, respectively.

A schematic illustration of the hybrid LPBF process is shown in **Fig. 3.2**. The N709 metal was assembled on the base plate and embedded in the powder chamber with the same level of powder delivery roller. The M789 powder was then deposited layer by layer on the N709 base metal using the LPBF process. During fabrication, the volumetric laser energy density was maintained in the range of 75-85 J/mm³. The laser power was kept at 250-270 W, and the scanning speed was held within 1050-1200 mm/s. The hatch distance was maintained between 0.10 and 0.11 mm, and a layer thickness of ~40 μm was used.

Two types of samples were manufactured: a) 10 mm cubic samples for the microstructure and texture characterization, and b) cylindrical samples with 86 mm in height and 14 mm in diameter for tensile tests. The cylindrical bars were then machined into tensile specimens according to ASTM E8, with a gauge length of 24 mm and a diameter of 6 mm.

Selected as-printed coupons were subjected to direct aging treatment at 773 K (500 °C) for 3 hours, typical for M789 material. It is worth noting that the term direct aging as used in this study means that no solution treatment was done except for aging, hence the term

“direct”. This was done to minimize the formation of retained austenite [16]. The aging treatment process was conducted under an argon atmosphere to minimize oxidation and samples were air-cooled to room temperature.

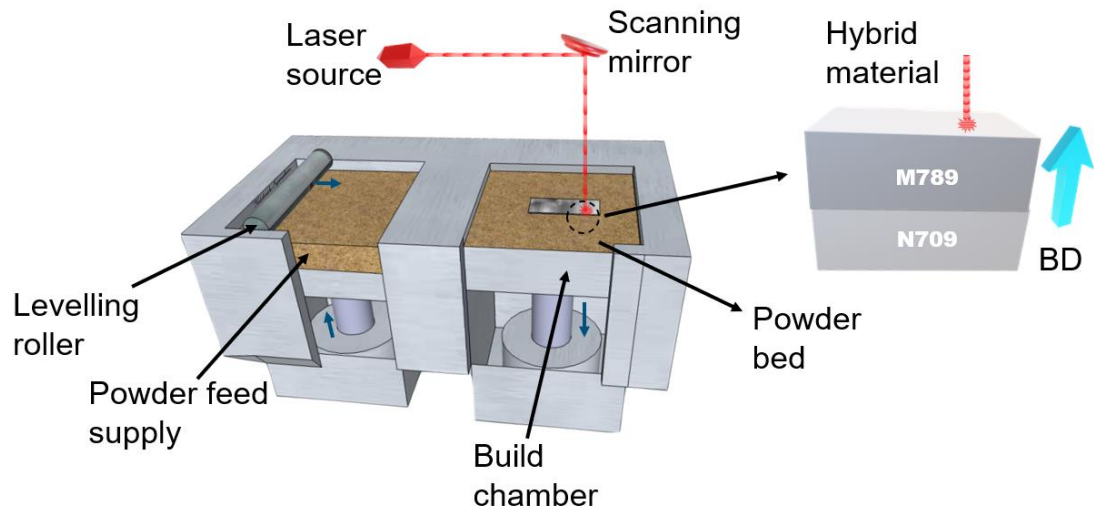


Figure 3.2. Schematic illustration of the hybrid LPBF process. The M789 steel is deposited on top of the wrought N709 steel.

The as-printed and aged cubic samples were sectioned parallel to the building direction, followed by sequential grinding up to 1200 grit SiC abrasive papers, and standard polishing up to 1 μm diamond suspensions. The relative density was obtained using a Zeta Instruments optical microscope. Ten regions were randomly selected to measure the relative density of the LPBF-M789 steel using the ImageJ software, and the average value is presented. The polished samples were then electrolytically etched in an ammonium persulfate solution at 6 volts for 10 seconds to reveal the detailed microstructure under an optical microscope and SEM.

3.3.2 Microstructural characterization and mechanical testing

An SEM analysis was conducted on both the as-printed and aged specimens to characterize the microstructure, and electron backscatter diffraction (EBSD) was performed on the samples to characterize the microtexture (Oxford Instruments HKL EBSD System with the Channel5 Software). The sample preparation for EBSD analysis involved an additional polishing step using a 0.02 μm silica suspension for 24 hours in a vibratory polisher. XRD phase characterization was performed on both as-printed and aged samples using a Bruker D8 Advance spectrometer with Cu K α radiation at an axis normal to the building direction. The X-Ray analytical instrument was operated at 40kV and 30mA at a step size and time of 0.02° and 1 second, respectively. Vickers hardness was measured by a Nanomechanics iMicro machine across the interface between the LPBF M789 and N709 base metal. Tensile tests were carried out according to ASTM E8 utilizing an Instron 1332 machine equipped with an extensometer. The fractography of the tensile specimens was revealed by means of SEM analysis.

3.4 Results

3.4.1 Microstructure of the as-printed material

A relative density of 99.6 ± 0.2 % was achieved in the LPBF-M789 region after fabrication. Optical micrograph (OM) and SEM images of the hybrid sample in the as-printed state are presented in **Figs. 3.3a, 3.3b** and **3.3c**. These images show the overlapping melt pools in the M789 region (see red broken lines and arrows in **Figs. 3.3a** and **3.3b**), consistent with the observations in the literature for the same alloy [16]. The melt pool has width and depth of around 145 μm and 95 μm , respectively.

The magnified SEM image in the M789 region in **Fig. 3.3b** displays a cellular-dendritic microstructure with dendrite arms aligned to the direction of heat dissipation during manufacturing (i.e., along the building direction). Moreover, the cellular structures are clustered close to the melt pool boundaries, where there is a relatively high thermal gradient and low solidification rate at the surface of the melt pool [22]. A mixture of the columnar and dendritic structures is due to the large undercooling and high solidification rate (estimated to be in the range of 10^6 - 10^8 K/s) [22]. The interface between M789 and N709 can be distinctively determined from the differences in morphology of their structures: N709 shows equiaxed grains with martensitic morphology and a possibility of precipitates as shown in [10,17]; by contrast, the interface, as depicted in **Fig. 3.3c**, has a mixture of partially formed melt pools with fine, oriented grains indicative of remelting of the top N709 layer when the M789 steel was-printed.

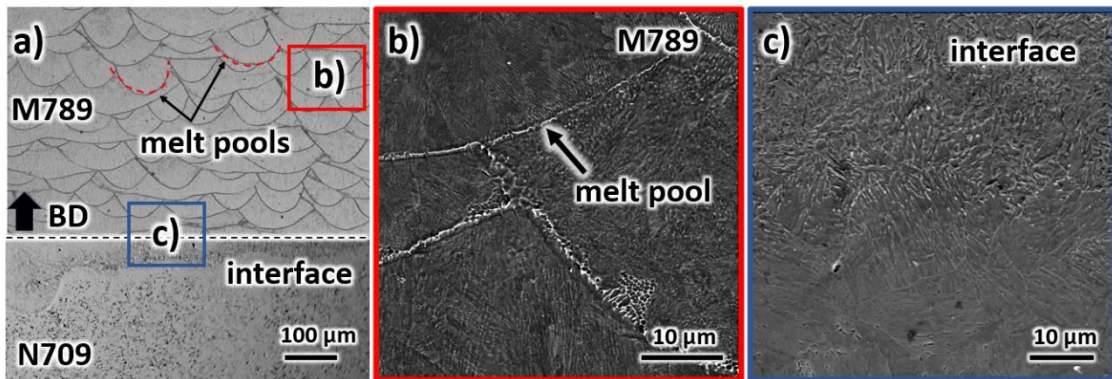


Figure 3.3. Microstructure of the as-printed hybrid LPBF-M789 and N709 steel: a) optical micrograph of the interface, b) SEM image of the LPBF-M789 region, and c) enlarged SEM image of the interface.

3.4.2 Microstructure of the directly aged sample

Direct aging at 500 °C for 3 hours on the as-printed sample allows a partial dissolution of the melt pool boundaries, as shown in **Fig. 3.4a**. Ideally melt pools boundaries dissolve

during solution treatment (full dissolution) at elevated temperatures of 1000 °C [15,16,18] but will not be completely dissolved during direct aging because of the low temperatures hence the partial dissolution. Direct aging was used because the rapid cooling in LPBF produces a complete martensitic structure [23]. As shown in **Fig. 3.4b**, the M789 region shows a significant difference in microstructure from the as-printed state (**Fig. 3.3b**). The overlapping melt pool boundaries, which did not completely dissolve during aging, contain visible lath martensitic structures (approximately 2 μm in width) inside the prior austenite grains. Note that the previously observed cellular and dendritic structures in the as-printed state are no longer evident, and the morphology almost resembles that of a typical heat-treated maraging steel [24]. At this level of magnification, the strengthening η -phase precipitates are not visible [15–17], which have been reported to exist based on the previous atom probe tomography analysis and JMatPro simulations. Similarities in the microstructures at the boundaries can be seen in **Fig. 3.4c**, with N709 having more distinct martensitic laths than M789. The interface features are still visible; however, the previously observed melt pools have disappeared.

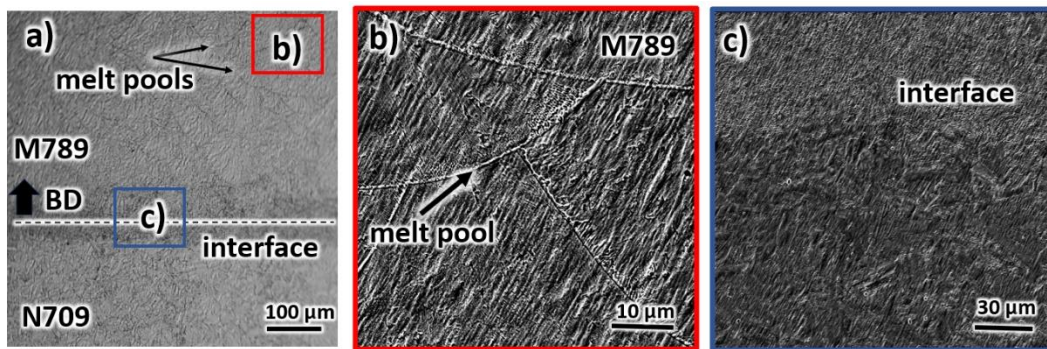


Figure 3.4. Microstructure after direct aging: a) optical micrograph of the interface, b) SEM micrograph of the heat-treated M789 region, c) enlarged SEM image of the M789-N709 interface.

3.5 Mechanical properties

3.5.1 Hardness across interface

The change in hardness across the interface before direct aging is depicted in **Fig. 3.5**. The properties of individual materials were measured as: 661 ± 16 HV in the N709 area and 486 ± 26 HV in the M789 region. Note that the M789-N709 interface is represented by a sharp drop in the hardness values. Generally, the additively manufactured metals are expected to have higher hardness values in the as-printed state due to the high cooling rates that promote grain size strengthening (complemented with high dislocation density) according to the Hall-Petch effect [18]. However, lower hardness values compared to wrought N709 were observed here. Interestingly, the hardness values of the LPBF-M789 steel close to the interface (approximately 100 μm from the interface) are slightly higher than the other data points away from the interface. This discrepancy might be due to the diffusion of alloying elements from N709 to M789 during printing; the latter alloy is expected to have a high diffusion rate of the alloying elements because of the higher temperature (i.e., melting of the alloy) associated with the LPBF.

The hardness profile of the hybrid material after direct aging is presented in **Fig. 3.6**. While the hardness values of the N709 steel essentially do not change as compared to those before heat treatment, the M789 steel shows a significant increase of the hardness from approximately 500 HV to 700 HV. These observations are consistent with the expected effect of precipitate strengthening for M789 [3].

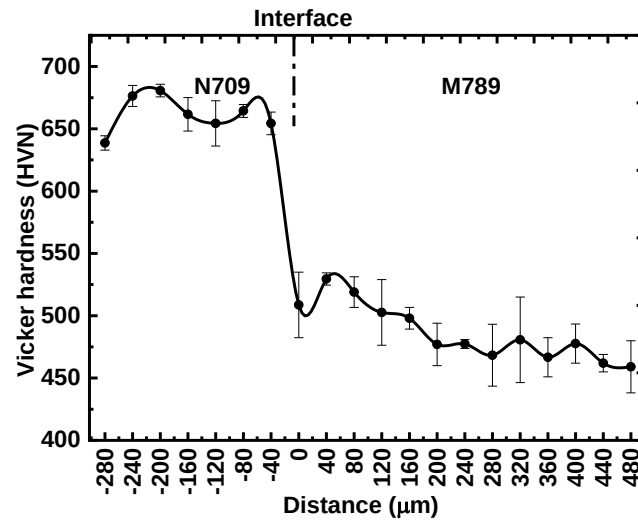


Figure 3.5. Vicker hardness measurement across the M789-N709 interface before direct aging.

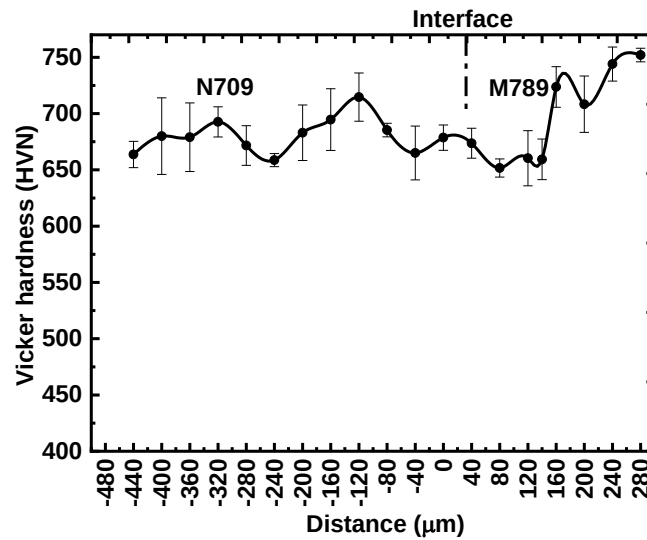


Figure 3.6. Vicker hardness measurement across the M789-N709 interface after direct aging.

3.5.2 Tensile tests

The uniaxial tensile testing results associated with the as-printed and aged states of the M789-N709 hybrid alloy are shown in **Fig. 3.7a**. For the as-printed sample, the tensile strength and the maximum strain to failure are measured at 1100 MPa and 0.13, respectively. On the other hand, the heat-treated sample has a tensile strength of 1600 MPa, with a maximum strain of 0.18 before fracture. From the location of the failure (**Fig. 3.7b**), for the as-printed sample, the failure is in the M789 side, while for the heat-treated material, the fracture is in the N709 part. It is important to note that the tensile strength of the LPBF-M789 steel (with similar processing history) was reported to be around 1800 MPa [16]. Since in both the as-printed and directly aged samples, the fracture location is not at the interface, it can be concluded that a sound metallurgical bond between M789 and N709 was achieved.

To further assess the results of the tensile tests, fractographic analysis was performed on the failed tensile samples, as displayed in **Fig. 3.8**. The maximum strain values to failure in both the as-printed and directly aged states suggest that the hybrid material possesses good ductility. The images in **Figs. 3.8a** and **3.8b** support this assessment, where the typical cup-and-cone features of ductile fracture are visible in the M789 and N709 steels. The M789 failure section has a mixture of large and small cup sections, with the large sections reaching an average of 60 μm in diameter. The N709 section shows similarly sized cup sections with cup diameter less than 50 μm .

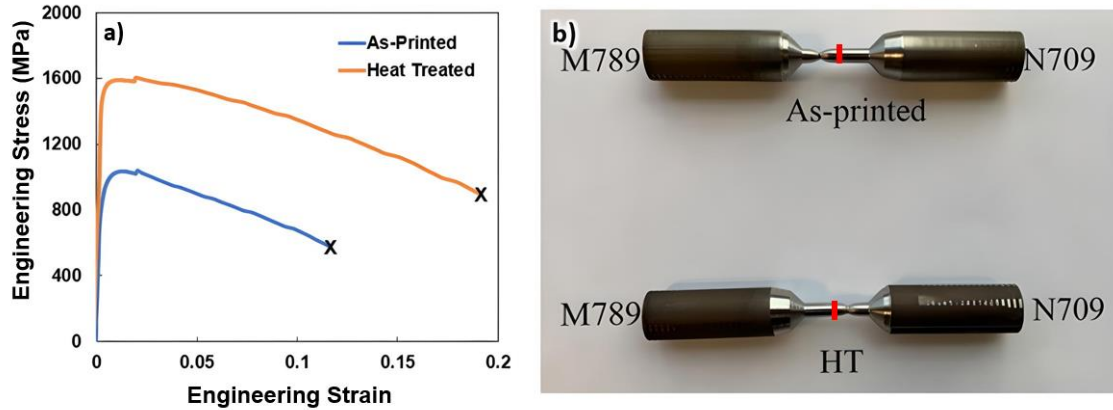


Figure 3.7. Uniaxial tensile testing results of the samples before and after aging showing: a) engineering stress-strain curves, and b) image of the fractured samples.

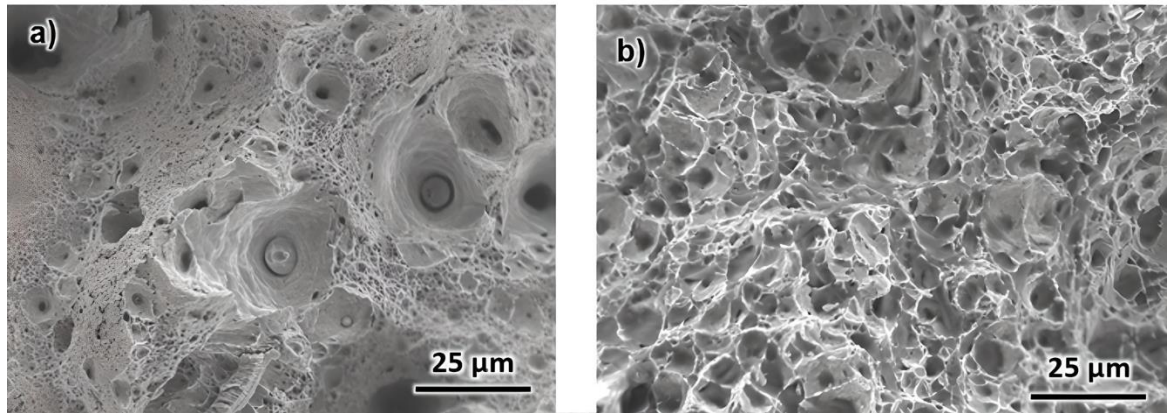


Figure 3.8. The fracture surfaces after tensile testing of: a) as-printed, b) directly aged samples. The as-printed sample failed on the M789 section, while the heat-treated one fractured on the N709 region.

3.6 Discussion

From the results presented above, especially for the as-printed sample, apparent heterogeneity in mechanical properties is observed (i.e., the hardness profile measurement in **Fig. 3.5**), which negatively impacts applications that require multi-directional stresses. A uniform structure is usually preferred if homogeneous mechanical properties are desired [25,26]. This issue has been solved by applying a specific heat treatment to address

the contrasting microstructural properties in the LPBF-M789 and wrought N709 alloys. After heat treatment, the microstructure, and mechanical properties of LPBF-M789 are comparable to conventional N709, as observed from the hardness profile of **Fig. 3.6**. Moreover, based on the tensile testing results, the bond between the M789 and N709 appears to be stronger than the individual alloys. A deeper analysis of the microstructure is presented below, complemented by the study of the precipitation that takes place during the post-processing heat treatment.

3.6.1 Texture analysis

The EBSD inverse pole figure (IPF) map of the as-printed material obtained in the vicinity of the interface is presented in **Fig. 3.9a**. A distinct interface similar to the previous SEM images can be observed (see **Figs. 3.9a-3.9c**). The interface consists of very fine grains (less than 10 μm), typical of AM process, which involves high cooling rates. Note that, during the start of the fabrication, N709 acted as a heat sink at the initial deposition of the M789 layer [18]. In addition, there is a clear distinction between the morphology of the grains, with M789 having relatively smaller grains, less than 20 μm compared to N709 with relatively large equiaxed grains approximately 30 μm . Here, the longitudinal directions of the M789 grains are along the building direction (BD), indicating that the grain growth direction follows the thermal gradient. Despite the temperature-dependency of the shape of the grains, the EBSD reveals a very weak texture, with the $\{111\}$ planes perpendicular to the building direction (BD) at a maximum intensity of 1.73.

In addition to crystallographic texture, kernel average misorientation (KAM) map was calculated from the EBSD data in **Fig. 3.9b**, showing about 44% of the area has misorientation angles of greater than 2° . This information is directly related to the

dislocation density in the material, which will be compared later to the results of the aged sample. It is well-known that AM processes accumulate large amounts of residual stresses hence the need for a post-printing heat treatment process [27]. Note that the residual stresses accumulated during the LPBF process can lead to a reduction in ductility and toughness, as presented in the stress-strain curves in **Fig. 3.7a**. A high volume of dislocations and residual stress generally increase the hardness of the M789 steel [8,16]; however, this is not consistent with the presented hardness results. This can be explained by the pre-treatment of N709 steel before printing which will be discussed later in this section. In addition to the analysis above, the phase fraction was also measured in the as-printed state (see **Fig. 3.9c**): 99.7% (0.3% austenite) of the phase being martensitic (red regions) with negligible amounts of retained austenite (yellow regions). To confirm the presence of retained austenite and since mis-indexing during the EBSD data acquisition is a possibility an XRD analysis carried out indicated the presence of a (111) γ and (200) γ peak with a quantitative amount of 0.4% retained austenite see (**Fig. 3.10**). Note that the amount of austenite has been reported to lower the maximum achievable strength post-aging [28].

The EBSD IPF map of the sample after direct aging is shown in **Fig. 3.11a**, where a similar degree of randomness of the orientations was observed (with a maximum texture intensity of 1.34). There is a significant drop in the area fractions with local misorientation angles of greater than 2° , from 44% to about 8%, as a result of aging, and consequently dislocation annihilation (see KAM map in **Fig. 3.11b**). Residual stresses that can aggravate failure were also minimized by the heat treatment [8]. In addition, the phase maps indicate an increased distribution of yellow regions (austenite) across the interface, amounting to

1.93% (see **Fig. 3.11c**). The presence of austenite appears to improve the ductility and toughness (compared to the as-printed state), as evidenced by the increased elongation before failure (18%) in the stress-strain curves of the directly aged specimen (see **Fig. 3.7a**). XRD results also confirm the presence of austenite which amounts to 2.03% (see **Fig. 3.12**.) However, it should be noted that an excess of austenite, a softer phase than martensite, can significantly lower the mechanical properties. In the literature, the combined effect of coarse precipitates (due to overaging), retained austenite, and microsegregation of alloying elements has been found to promote austenite reversion [29]. The heat treatment employed in this work has been found to promote the formation of fine precipitates with significant volume densities, ultimately giving high strength and toughness [16].

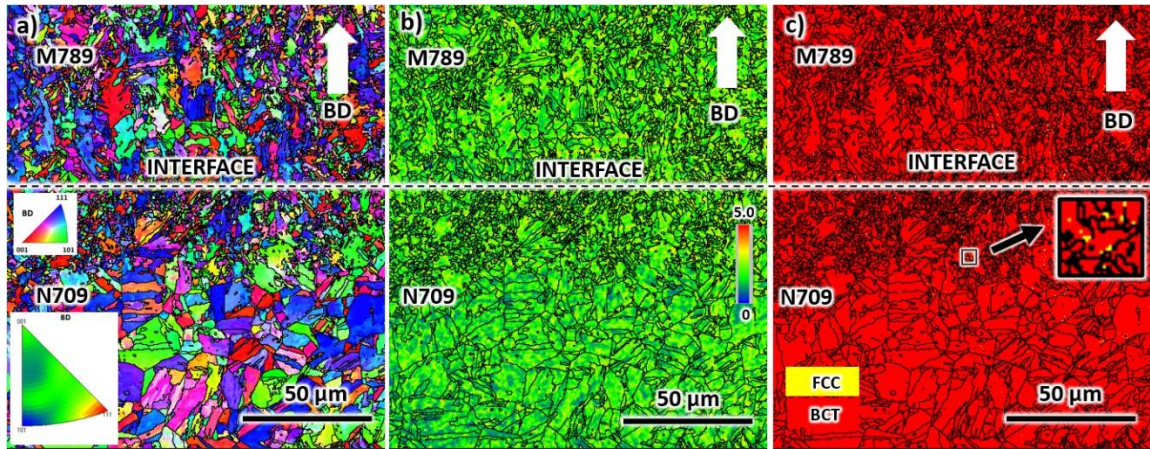


Figure 3.9. EBSD analysis of the as-printed sample showing: a) IPF map (BD), b) kernel average misorientation (KAM) map, and c) phase map. The structure is martensitic with an almost random texture.

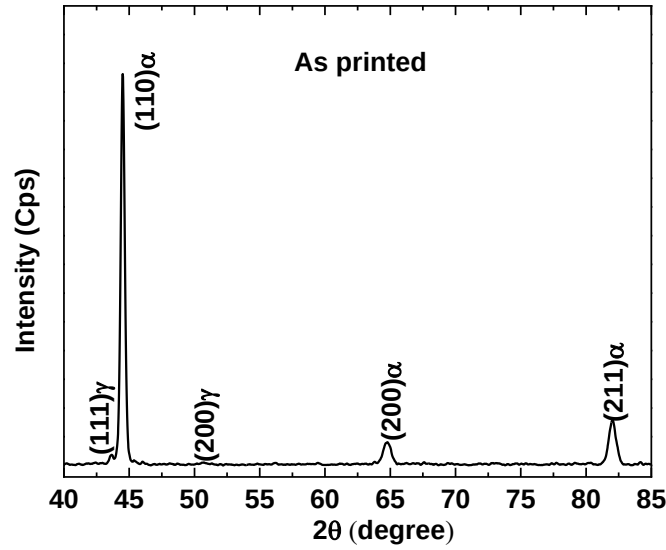


Figure 3.10. XRD phase analysis of as-printed M789-N709.

3.6.2 Precipitation kinetics simulation

Thermodynamic calculations were carried out using the JMatPro simulation software (Version 12) using the Fe-based module, in which N709 and M789 were analyzed separately. The simulations are based on the Johnson-Mehl-Avrami equation, which assumes a spherical morphology and isothermal conditions, as shown in Equation (3.1):

$$x = 1 - e^{\left(-\frac{\pi}{3}N_r G_r^3 t^4\right)} \quad (3.1)$$

where x is the volume fraction of the precipitate during solid-state transformation, N_r and G_r are the nucleation rate and growth rate, respectively, at heat treatment time t . For M789 steel, the simulation reveals an estimated value of 5% of the ETA-Ni₃(Ti, Al) (η -phase) after the aging process, as displayed in **Fig. 3.13a**.

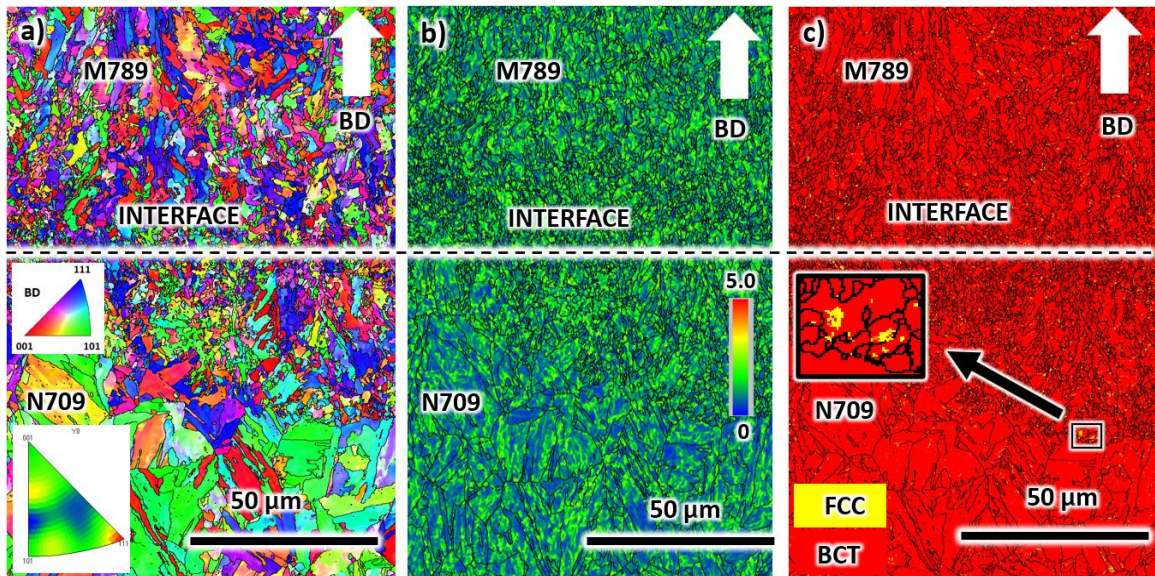


Figure 3.11. The EBSD analysis of the heat-treated sample showing: a) IPF map (BD), b) kernel average misorientation (KAM) map, c) phase map. The structure is martensitic with a relatively random texture.

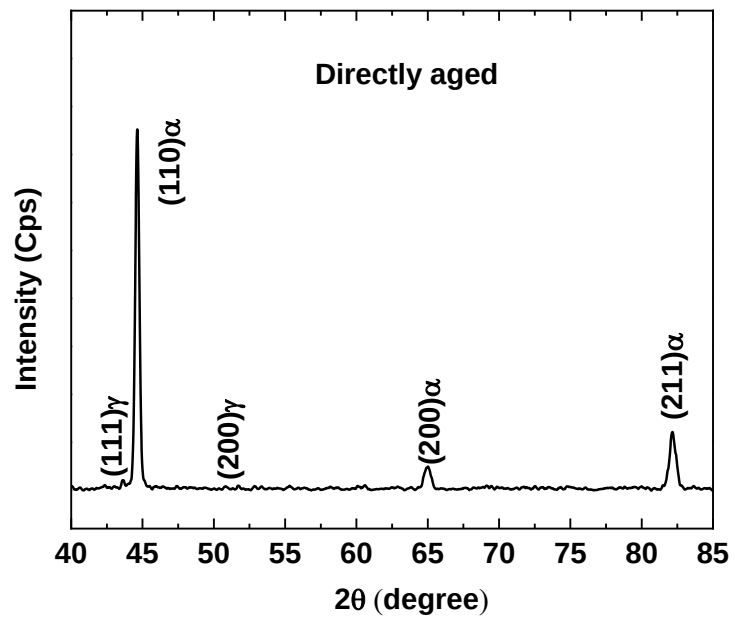


Figure 3.12. XRD phase analysis for directly aged M789-N709.

The presence of these hardening precipitates agrees with the spike in hardness values after crossing the interface (from the N709 side), as reported in the hardness profile in **Fig. 3.6**. This metallurgical phenomenon improves interstitial solid solution strengthening as they impede dislocation motion, according to the Orowan-Ashby theory [30]. To further understand why the failure occurred in M789 before heat treatment, another simulation was carried out using N709 steel as an input; the output is presented in **Fig. 3.13b**. Before depositing M789, N709 had a dominant (Fe, Ni) Al phase amounting to 3.5%. This phase has been confirmed in the form of β -NiAl precipitates. The presence of Ni and Al in this steel grade has been reported to promote the formation of β -NiAl precipitates enclosed in lath martensite at aging temperatures between 450 °C-575 °C. Even at temperatures exceeding 575 °C for 4 hours of aging, the alloy has been reported to maintain a coherent phase with some reverted austenite which is an indication of good resistance to over aging [31]. The comparison in the strengthening effect of η -phase and β -NiAl may be a subject of further investigation.

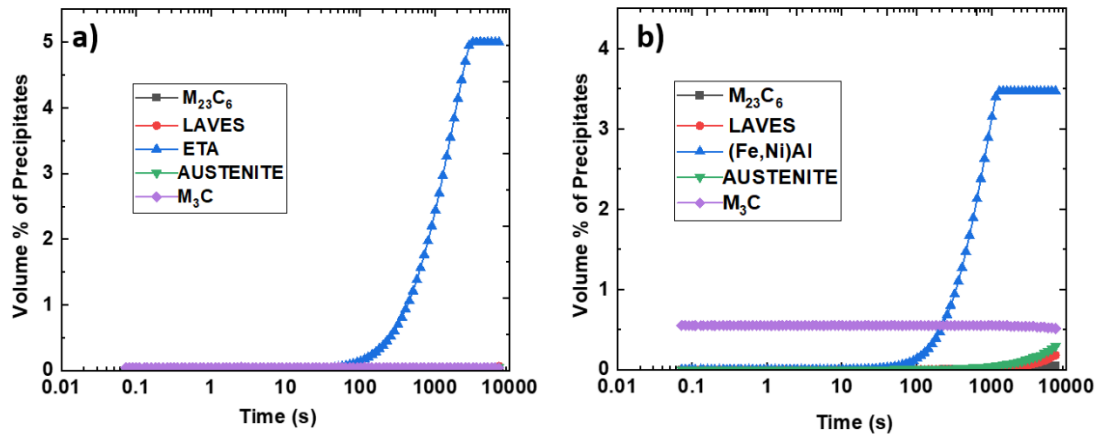


Figure 3.13. Precipitation simulations indicating the dependence of the volume percentages of precipitates on holding time for: a) M789 steel, b) N709 steel.

3.7 Conclusions

The mechanical and microstructural properties of the LPBF M789-N709 hybrid material were assessed to determine the feasibility of practical use of this new material. Based on the experimental and simulation results, the following conclusions can be drawn:

1. A strong metallurgical bond is formed between the M789 and N709 steels by the LPBF process, as evidenced by the fact that the material fractured either in the M789 section (as-printed) or in the N709 section (direct aging) during tensile testing, not on the interface.
2. The fracture of the as-printed material on the M789 part during the tensile test may be attributed to the strengthening of the β -NiAl precipitates in the wrought N709 section before direct aging and η -phase after aging which strengthened the M789 section.
3. The hardness of the M789 section after heat treatment was considerably increased, due to precipitation strengthening, while that of the base N709 section is essentially not changed.
4. The manufactured M789-N709 hybrid material appears to have a randomly oriented martensitic structure after the LPBF process, with a very weak $\langle 111 \rangle // BD$ texture (maximum intensity of 1.73) before aging and after direct aging (maximum intensity of 1.34).

3.8 References

- [1] Y. Tian et al., ‘Rationalization of Microstructure Heterogeneity in INCONEL 718 Builds Made by the Direct Laser Additive Manufacturing Process’, Metall. Mater.

- Trans. A, vol. 45, no. 10, pp. 4470–4483, Sep. 2014, doi: 10.1007/s11661-014-2370-6.
- [2] A. Armillotta, R. Baraggi, and S. Fasoli, ‘SLM tooling for die casting with conformal cooling channels’, *Int. J. Adv. Manuf. Technol.*, vol. 71, no. 1–4, pp. 573–583, Mar. 2014, doi: 10.1007/s00170-013-5523-7.
- [3] Y. Tian, K. Chadha, S. H. Kim, and C. Aranas, ‘Strengthening mechanisms in a heatvar hot work tool steel fabricated by laser powder bed fusion’, *Mater. Sci. Eng. A*, vol. 805, p. 140801, Feb. 2021, doi: 10.1016/j.msea.2021.140801.
- [4] Y. Tian, R. Gauvin, and M. Brochu, ‘Microstructure Evolution and Rapid Solidification Behavior of Blended Nickel-Based Superalloy Powders Fabricated by Laser Powder Deposition’, *Metall. Mater. Trans. A*, vol. 47, no. 7, pp. 3771–3780, Jul. 2016, doi: 10.1007/s11661-016-3505-8.
- [5] Y. Tian, J. A. Muñiz-Lerma, and M. Brochu, ‘Nickel-based superalloy microstructure obtained by pulsed laser powder bed fusion’, *Mater. Charact.*, vol. 131, pp. 306–315, Sep. 2017, doi: 10.1016/j.matchar.2017.07.024.
- [6] K. Chadha, Y. Tian, J. Spray, and C. Aranas, ‘Effect of Annealing Heat Treatment on the Microstructural Evolution and Mechanical Properties of Hot Isostatic Pressed 316L Stainless Steel Fabricated by Laser Powder Bed Fusion’, *Metals*, vol. 10, no. 6, p. 753, Jun. 2020, doi: 10.3390/met10060753.
- [7] H. Fayazfar et al., ‘A critical review of powder-based additive manufacturing of ferrous alloys: Process parameters, microstructure and mechanical properties’, *Mater. Des.*, vol. 144, pp. 98–128, Apr. 2018, doi: 10.1016/j.matdes.2018.02.018.

- [8] T. DebRoy et al., ‘Additive manufacturing of metallic components – Process, structure and properties’, *Prog. Mater. Sci.*, vol. 92, pp. 112–224, Mar. 2018, doi: 10.1016/j.pmatsci.2017.10.001.
- [9] K. Chadha, Y. Tian, P. Bocher, J. G. Spray, and C. Aranas, ‘Microstructure Evolution, Mechanical Properties and Deformation Behavior of an Additively Manufactured Maraging Steel’, *Materials*, vol. 13, no. 10, Art. no. 10, Jan. 2020, doi: 10.3390/ma13102380.
- [10] U. K. Viswanathan, G. K. Dey, and M. K. Asundi, ‘Precipitation Hardening in 350 Grade Maraging Steel’, *Metall. Mater. Trans. A*, vol. 24, no. 11, pp. 2429–2442, Nov. 1993, doi: 10.1007/BF02646522.
- [11] R. E. Smallman and A. H. W. Ngan, *Physical metallurgy and advanced materials*, 7th ed. /., 1 online resource (xxi, 650 pages): illustrations vols. Amsterdam; ButterworthHeinemann, 2007. [Online]. Available: <http://www.books24x7.com/marc.asp?bookid=37245>
- [12] C. Turk, H. Zunko, C. Aumayr, H. Leitner, and M. Kapp, ‘Advances in Maraging Steels for Additive Manufacturing’, *BHM Berg- Hüttenmänn. Monatshefte*, vol. 164, no. 3, pp. 112–116, Mar. 2019, doi: 10.1007/s00501-019-0835-z.
- [13] P. B. Srinivasan, C. Marikkannu, S. Ramu, and K. Balakrishnan, ‘Corrosion of maraging steels in chloride solutions’, *Br. Corros. J.*, vol. 29, no. 2, pp. 132–135, Jan. 1994, doi: 10.1179/000705994798267782.
- [14] A. F. Avelino, W. S. Araújo, D. F. Dias, L. P. M. dos Santos, A. N. Correia, and P. de Lima-Neto, ‘Corrosion investigation of the 18Ni 300 grade maraging steel in

- aqueous chloride medium containing H₂S and CO₂', *Electrochimica Acta*, vol. 286, pp. 339–349, Oct. 2018, doi: 10.1016/j.electacta.2018.08.042.
- [15] R. Palad, Y. Tian, K. Chadha, S. Rodrigues, and C. Aranas, 'Microstructural features of novel corrosion-resistant maraging steel manufactured by laser powder bed fusion', *Mater. Lett.*, vol. 275, p. 128026, Sep. 2020, doi: 10.1016/j.matlet.2020.128026.
- [16] Y. Tian, R. Palad, and C. Aranas, 'Microstructural evolution and mechanical properties of a newly designed steel fabricated by laser powder bed fusion', *Addit. Manuf.*, vol. 36, p. 101495, Dec. 2020, doi: 10.1016/j.addma.2020.101495.
- [17] Y. Tian, R. Palad, L. Jiang, T. Dorin, K. Chadha, and C. Aranas, 'The effect of heat treatments on mechanical properties of M789 steel fabricated by laser powder bed fusion', *J. Alloys Compd.*, vol. 885, p. 161033, Dec. 2021, doi: 10.1016/j.jallcom.2021.161033.
- [18] S. Shakerin, A. Hadadzadeh, B. S. Amirkhiz, S. Shamsdini, J. Li, and M. Mohammadi, 'Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique', *Addit. Manuf.*, vol. 29, p. 100797, Oct. 2019, doi: 10.1016/j.addma.2019.100797.
- [19] K. Chadha, Y. Tian, J. Pasco, and C. Aranas, 'Dual-metal laser powder bed fusion of iron- and cobalt-based alloys', *Mater. Charact.*, vol. 178, p. 111285, Aug. 2021, doi: 10.1016/j.matchar.2021.111285.
- [20] L. M. S. Santos, J. A. M. Ferreira, J. D. Costa, and C. Capela, 'Fatigue Performance of Hybrid Steel Samples with Laser Sintered Implants', *Procedia Eng.*, vol. 160, pp. 143–150, 2016, doi: 10.1016/j.proeng.2016.08.874.

- [21] C. Tan, K. Zhou, W. Ma, and L. Min, 'Interfacial characteristic and mechanical performance of maraging steel-copper functional bimetal produced by selective laser melting based hybrid manufacture', *Mater. Des.*, vol. 155, pp. 77–85, Oct. 2018, doi: 10.1016/j.matdes.2018.05.064.
- [22] J. P. Oliveira, A. D. LaLonde, and J. Ma, 'Processing parameters in laser powder bed fusion metal additive manufacturing', *Mater. Des.*, vol. 193, p. 108762, Aug. 2020, doi: 10.1016/j.matdes.2020.108762.
- [23] Y. Bai, D. Wang, Y. Yang, and H. Wang, 'Effect of heat treatment on the microstructure and mechanical properties of maraging steel by selective laser melting', *Mater. Sci. Eng. A*, vol. 760, pp. 105–117, Jul. 2019, doi: 10.1016/j.msea.2019.05.115.
- [24] E. I. Galindo-Nava and P. E. J. Rivera-Díaz-del-Castillo, 'Understanding the factors controlling the hardness in martensitic steels', *Scr. Mater.*, vol. 110, pp. 96–100, Jan. 2016, doi: 10.1016/j.scriptamat.2015.08.010.
- [25] C. V. S. Murthy, A. Gopala Krishna, and G. M. Reddy, 'Microstructure and mechanical properties of similar and dissimilar metal gas tungsten constricted arc welds: Maraging steel to 13-8 Mo stainless steel', *Def. Technol.*, vol. 15, no. 1, pp. 111–121, Feb. 2019, doi: 10.1016/j.dt.2018.04.005.
- [26] F. F. Conde et al., 'Effect of thermal cycling and aging stages on the microstructure and bending strength of a selective laser melted 300-grade maraging steel', *Mater. Sci. Eng. A*, vol. 758, pp. 192–201, Jun. 2019, doi: 10.1016/j.msea.2019.03.129.

- [27] T. LeBrun, T. Nakamoto, K. Horikawa, and H. Kobayashi, 'Effect of retained austenite on subsequent thermal processing and resultant mechanical properties of selective laser melted 17–4 PH stainless steel', *Mater. Des.*, vol. 81, pp. 44–53, Sep. 2015, doi: 10.1016/j.matdes.2015.05.026.
- [28] W. Turnier Trottier, A. Kreitzberg, and V. Brailovski, 'Structure and Mechanical Properties of Laser Powder Bed-Fused and Wrought PH13-8Mo-Type Precipitation Hardening Stainless Steels: Comparative Study', *J. Manuf. Mater. Process.*, vol. 5, no. 3, p. 67, Jun. 2021, doi: 10.3390/jmmp5030067.
- [29] E. A. Jäggle, P.-P. Choi, J. Van Humbeeck, and D. Raabe, 'Precipitation and austenite reversion behaviour of a maraging steel produced by selective laser melting', *J. Mater. Res.*, vol. 29, no. 17, pp. 2072–2079, Sep. 2014, doi: 10.1557/jmr.2014.204.
- [30] V. K. Vasudevan, S. J. Kim, and C. M. Wayman, 'Precipitation reactions and strengthening behaviour in 18 Wt Pct nickel maraging steels', *Metall. Trans. A*, vol. 21, no. 10, pp. 2655–2668, Oct. 1990, doi: 10.1007/BF02646061.
- [31] V. Seetharaman, M. Sundararaman, and R. Krishnan, 'Precipitation hardening in a PH 13-8 Mo stainless steel', *Mater. Sci. Eng.*, vol. 47, no. 1, pp. 1–11, Jan. 1981, doi: 10.1016/0025-5416(81)90034-3.

4. Precipitation behaviour at the interface of an additively manufactured M789-N709 hybrid alloy

This chapter provides a detailed analysis of the precipitate evolution along the M789-N709 interface. It examines the interface for changes in chemical composition and texture resulting from dilution during the initial printing of M789 on the N709 baseplate. Building upon Chapter 3, Chapter 4 proposes the mechanism responsible for achieving a robust interface. The novelty of this work lies in its exploration of precipitate evolution along the interface, facilitated by thermodynamic and kinetic simulations, as well as APT analysis.

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*Kudakwashe Nyamuchiwa served as the lead investigator of this work and was primarily responsible for writing this manuscript and performing the experimental work. Co-authors contributed to defining the problem, facilitating experiments, and finalizing the manuscript.

4.1 Abstract

A novel hybrid alloy, designed for plastic injection dies, was fabricated by depositing M789 steel on wrought N709 steel through laser powder bed fusion (LPBF). The M789-N709 interface (with a thickness of about 300 μm) is characterized by the variation of titanium content from 0% in the N709 section to about 1% near the M789 region. A thermodynamic and kinetic simulation after aging treatment was performed to reveal the precipitation behaviour in the M789-N709 interface. Different sizes and amounts of $\text{ETA-Ni}_3(\text{Ti, Al})$ strengthening precipitates exist, with a maximum volume fraction and radius of about 5% and 5 nm, respectively (near the M789 region). Moreover, the volume fraction of precipitates significantly increases with Ti additions of up to 0.6 wt.% (i.e., 100 μm from the N709 region); however, beyond this threshold, the increase in the amount of precipitation becomes gradual. The thermodynamic and kinetic simulations performed in the interface are validated by EDS, EPMA and APT results; the measured precipitate size from the APT analysis is consistent with the simulation results. Two morphologies of precipitates were detected: elongated and spherical; both contribute to the robustness of the interface. The robust M789-N709 interface formed during the process shows good compatibility between the alloys, which is critical for extended tool life. These new insights about the precipitation at the interface of M789-N709 alloy are crucial in assessing the feasibility of N709 as a cost-effective base material to minimize the use of M789 when printing plastic injection dies.

4.2 Introduction

Additive manufacturing (AM) technologies have progressed from mere prototyping to producing more sophisticated parts that find their use in diverse industries [1]. Further

advancements have resulted in manufacturing alloys utilizing processes such as laser powder bed fusion (LPBF) [2]. This technique uses laser energy that selectively melts powders on a bed following a computer-aided design (CAD) model [3]. The process is repeated layer after layer until the desired component is fabricated. As reported in the literature [4,5], the LPBF technique produces near-net shape components with high packing densities and mechanical properties, which are attractive features for maraging steel components used in high-strength applications such as aircraft landing gears and high-strength tools. More specifically, Allam et al. [6] showed the importance of heat treatment post-processing of additively manufactured maraging steels, where the volume fraction of precipitates in maraging steel 300 is increased to improve its tensile strength.

As can be seen in another work [7], post-heat treatment of additively manufactured maraging steel significantly improves the mechanical properties in comparison to its as-printed state. However, a proper heat treatment schedule must be developed to avoid the formation of excess amounts of retained austenite, which can compromise the mechanical properties [8]. Moreover, post-processing of AM alloys is not just limited to heat treatment alone. As shown by Chadha et al. [9], the application of hot isostatic pressing improves the ductility of AM alloys at the expense of yield/tensile strength, complemented by a reduction of porosity. These studies indicate the importance of understanding the possible post-processing techniques after AM to generate a product with desirable mechanical properties.

As the use of LPBF technology increases, shortcomings become more evident that slow down the adoption of this technology. It is important to note that LPBF metal additive manufacturing is a costly process compared to conventional processing techniques (such

as casting, forging, rolling, etc.) [10]. Its application is limited to high-end components and small build sizes between 300-400 mm [11]. System productivity rates of conventional processes are estimated to exceed 100kg/h. In comparison, LPBF has less than 60g/h building rates with optimized process parameters, which inevitably increases overhead costs [12,13]. In addition to the slow building rates, post-processing, such as machining and heat treatments, are necessary to attain properties that match traditionally formed components, as highlighted by Prakash et al. [14], and Popov et al. [15]. Service parts produced by the metal additive manufacturing (M-AM) process often have rough and stair-cased surface finish in addition to internal residual stresses and part distortion in their as-printed state. Residual stresses arise from the rapid heating and cooling experienced in additive manufacturing during powder melting and layer melting back during dilution [16]. Jayanath et al. [17] reported that localized stresses in metal additive manufactured products are caused by non-uniform thermal deformations, which often result in non-uniform elastic distortions during cooling to room temperature. Such mechanical constraints limit the part's ability to meet structural design fittings, ultimately leading to premature failure during fatigue loading caused by accumulating and propagating dislocations [18,19]. These factors have slowed the adoption of this technology, combined with currently limited means to expand its usefulness and full potential [20].

Currently, research has been inclined towards hybrid material processing, a combination of low-cost conventional processing techniques and additive manufacturing to address some of these shortcomings [11,21]. This combination offers a unique opportunity to reduce costs and parameterization [11]. Several case studies have been made. For example, Ti5Al2.5Sn/Ti6Al4V hybrid system [22] generated a defect-free interfacial bond with

approximately 70 μm of interdiffusion range between the alloys. Similarly, a 316/Cu10Sn hybrid system was reported to have a crack-free interface [23]. However, some reports have highlighted interfacial bonding cracks and undesirable alloy diffusions. A study by Liu et al. [24] observed Cu from C18400 alloy, diffused into the 316L austenitic grain boundaries, and this lowered the ultimate strength due to physical property mismatch. In another study, a brittle Al_2Cu intermetallic phase was produced between AlSi10Mg and C18400. The alloy failure was exacerbated by the variation of thermal coefficients inside the material [25]. Attempts to modify the interface properties were also made on MS1 – H13 [26] and MS1-C300 [5] alloy systems to eliminate microstructural heterogeneity between the substrate and printed alloys by designing suitable heat treatments that incorporated solution treatment and aging. These reports offer a glimpse of how a combination of traditional manufacturing techniques (milling, casting, etc.) and AM could potentially provide new manufacturing solutions set to overcome challenges relevant to mass production, improved geometrical complicity, accuracy, and damage repair using net-shape construction [4,15]. Needless to say, there are inherent problems in these hybrid solutions that arise due to complex thermal histories, involving directional heat extraction, varying coefficients of thermal expansion (CTE), repeated melting, and fast localized solidification [21]. Dissimilar metal joining in additive manufacturing often inherits alloy elemental variations, inhomogeneous microstructures, and weak interfaces similar to welding [27]. Minor alterations to an alloy's prescribed composition or thermal history significantly alter its performance and properties, such as resilience, toughness, and resistance to corrosion.

More recently, M789 steel, a corrosion-resistant maraging steel with tensile strength properties of around 1800 MPa [4,28], was successfully printed on wrought N709, a precipitation-hardening steel in the PH 13-8 Mo family [7]. After testing the bonding between the two materials, this dual alloy achieved a tensile strength of about 1600 MPa after direct aging treatment. In both the as-printed and directly aged state, failure was detected away from the interface indicating a robust interface. In maraging steels, Ti and Al play critical roles in forming strengthening precipitates with Ni in different stoichiometric combinations, such as the ordered NiAl phase and $\text{Ni}_3(\text{Ti}, \text{Al})$ phase [29,30]. The formation of the NiAl phase has been proposed to form from solute-rich clusters inside the lath martensite matrix with spherical morphology. Conversely, Ti-containing maraging steels prefer creating a coherent $\text{ETA-Ni}_3(\text{Ti}, \text{Al})$ (η -phase) phase at very low incubation periods [31,32]. The robustness of the M789-N709 interface sparked the interest to perform an in-depth study on the M789-N709 interface and texture evolution, identifying its chemistry and how it influences the strengthening precipitation behaviour. The new insights regarding the properties of the M789-N709 interface are crucial in determining whether the wrought N709 can be utilized as a cost-effective base material to minimize the use of AM technology and M789 alloy in fabricating plastic injection dies with complex geometries.

4.3 Experimental Procedure

4.3.1 Materials and Manufacturing

An EOS M290 machine was utilized to manufacture the hybrid LPBF-M789 on N709 steel. The N709 metal was set up on the base plate and embedded in the powder chamber at the same level as the powder delivery roller. The N709 was supplied as wrought material, and

the M789 powder was produced by means of the gas atomization process. The powder flowability of the M789 powder was determined to be 15.84s/50g using a Hall Flowmeter. The distribution of particles as measured by the CAMSIZERX2 digital image processing particle size analyzer indicated a particle size in the range of 15 to 60 μm . The chemical compositions of the individual M789 powder and N709 base plate, as specified by the manufacturer and measured using an inductively coupled plasma (ICP), are presented in **Table 4.1**. The M789 powder was then deposited layer upon layer on the N709 base metal using the LPBF process. During the fabrication, the volumetric laser energy density was kept in the range of 75-85 J/mm^3 . The laser power was maintained at 250-270 W, and the scanning speed was held between 1050-1200 mm/s. The hatch distance was held between 0.10 and 0.11 mm, and a layer thickness of $\sim 40 \mu\text{m}$ was used. 10 mm cubic samples were prepared for crystallographic texture characterization. Then, selected as-printed coupons were subjected to direct aging treatment at 773 K (500 °C) for 3 hours, typical for M789 material [4,28]. The aging treatment process was performed under an argon atmosphere to reduce oxidation, and samples were air-cooled to room temperature. The as-printed and aged cubic samples were partitioned parallel to the building direction, followed by sequential grinding up to 1200 grit SiC abrasive papers and standard fine polishing up to 0.02 μm silica suspensions.

Table 4.1. The chemical compositions of the M789 powder and N709 steel (in wt.%)

Steel	C	Cr	Ni	Mo	Al	Ti	Fe
M789	<0.02	12.20	10.00	1.00	0.60	1.00	Balance
N709	0.03	12.70	8.10	2.20	1.10	-	Balance

4.3.2 Materials characterization

The polished as-printed and aged samples were initially analyzed using an electron backscatter diffraction (EBSD) technique to extract the microtexture crystallographic

information on M789, N709 and the interface. This was accomplished by utilizing a Hitachi SU-70 electron microscope equipped with Channel 5™ software. Then, electron diffraction spectroscopy (EDS) was performed using a JOEL 6010 electron microscope equipped with an EDS detector on each sample to quantify the changes in element composition across the interface. To acquire an in-depth information regarding the compositional gradient of the M789-N709 hybrid system, the samples were further examined using an electron probe microanalyzer (EPMA) at 10 µm spacings for both as-printed and directly aged samples using a JEOL JXA-733 electron microprobe. The EPMA was operated with a probe current of 50 nA and an accelerating voltage of 20kV. The backscattered electron images were collected with the aid of dPict32 v.4.18 (Geller MicroAnalytical Labs). Microprobe automation was provided by dSpec v. 4.36 and dQant32 v. 3.68 (Geller MicroAnalytical Labs). The observations from EDS and EPMA were compared with thermodynamic simulation provided by JMatPro v12 (Fe module) simulation software. Vickers hardness was evaluated using a Nanomechanics iMicro machine. Finally, to further confirm the results from EDS, EPMA and thermodynamic simulation, an atomic probe tomography (APT) analysis was performed using a Local Electrode Atom Probe (LEAP 5000XR) instrument running CAMECA Integrated Visualisation and Analysis Software (IVAS) version 3.8.4.

4.4 Results and discussion

4.4.1 Variation of alloying elements from M789 to N709

The concentration profiles of each alloying element at the interface of M789-N709 were extracted from EPMA and EDS results, as presented in **Figs. 4.1** and **4.2**, for as-printed and directly aged samples, respectively. **Figs. 4.1a** and **4.1c** show the EPMA scan, while

Figs. 4.1b and **4.1d** display the EDS scan across the interface of the as-printed hybrid sample. Note that major and minor alloying elements were separated (based on weight percentage). This was done to examine the preferred distribution of alloying elements at the transition region and explain the mechanism of the previously reported tensile strength of 1600MPa of the M789-N709 hybrid system (accompanied by a robust interface) in the previous work of the present authors [7]. The overall chemical composition from both EPMA and EDS measurements agrees with the sample chemistry presented in **Table 4.1** [4]. Of particular interest is Ti present in M789 (see **Figs. 4.1a** and **4.1b**), which promotes the precipitation of the $\text{ETA-Ni}_3(\text{Ti, Al})$ during aging [4,28]; this phase can provide a robust interface stronger than the base materials.

The EPMA and EDS concentration profiles for Ti show a maximum concentration of 1.1 wt.% (see **Fig 4.1a**) and 0.98 wt. % (see **Fig. 4.1b**), respectively. A sharp change in Ti content can be observed, suggesting minimal diffusion from N709 to M789 with a diffusion gradient of approximately 0.004 Ti wt. %/ μm . With the fast cooling rates during the LPBF process, short diffusion distances are expected for the as-printed sample [3]. Both the EPMA and EDS show the partial melting and dilution phenomenon during the initial deposition of M789 [32–34]. The effect of this dilution is approximated to be within 300 μm and diminishes with subsequent M789 layers. This accounts for about 8 layers in the fusion zone since each powder layer is approximately 40 μm . This creates a transition region with properties unique to the parent alloys (i.e. N709 and M789).

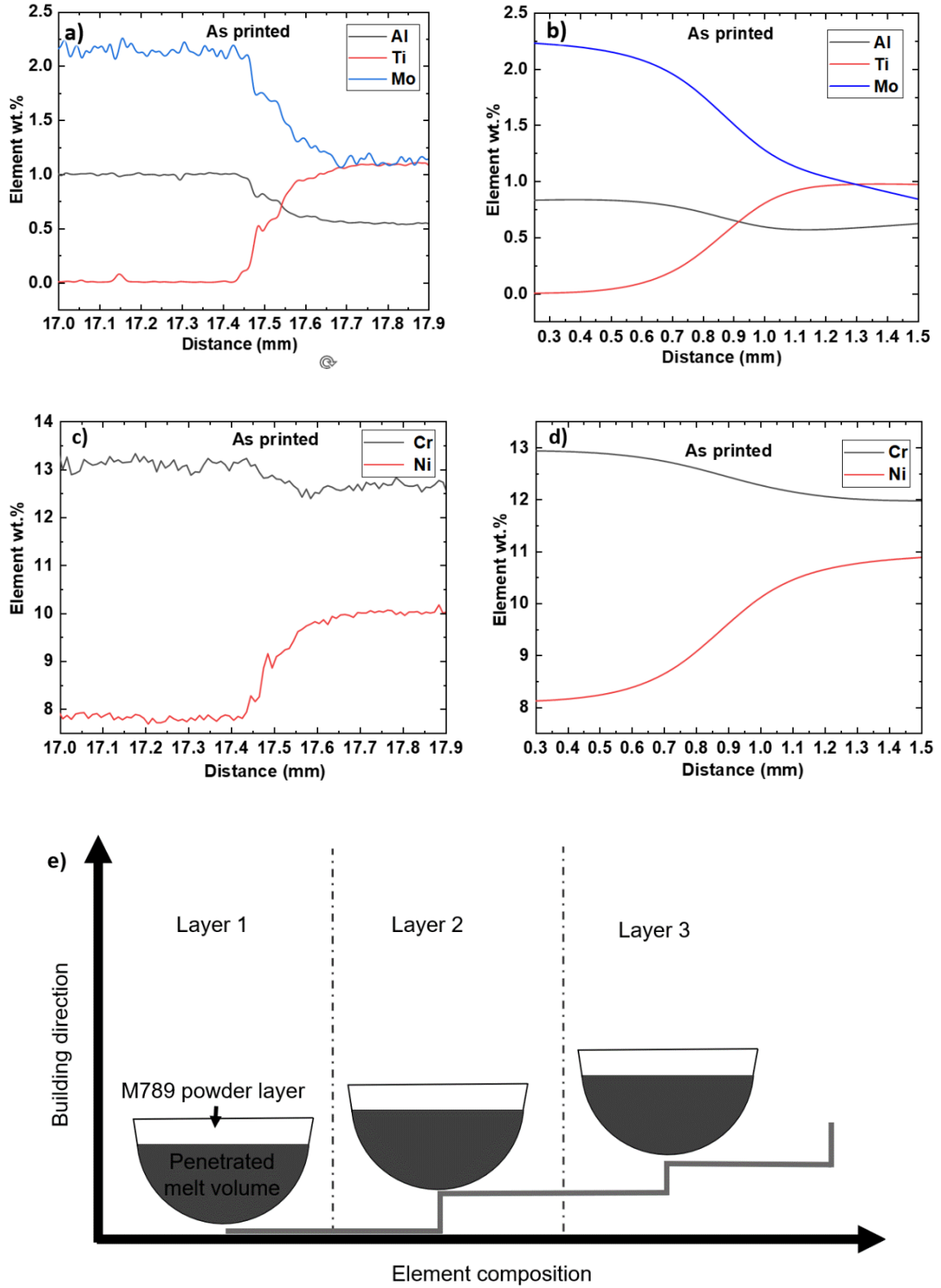
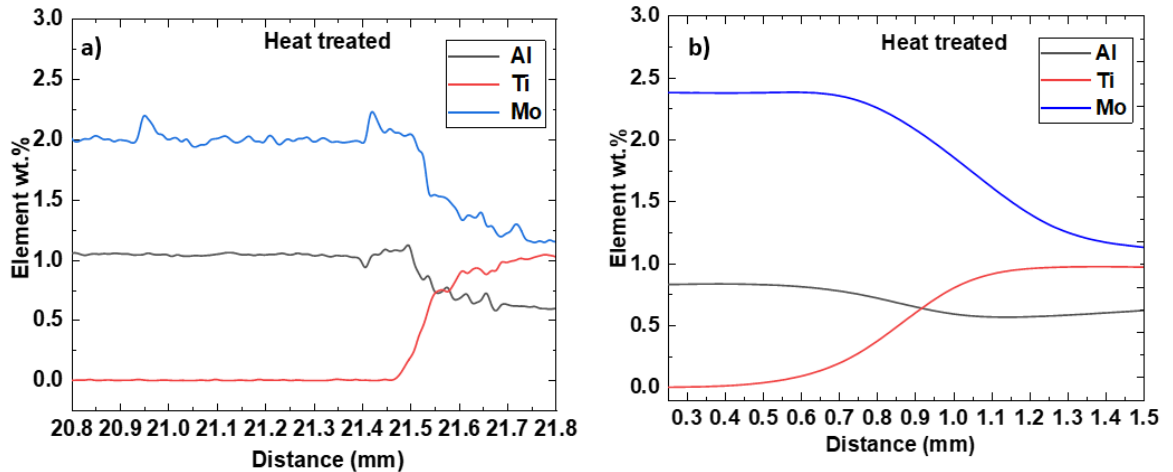


Figure 4.1. The compositional gradient of the as-printed M789-N709 alloy is presented: a, b) EPMA and EDS analysis of Al, Ti, and Mo, c,d) EPMA and EDS analysis of Cr and Ni, and e) schematic of the compositional gradient change of mixed deposited layers.

In the previous work, the interface was reported to have a fine microstructure corresponding to the high cooling rates that favour nucleation [7]. The Marangoni flow facilitates the mixing of alloying elements, which can potentially segregate heavier alloying elements such as Mo along grain boundaries. The efficiency of bonding is determined by the degree of wettability of the molten alloy on the substrate, and it facilitates fast nucleation due to epitaxial solidification [32,35]. The concentration profile differences of Cr and Ni across the M789 and N709 sections are almost similar, with less than 4% difference in composition (see **Figs. 4.1c** and **4.1d**), while for Mo and Al, it is approximately 50%. Cr has been reported to diffuse faster than Ni and Mo in Fe [5]. However, since the composition of Cr in both M789 and N709 is similar, the concentration profiles only show a slight dip. With the fast cooling rates associated with LPBF, a minimum amount of diffusion is expected during the process; however, the dilution mechanism plays a major role in producing concentration gradients observed in the composition profiles [36] (**Figs. 4.1c-4.1d**). The gradual change in the composition resulted in interface chemistry composed of the deposited M789 diluted with N709 substrate, as represented in **Fig. 4.1e**. The penetrated metal layer is diluted with the added M789 powder along the building direction. This continues until the remelted depth is completely M789. The composition of each mixed layer is presented as a stepped composition along the interface and is shifted by the corresponding penetration depth.

After direct aging, some changes can be observed in the elemental concentration obtained from EPMA results in **Figs. 4.2a** and **4.2c**. The corresponding EDS profiles in **Figs. 4.2b** and **4.2d** show a similar trend with the EPMA profiles, with a minor difference in the volume fraction of detected Cr and Mo. Although Mo segregation can be associated with

the localization of metastable orthorhombic Ni_3Mo phase that occupies the $\langle 111 \rangle$ of the martensitic matrix [37,38], this phenomenon was not detected in this work through multi-scale characterization techniques presented in the later sections. Therefore, it can be inferred that the fluctuations detected in EPMA are due to the locality of elemental measurement rather than segregation (note that EDS measurements of both Cr and Mo agree with the results of the as-printed sample). Moreover, since the η -phase exhibits no solubility for Cr and Mo, it is possible that they were expelled from the precursor phase resulting in elemental fluctuations at the precipitate-matrix interface [31]. At the aging temperature of 500 °C for 3 hours, there is no significant diffusion of alloying elements. Nevertheless, if solution treatment had been carried out at temperatures of 1000 °C, some diffusion would be expected alongside grain coarsening. Considering the dilution region for Ti, its diffusion distance is estimated to be within the transition region of 200 μm , and the concentration gradient remained the same as before.



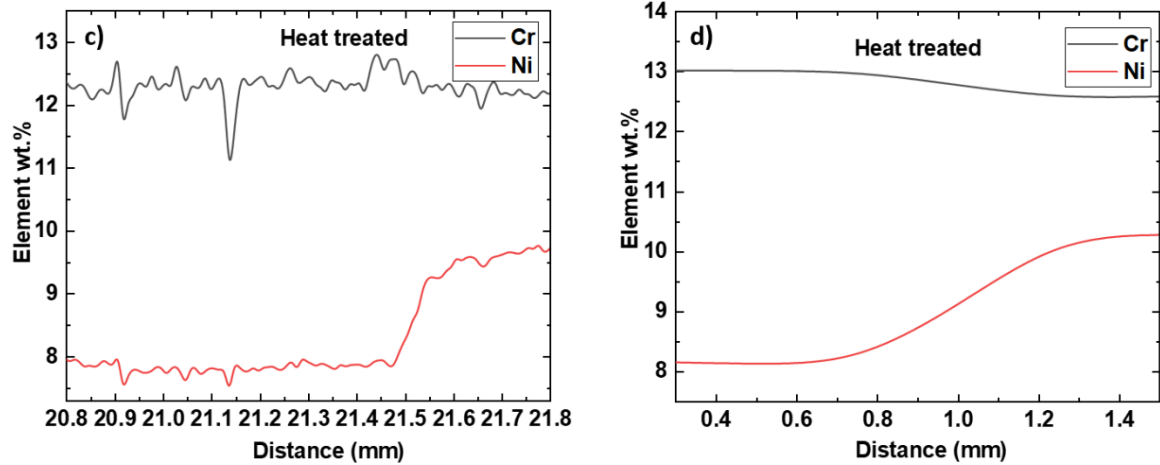


Figure 4.2. The compositional gradient of the directly aged M789-N709 alloy is presented: a, b) EPMA and EDS analysis of Al, Ti, and Mo, c,d) EPMA and EDS analysis of Cr and Ni.

4.5 Grain misorientation before and after aging treatment

The microstructural features of the interface are distinct in both the as-printed and aged samples; it consists of fine grains with a size of less than 10 μm (see **Figs.4.3** and **4.4**) compared to the M789 and N709 sections with grain sizes of approximately 10 μm and 15 μm , respectively. A fully martensitic structure was formed in the as-printed state, with less than 1% retained austenite (see **Fig. 4.3b**). As previously reported, a robust interface was obtained in both the aged and as-printed states, with failure detected away from the interface [7]. Good wetting and dilution are necessary for efficient bonding and epitaxial solidification between the substrate and the deposited M789 [35]. A melt back of the previously formed layer is required for microstructural continuity throughout multiple successively melted and solidified metal layers. During initial deposition, homologous wetting [32] is expected since the deposited M789 has a similar chemical composition to

N709.

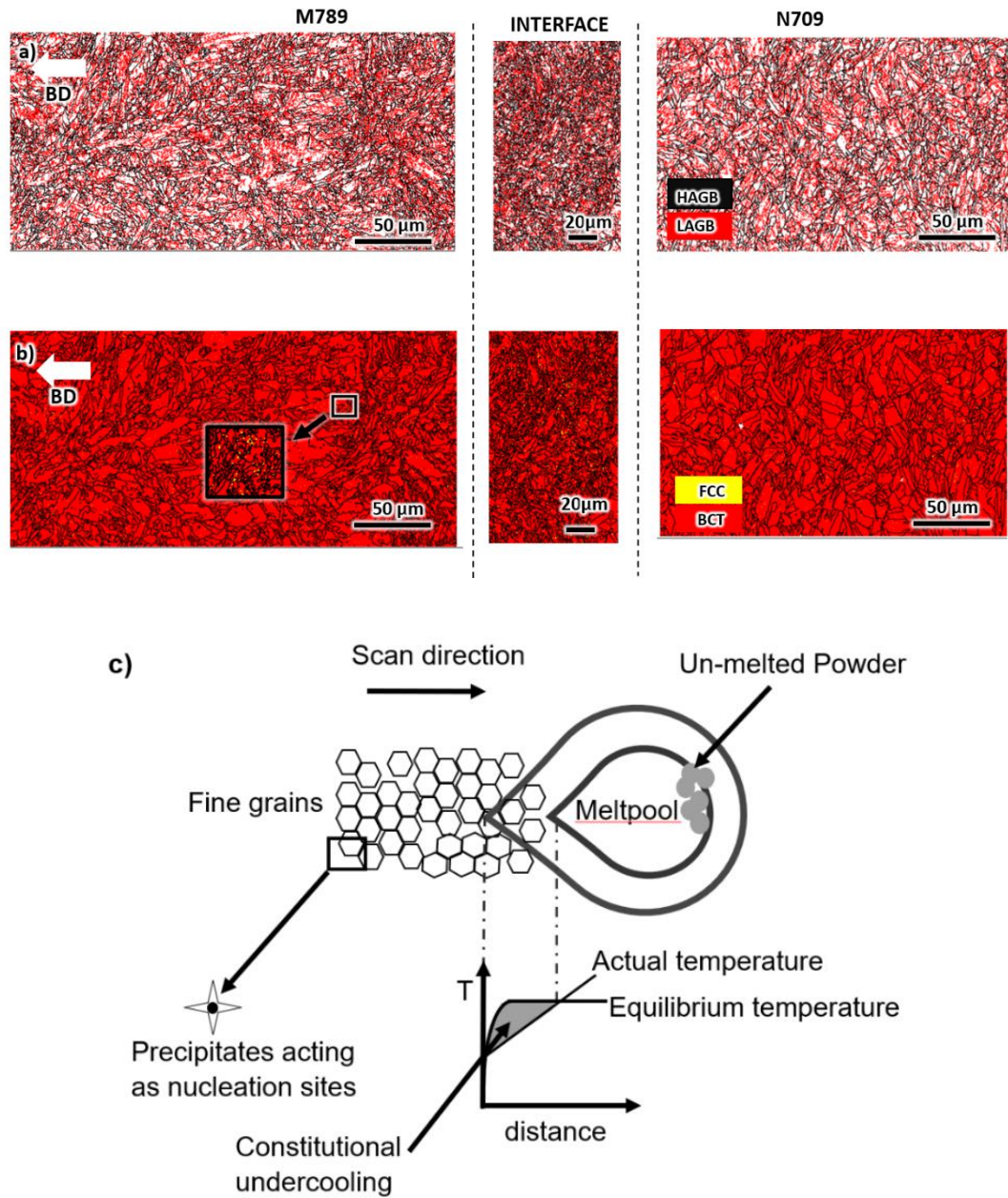


Figure 4.3. The EBSD results showing: a) grain boundary, (b) phase maps associated with the as-printed M789-N709 hybrid system, and c) mechanism for nucleation at the interface.

The interface is characterized by very fine grains, and this could be the combined effect of a small critical radius for nucleation (r^*) and low Gibbs energy (ΔG^*) [39]. The energy

barrier to epitaxial solidification is minimized, and nucleation takes place rapidly [32]. Solidification is most favoured when temperatures are lower than the equilibrium temperature with an increased degree of undercooling (see **Fig. 4.3c**). The undercooling is increased by the high scanning speed and the presence of substrate material that acts as a heat sink, promoting a faster rate of nucleation. In addition, calculations for the critical radii for nucleation were estimated to be between 2nm to 4nm for undercooling that is within 250 to 450 °C. Further grain refinement at the interface is achievable because of the presence of precipitates in the N709 that act as nucleation sites further decreasing the energy barrier for heterogeneous nucleation [40]. With the contribution of a fast-cooling rate as the laser melts the surface, well-refined grains are produced, as seen in **Figs. 4.3** and **4.4**. As the M789 layer was deposited beyond the interface, the grains adopted a columnar structure growing parallel to the build direction following the heat gradient [41]. From **Fig. 4.3a**, low angle grain boundaries (LAGBs) are measured at 74.80%, 65.94%, and 77.39% for M789, interface and N709 sections, respectively. In this work, LAGBs are grains with grain misorientation between 2° and 15°. The regions with LAGBs are typically associated with the dislocation density in the material. Comparing the results of the as-printed to aged sample, the latter in **Fig. 4.4a** displayed an amount of LAGB as follows; 73.14%, 63.04% and 77.38% for M789, interface and N709 sections, respectively. The LAGB values show a 2% change in the dislocation density after direct aging. Also, the phase remained primarily martensitic (see **Fig. 4.4b**) after direct aging.

The LAGBs and phase maps in **Figs. 4.3** and **4.4** are complemented with the Kernel Average Misorientation (KAM) maps shown in **Figs. 4.5** and **4.6**. These maps display the intergranular misorientations in the M789, interface, and N709 sections.

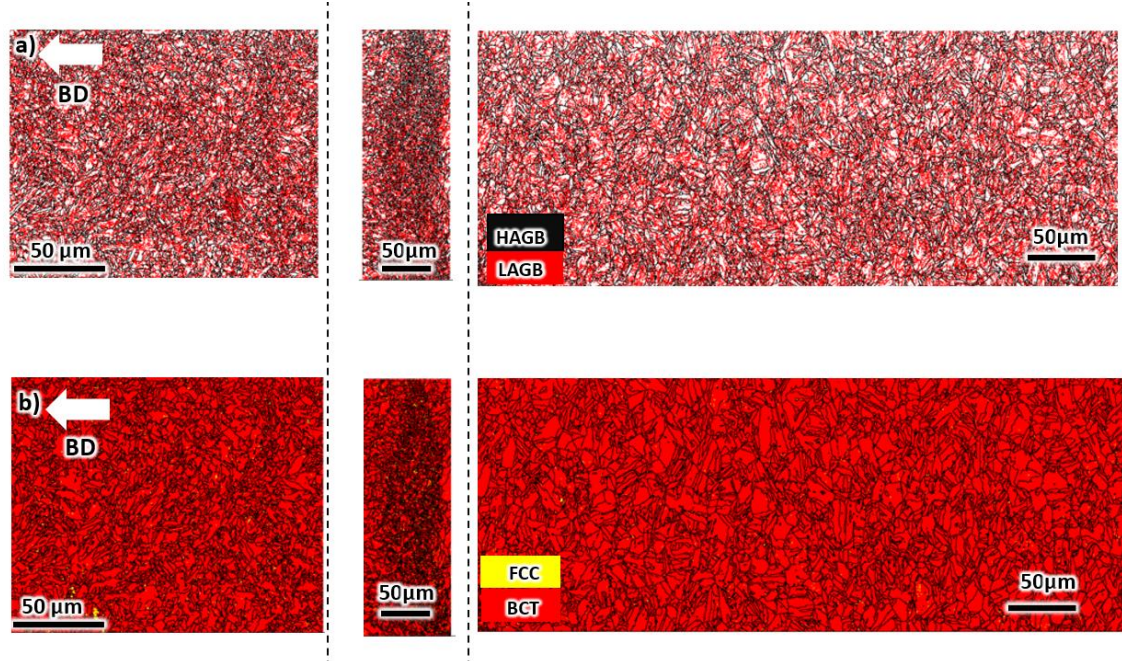


Figure 4.4. The EBSD results showing: a) grain boundary, and (b) phase maps associated with the directly aged M789-N709 hybrid system.

Similar to LAGBs, the misorientations in KAM maps are also related to the dislocation density, and they reveal the distribution of mesoscopic strain [42] within the alloy and are measured up to 5° . A uniform distribution of local misorientation is presented in most regions with low KAM values ranging from 1.5 to 2.5° . The grain interiors are marked with low misorientations. This is observed throughout the whole sections of the as-printed sample, including the wrought N709 substrate. Furthermore, the directly aged sample (see **Fig. 4.6**) depicts a similar trend of lattice gradient related to the lattice distortion caused by dislocations. Analysis using KAM maps reveals a more obvious change in the dislocation density compared to the LAGB calculations with 40% local misorientation with angles greater than 2° in the as-printed state and 36% after heat treatment. It appears from the calculated LAGB values and KAM maps that directly aging this hybrid system does not

eliminate all dislocations. The existing dislocation density (uniformly distributed within the martensitic matrix) fosters an improved age-hardening response in maraging steels by offering substantial nucleation sites for intermetallic precipitates formed during aging. Furthermore, the same dislocation can potentially act as low-energy diffusion paths. However, with high solution treatment temperatures, the dislocations are expected to lessen, possibly reducing the mechanical properties due to grain coarsening [26,28].

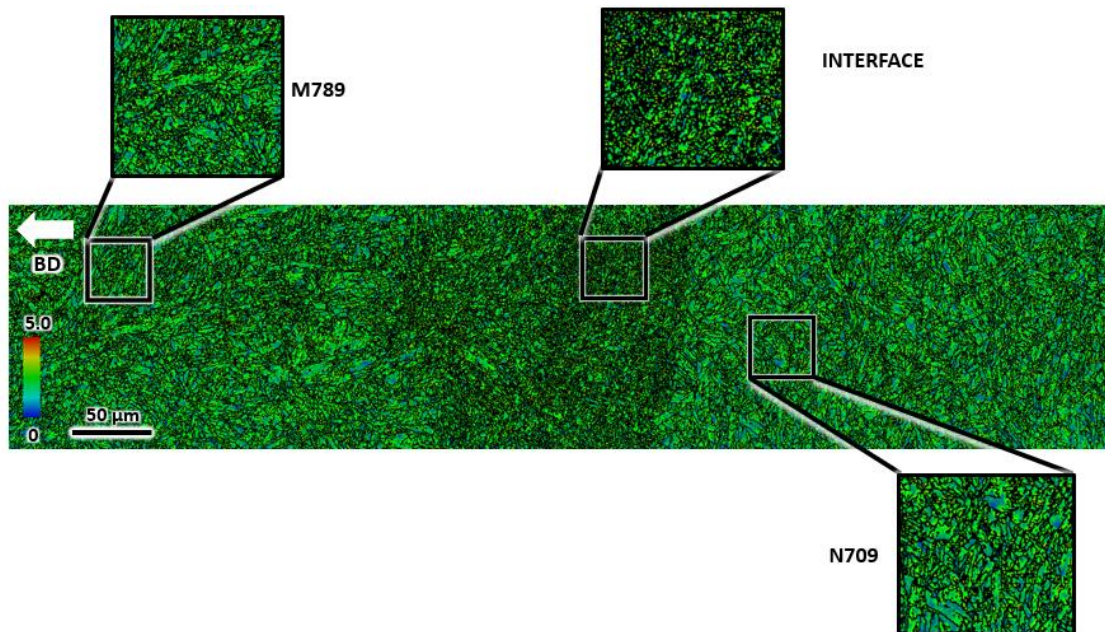


Figure 4.5. The Kernel Average Misorientation (KAM) map obtained from as-printed M789-N709 hybrid alloy.

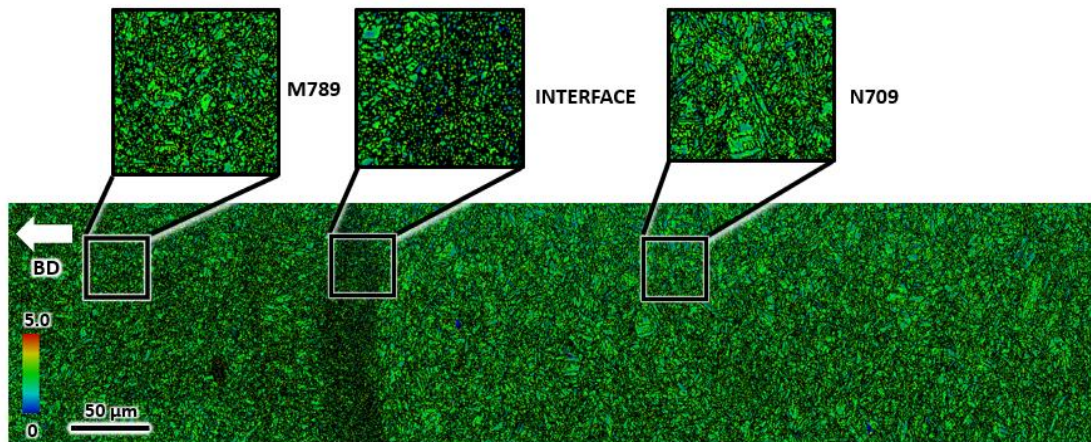


Figure 4.6. The Kernel Average Misorientation (KAM) map obtained from directly aged M789-N709 hybrid alloy.

4.6 Crystallographic Texture analysis

The crystallographic texture of the as-printed and directly aged samples measured using EBSD analysis are presented in pole figures in **Figs. 4.7 and 4.8**, respectively. **Fig. 4.7a** (M789 region) shows a weak texture of $\langle 111 \rangle$ parallel to the building direction (BD), consistent with the previous reports [4,7,28]. The maximum texture intensity was detected at 3.5. The weak texture further diminishes in the interface (with a maximum intensity of 2.3), as shown in **Fig. 4.7b**. The substrate exhibits a random texture with no preferred orientation with a measured maximum texture intensity of 2.2. Randomness in texture can be attributed to the scan strategy used that constantly changes the heat flux direction avoiding a dominant texture along the easy $\langle 100 \rangle$ growth direction [41]. In the directly aged sample, the texture appears to maintain its randomness, which has a weak $\langle 111 \rangle // \text{BD}$. The display of this weak texture can be seen in **Figs. 4.8a and 4.8b**, with a maximum texture intensity of less than 2. The precipitate phases formed appear to be coherent with

the parent phase and exhibit the randomness presented in the pole figures. With this random texture, isotropic mechanical properties are expected.

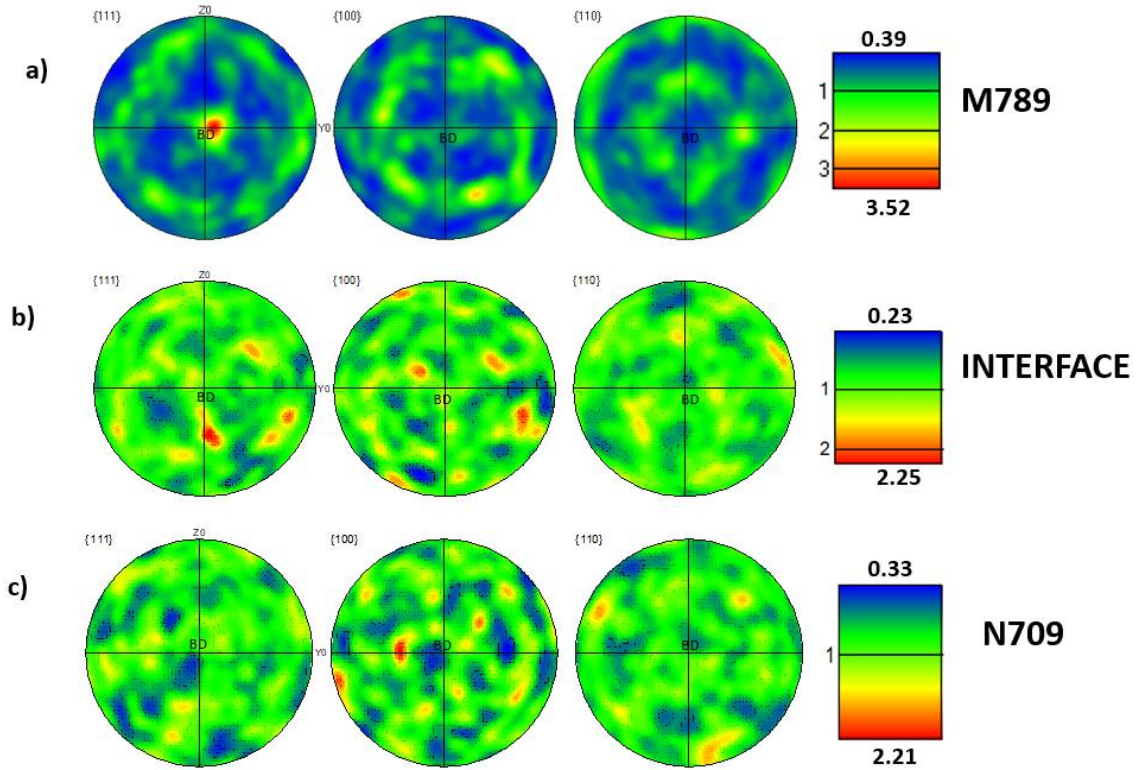


Figure 4.7. The pole figures of the: a) M789, b) M789-N709 interface, and c) N709 sections in the as-printed state.

4.7 Titanium profile at the interface of M789-N709 alloy

As discussed in the previous section, M789 and N709 have similar alloying elements except for Ti content, which varies from the interface to the M789 section, as shown in the EPMA results. The change in Ti across the interface (with about 300 μm thickness) is depicted in **Fig. 4.9**, displaying the values taken every 10 μm . The presence of Ti was previously established to be responsible for the formation of the $\text{ETA-Ni}_3(\text{Ti, Al})$ in M789, amounting to 5% post-aging [7,43].

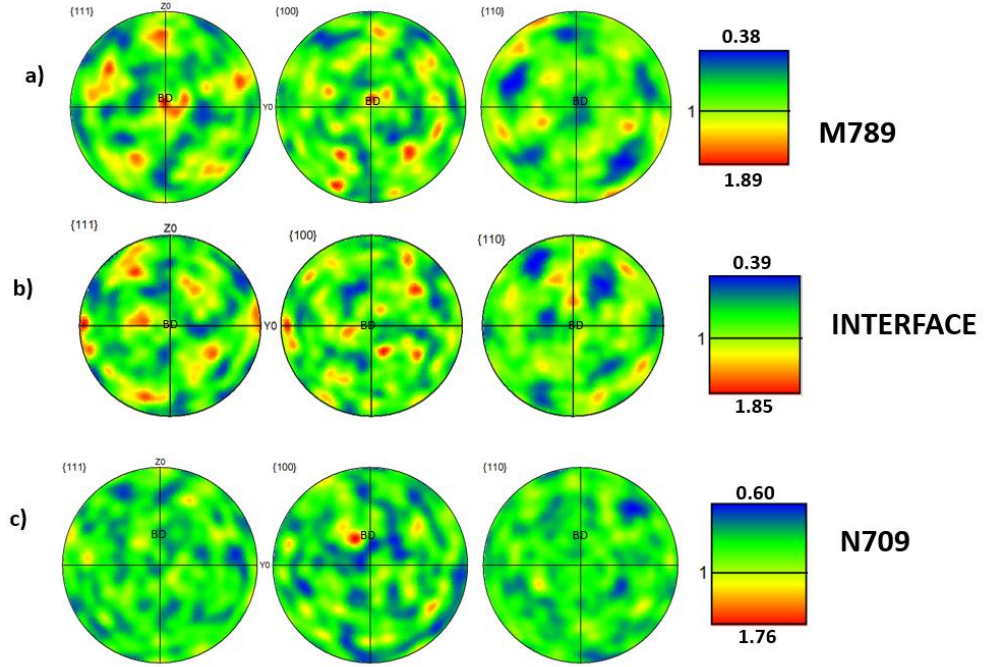


Figure 4.8. The pole figures of the: a) M789, b) M789-N709 interface, and c) N709 sections in the directly aged state.

This phase has rod-like features with a hexagonal $D0_{24}$ structure with Ti atoms occupying the close-packed (0004) planes in a triangular manner [44].

To further determine the Ti gradient in the interface, a precipitation simulation was performed using the JMatPro simulation software, applying the wt.% Ti measurements obtained from the EPMA results. The results revealed that the maximum volume percentage of the $\text{ETA-Ni}_3(\text{Ti, Al})$ corresponds to the highest wt.% Ti of 1.02 wt.%. Simulations are based on the Johnson-Mehl-Avrami equation, which assumes a spherical morphology and isothermal conditions, as shown in Equation (4.1):

$$x = 1 - e^{\left(-\frac{\pi}{3} N_r G_r^3 t^4\right)} \quad (4.1)$$

where x is the volume fraction of the precipitate during solid-state transformation, N_r and G_r are the nucleation and growth rates, respectively, at heat treatment time t . It is well known that Ti addition in maraging steels lowers the martensite temperature and increases the amount of retained austenite and twinned martensite [45]. As such, its addition must be controlled to promote the formation of the ETA-Ni₃(Ti, Al), which has high strength properties at low aging temperatures [46]. Hence, the volume of the ETA-Ni₃(Ti, Al) is expected to increase with Ti additions. The hardening effects of Ti are limited to additions up to 1.2 %. A composition more than this has been known to reduce ductility [45]. As observed from the simulation results (see **Fig. 4.10**), the volume of precipitates appears to be linearly dependent on wt.% of Ti.

To effectively observe the individual changes in the evolution of the ETA-Ni₃(Ti, Al), the exact compositions at each point in the interface (treating them as individual alloys) were considered based on the wt.% of Ti. The results of these simulations are presented in **Figs. 4.11-4.13**. The amount of ETA phase precipitates increases with aging time and the wt.% of Ti. For concentrations of Ti ranging from 0 wt.% to 0.0067 wt.%, as detected by the EPMA, display no evolution of the ETA-Ni₃(Ti, Al), as presented in **Fig. 4.11a**. However, there is an effective strength contribution of (Fe, Ni)Al/B2 phase, which has a volume presence of about 3.38 wt.%. The volume percentage of this phase is similar to that of the N709 substrate, which was previously reported to have a dominant (Fe,Ni)Al phase amounting to 3.5% [7]. The presence of Ni and Al in this steel grade has been reported to promote the formation of β -NiAl precipitates enclosed in lath martensite at aging temperatures between 450 °C to 575 °C [46].

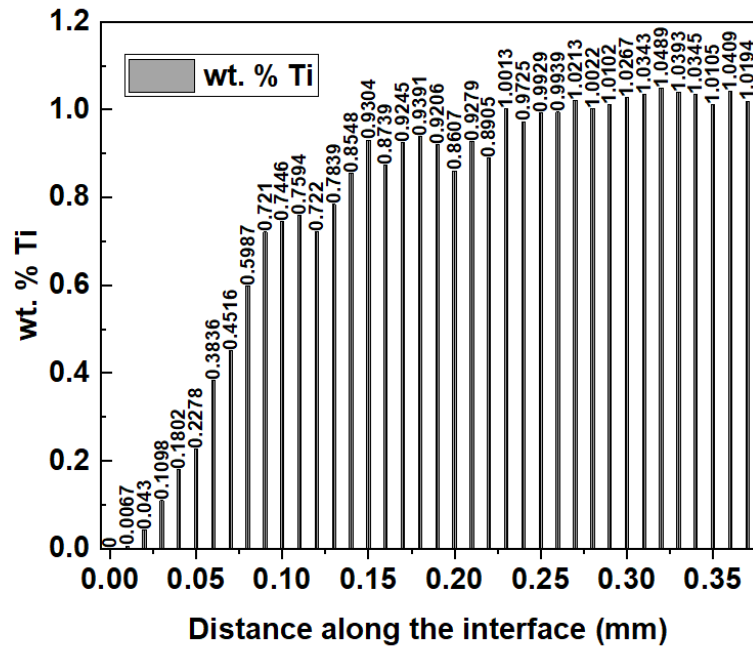


Figure 4.9. The measured variation of Ti content in the interface of M789 and N709.

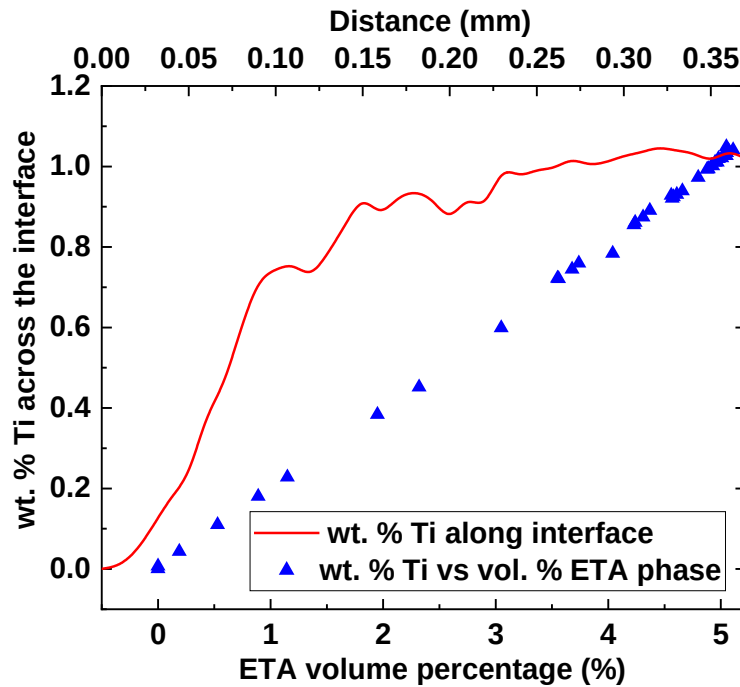


Figure 4.10. The simulated evolution of precipitates with change in Ti composition across the interface using the experimentally measured titanium content.

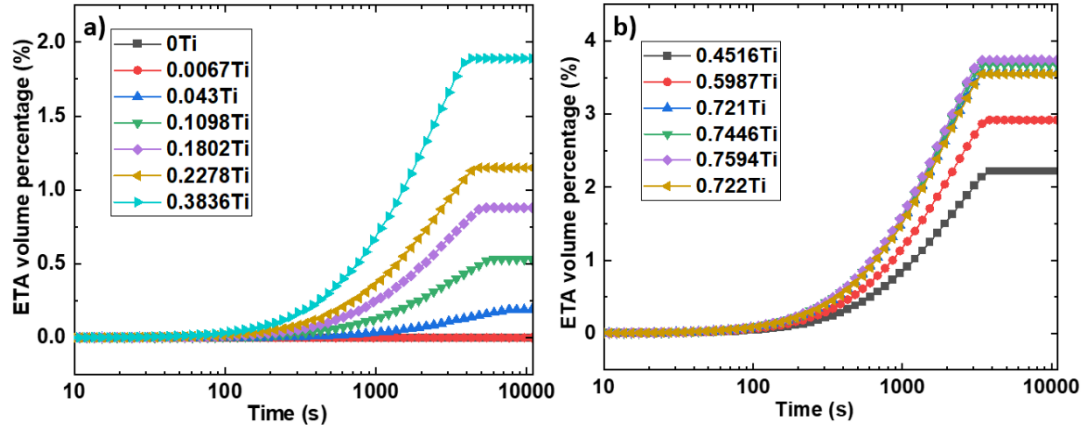


Figure 4.11. ETA-Ni₃(Ti, Al) (η -phase) evolution with Ti change at the interface from 0 wt.% to 0.722 wt.%.

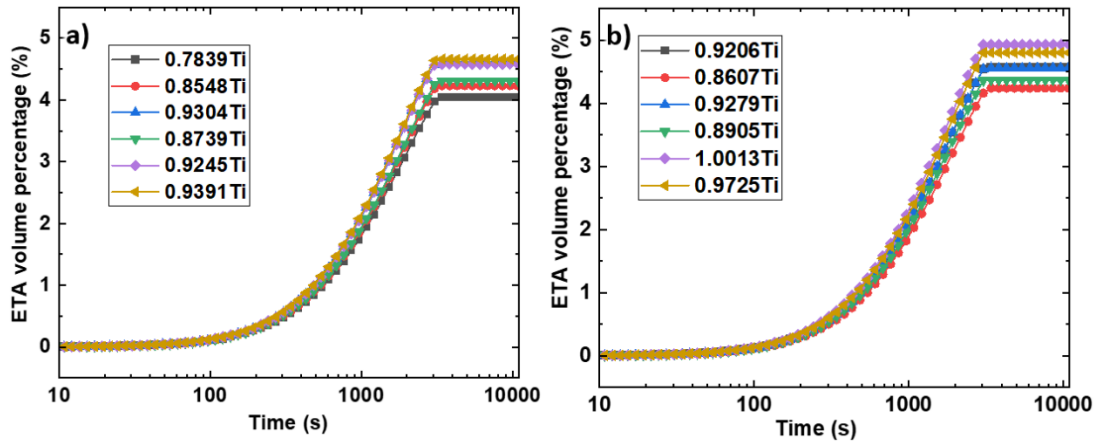


Figure 4.12. ETA-Ni₃(Ti, Al) (η -phase) evolution with Ti change at the interface from 0.7839 wt.% to 0.9725 wt.%.

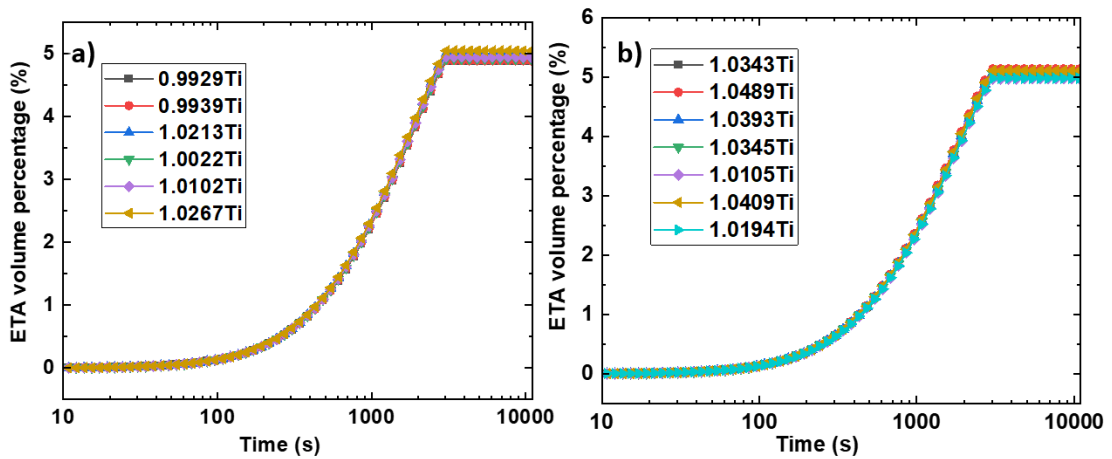


Figure 4.13. ETA-Ni₃(Ti, Al) (η -phase) evolution with Ti change at the interface from 0.9929 wt.% to 1.0194 wt.%.

The high B2 phase volume percentage corresponds to an Al content of between 1.1 wt.% to 1.2 wt.%. At this composition, Ni and Al have strong interactions as they tend to form B2 intermetallics [46]. From **Figs. 4.11a** and **11b**, the increase in Ti content provides a significant increase in ETA-Ni₃(Ti, Al). However, from **Fig. 4.12a** to **Fig. 4.13b**, it can be noted that the addition of Ti only provides a slight increase in the percentage of ETA-Ni₃(Ti, Al).

Further addition of Ti can be seen to increase the volume of ETA-Ni₃(Ti, Al), as noted above, while diminishing the (Fe, Ni)Al, as presented in **Fig. 4.11b** and **Fig. 4.14**, respectively. Based on Mossbauer spectroscopy, it was proposed that Ti-rich clusters act as inter-metallide nuclei, which assist in the formation of the ETA-Ni₃(Ti, Al) [47]. These rich clusters have been reported [37] to expel other accompanying alloying elements such as Mo and Fe in preference for the low formation energy of the ETA-Ni₃(Ti, Al). In **Fig. 4.14**, some anomalies in B2 compositions have been observed (marked in blue circles). The value of the (Fe,Ni)Al was seen to divert from the observed diminishing tendency (up to 0.8485 wt.% Ti). This is due to higher-than-normal Al concentrations that have been measured (approximately 1.1666 and 0.914 wt.%). More NiAl variants are formed when Al is added to Fe-Ni-Mn-Al-Ti at low Ti concentrations.

The (Fe,Ni)Al phase completely diminishes at approximately 0.8905 wt.% Ti at which the wt.% of Al cannot contribute to the formation of the phase. This observation suggests that Ti and Al have an affinity for Ni, with Ti exhibiting a stronger attraction and strengthening capabilities in agreement with Niu et al. [37]. This is verified by the APT results in the subsequent discussion section.

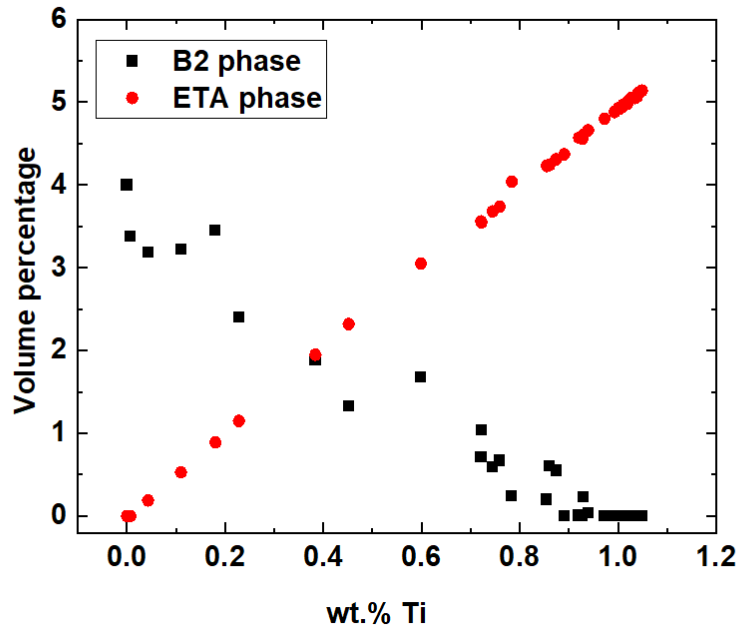


Figure 4.14. The dependence of ETA-Ni₃(Ti, Al) and B2-(Fe,Ni)Al phase on the Ti content in the interface after aging treatment.

4.8 Precipitation in the interface

The precipitation of intermetallic precipitates largely influences the mechanical properties of maraging steels [48]. As seen from **Figs. 4.15a** and **4.15b**. Nano indentations were carried along the interface. Before aging (see **Fig. 4.15a**), the M789 section shows a relatively low hardness between 300 to 350HV, as indicated by the contour map. Conversely, the N709 section shows a higher hardness ranging above 500Hv because it was peak aged before depositing M789. In comparison with the aged state in **Fig. 4.15b**, a more homogenous hardness distribution was observed. Both sections of the interface show some regions with hardness of more than 500HV. Additionally, the size, coherency and distribution of precipitates are crucial in attaining the target mechanical properties [49]. The increase in M789 hardness can be understood further, as shown in **Fig. 4.15c**; the

simulation shows that by increasing Ti content, the final precipitate size tends to increase up to approximately 5 nm. The strengthening effect of these precipitates is achieved from the lattice distortion that these precipitates contribute as they cause expansion and compression effects inside the lath martensitic matrix [37].

Furthermore, to verify the presence, morphology and size of precipitates, an EDS analysis was performed; the results are presented in **Fig. 4.16**. Nanoscale spherical precipitates can be detected, which show the presence of Ti and Ni with nearly similar distribution in **Figs. 4.16a** and **4.16b**. The precipitates are reported to undergo nucleation and growth earlier in the aging stage [50]. The formation of these precipitates depletes Ni within the martensite lattice. Since Ni is a strong austenite stabilizer, the formation of these precipitates minimizes the appearance of reverted austenite [51]. At the same time, it is the main element that constitutes intermetallic precipitates that are formed during aging [46]. Trace amounts of Al-based precipitates can be seen in **Fig. 4.16c**, in agreement with the kinetics simulation that showed a diminishing amount of (Fe,Ni)Al precipitates with increasing Ti content (see **Fig. 4.14**).

The rapid formation of clusters during aging can be accounted to the occurrence of pipe diffusion [8], because of the presence of high dislocation density in the as-printed state. There are no Mo precipitates observed which are often in the form of the Fe_2Mo -laves phase. Since a relatively short aging time is used in the current study (i.e., 3 hours), Mo-rich phases cannot form. However, at extended aging times approaching 40 to 100 hours, large-sized Mo-rich phases can form around the ETA phase as core-shell structures [50]. As a result, the Mo minimizes the diffusion of other alloying elements inside the precipitate.

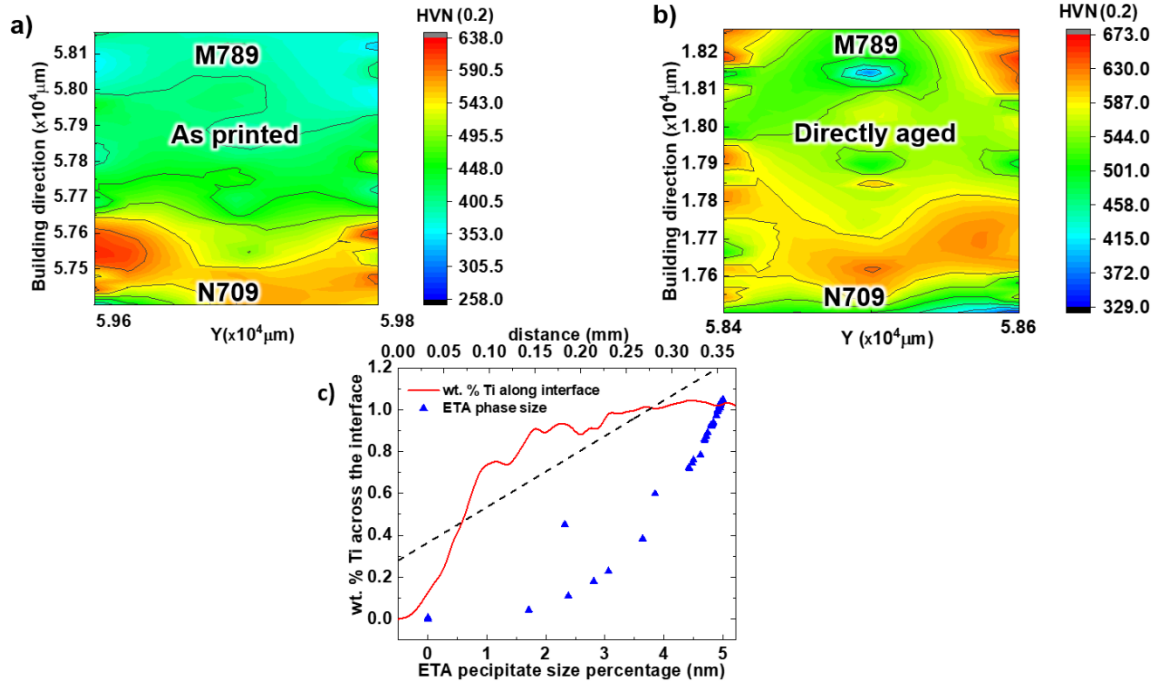


Figure 4.15. a) Nano hardness before direct aging, b) nano hardness after aging, and c) the size of ETA-Ni₃(Ti,Al) precipitate in the interface, including the corresponding Ti content.

To further understand the nature of precipitates, an APT analysis was carried out to show the distribution and morphology of precipitates observed from the EDS analysis. From **Fig. 4.17**, plate and spherical-like precipitates can be observed over an APT volume of 130916 nm³ with lengths averaging between 5nm and 18nm with widths between 2nm to 4nm. The difference in precipitate morphology in the case of spherical precipitates has been proposed to be caused by a lack of time or hindrance of the phase interface at the initial aging stage. On the other hand, precipitates that have minimal interfacial energy are expected to grow beyond spherical precipitates forming rod-like features [50].

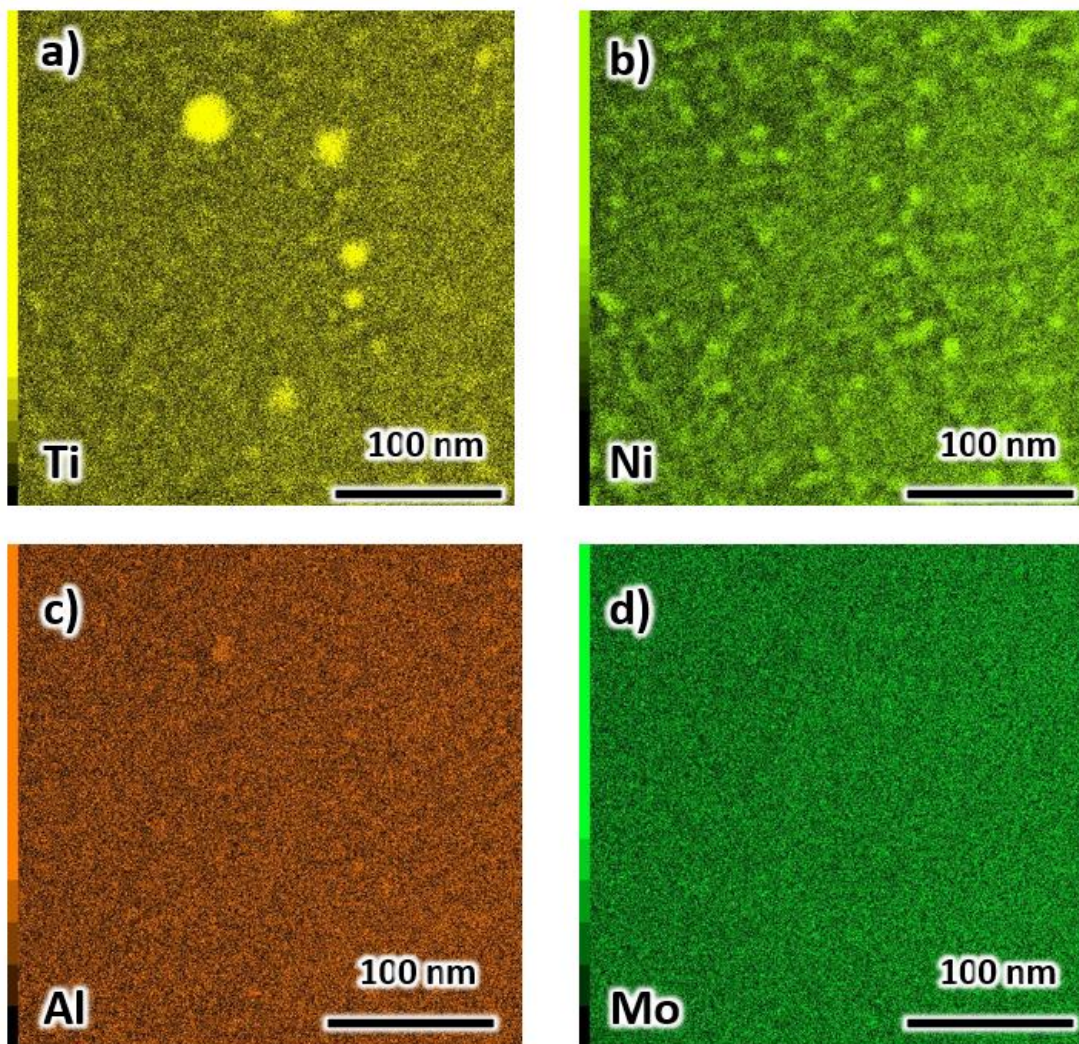


Figure 4.16. EDS map revealing the formation of the precipitate showing the distribution of: a) Ti, b) Ni, c) Al, and d) Mo.

Due to the low atomic percentage of Ti measuring 0.0032 %, the presence of Ti is thought to have been overshadowed by Al with an at.% of 2.98 as measured by the APT. Under this volume, 142 particles were identified and had a density of $1.08 \times 10^{24} \text{ m}^{-3}$. It is worth noting that the APT has some limitations in determining the composition of miniature precipitates because of their different evaporation fields. These fields generate a relatively smaller radial curvature as they appear on the surface of the sample. Subsequently, there is

a localized electric field and ionic path distortion deriving from the matrix, which results in an overlap of ion pathways. This often results in the composition of the matrix reporting in the composition of the intermetallic precipitates [52,53].

In **Fig. 4.18**, proxigrams are generated based on the isosurfaces mapped in green and maroon regions in **Fig. 4.17**. It can be observed that the precipitates contain significant amounts of Ni and Al, and reduced amounts of Fe and Cr. The statistical proximity histogram (corresponds to Ni with 17.5 at.% iso concentration surface of the elongated plates) revealed a composition of Ni- and Al-rich concentrations of 43.2 ± 2.2 at.% and 26.9 ± 1.5 at.%, respectively (see **Fig 4.18a**). On the other hand, at 21 at. % Ni concentration, the composition of the spherical particles was found to have Ni and Al compositions of 56 ± 4.8 at.% and 32.3 ± 5.1 at.%, respectively (see **Fig. 4.18b**). The spherical precipitates show a Ni enrichment of approximately 23 at.%. It appears that Ni concentration in the spherical-shaped precipitates is favoured more than the elongated ones. Furthermore, there is less enrichment of Fe, amounting to 5.5 ± 2.2 at.% in the spherical precipitates compared to $18.8 \pm 1.5\%$ at.% in the elongated precipitates. The distribution of the precipitates and their frequency is represented in the histogram displayed in **Fig. 4.19**. The precipitate size ranges from 1 to 11nm. The radii are given based on the Guinier fit, giving a close resemblance to the precipitate size. The average diameter was 4.39 ± 1.66 nm; this value is comparable with the JMatPro simulations in **Fig. 4.15** in the interface near the M789 section, and the previous work of the current authors [54]. Moreover, the 4nm to 6 nm range occupies the highest frequency of particle size distribution.

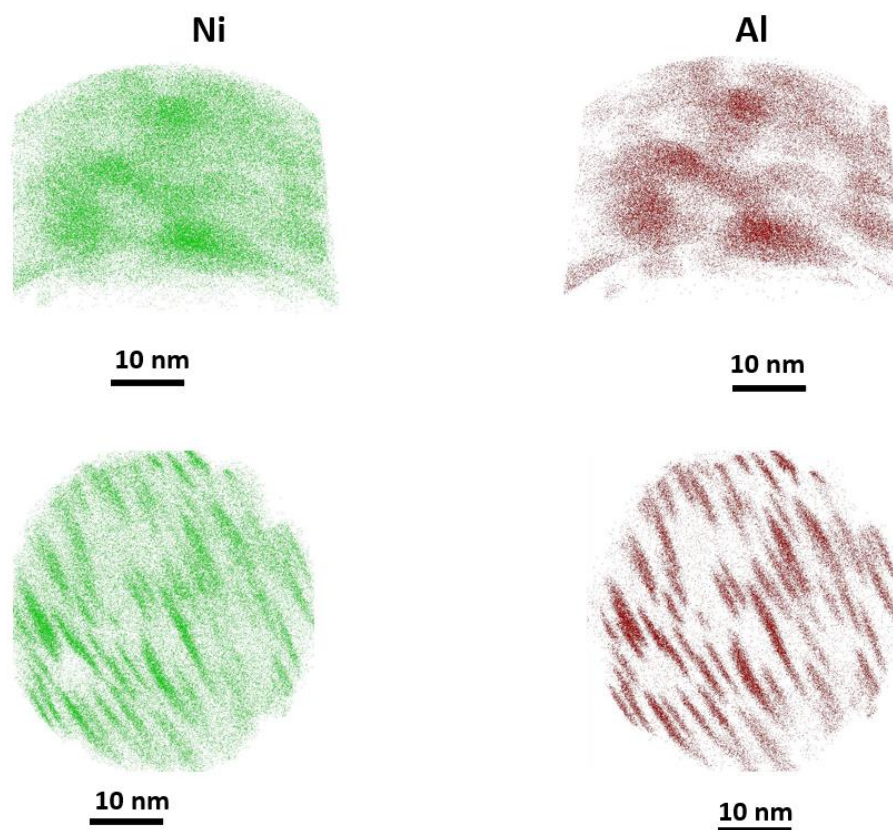


Figure 4.17. The atom probe tomography (APT) results display the distribution and morphology of precipitates close to the M789 region.

The nucleation and growth of precipitates are dependent on the segregation energy and the degree of diffusion of the solute atoms involved, and the interaction energy associated with the alloying elements. From the robustness of the interface, a combined effect of different sizes and morphologies of precipitates (presented in this work) contributes to the strength of the current hybrid system [46].

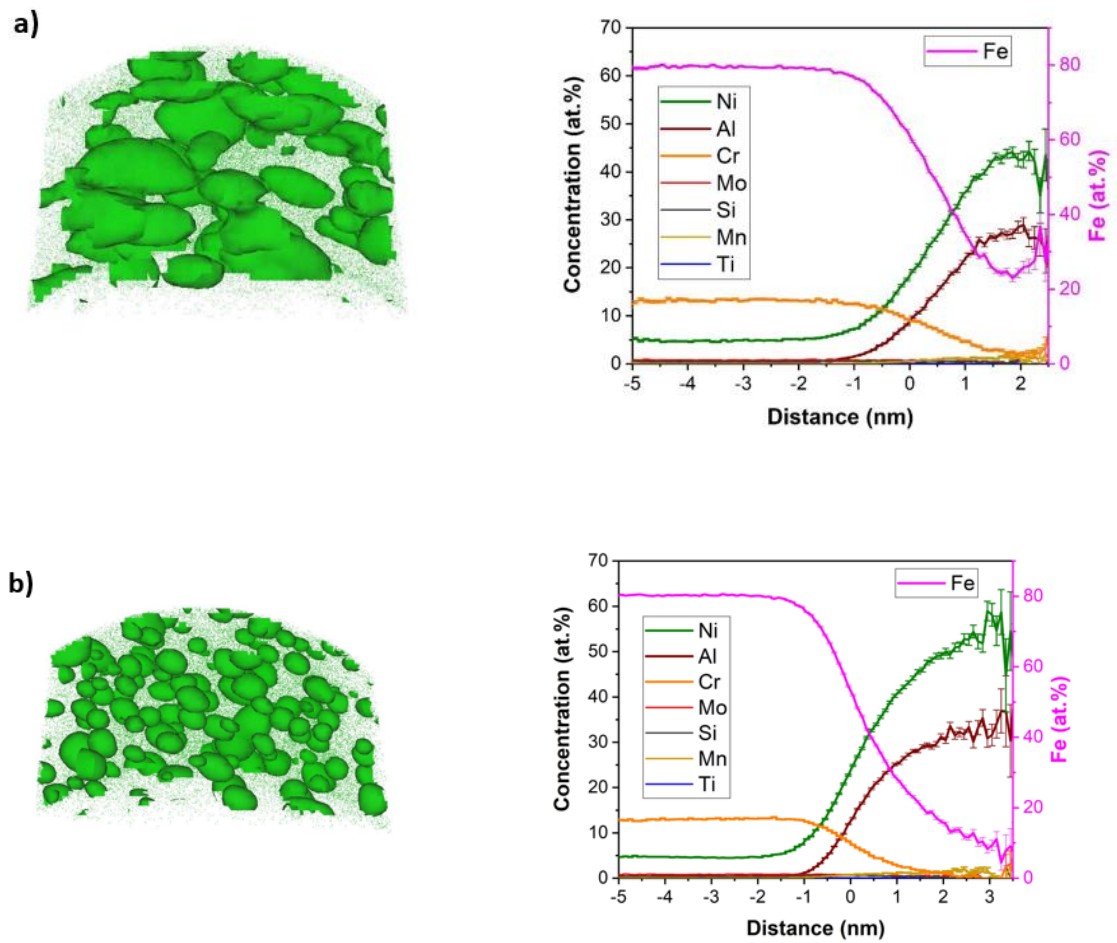


Figure 4.18. The atom probe tomography results showing: a) atomic map of Ni with 17.5 at.% Ni iso-concentration surface (elongated plates), and b) atomic map with 21 at.% Ni iso-concentration surface (spherical).

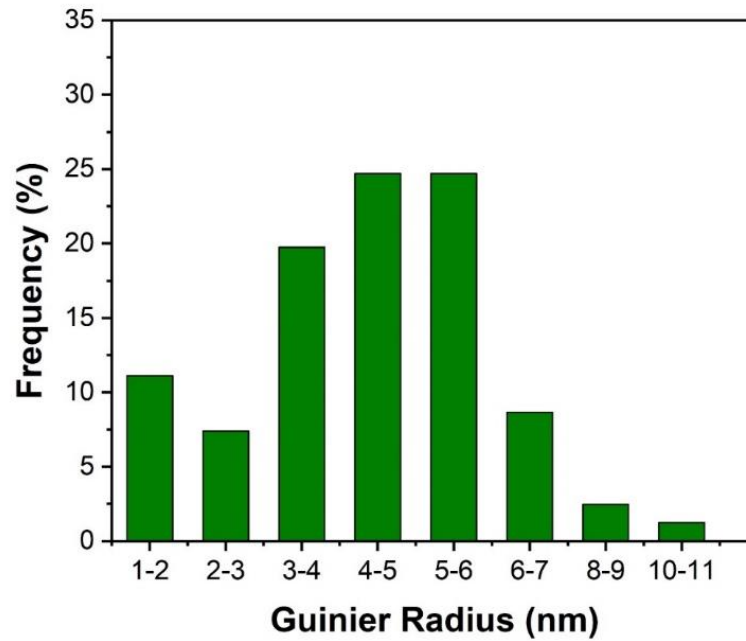


Figure 4.19. The frequency distribution plot associated with the measured radius of precipitates from the atom probe tomography (APT) analysis in Fig. 4.18.

4.9 Conclusion

The elemental concentration profile and precipitation behaviour of M789-N709 hybrid system, with emphasis on the interface region, were assessed using various characterization techniques to further understand its microstructural evolution during post-processing heat treatment. Based on the combined experimental and simulation results, the following conclusions can be drawn:

1. The EBSD analysis on M789, interface and N709 regions of the hybrid alloy system revealed that the as-printed material contains significant dislocation density, as depicted by the measured low angle grain boundaries and Kernel average misorientations. After direct aging, the observed low angle grain boundaries, and

Kernel average misorientations slightly decreased compared to that of the as-printed sample. This result is due to the low aging temperature that limits the complete removal of dislocations and the absence of solution heat treatment. Moreover, the crystallographic texture from EBSD taken from M789, interface and N709 regions suggests that a random arrangement of grains was generated, which promotes the isotropic behaviour of the material.

2. An interface with a thickness of approximately 300 μm was generated after printing M789 alloy on wrought N709. The interface is characterized by the variation of Ti from 0 wt.% in the N709 region to about 1 wt.% in the M789 region. This variation results in different sizes and amounts of $\text{ETA-Ni}_3(\text{Ti,Al})$ strengthening precipitates, with a maximum fraction of precipitate of about 5% and a radius of 5 nm (near the M789 region).
3. Thermodynamic and kinetic simulations were performed based on the measured chemistry of the EPMA; the results showed that $\text{ETA-Ni}_3(\text{Ti,Al})$ precipitates with sizes of approximately 1 nm started to form in the region 150 μm away from the N709 region. Moreover, the precipitate significantly increases with Ti additions of up to 0.6 wt.%. Beyond this threshold, the increase in the amount of precipitation is gradual.
4. The thermodynamic and kinetic simulation performed in the interface of the M789-N709 interface is validated by EDS, EPMA and APT results. The measured precipitate size is consistent with the simulation results. Two morphologies were detected: elongated and spherical. These precipitates contribute to the robustness of the interface.

4.10 References

- [1] S. Parupelli and S. Desai, “A Comprehensive Review of Additive Manufacturing (3D Printing): Processes, Applications and Future Potential,” *Am. J. Appl. Sci.*, vol. 16, no. 8, Sep. 2019, Accessed: Jun. 08, 2021. [Online]. Available: <https://par.nsf.gov/biblio/10124832-comprehensive-review-additive-manufacturing-printing-processes-applications-future-potential>
- [2] F. Klocke, K. Arntz, M. Teli, K. Winands, M. Wegener, and S. Oliari, “State-of-the-art Laser Additive Manufacturing for Hot-work Tool Steels,” *Procedia CIRP*, vol. 63, pp. 58–63, 2017, doi: 10.1016/j.procir.2017.03.073.
- [3] T. DebRoy et al., “Additive manufacturing of metallic components – Process, structure and properties,” *Prog. Mater. Sci.*, vol. 92, pp. 112–224, Mar. 2018, doi: 10.1016/j.pmatsci.2017.10.001.
- [4] Y. Tian, R. Palad, and C. Aranas, “Microstructural evolution and mechanical properties of a newly designed steel fabricated by laser powder bed fusion,” *Addit. Manuf.*, vol. 36, p. 101495, Dec. 2020, doi: 10.1016/j.addma.2020.101495.
- [5] H. Azizi et al., “Metallurgical and mechanical assessment of hybrid additively manufactured maraging tool steels via selective laser melting,” *Addit. Manuf.*, vol. 27, pp. 389–397, May 2019, doi: 10.1016/j.addma.2019.03.025.
- [6] T. Allam, K. G. Pradeep, P. Köhnen, A. Marshal, J. H. Schleifenbaum, and C. Haase, “Tailoring the nanostructure of laser powder bed fusion additively manufactured maraging steel,” *Addit. Manuf.*, vol. 36, p. 101561, Dec. 2020, doi: 10.1016/j.addma.2020.101561.
- [7] Y. Tian, K. Nyamuchiwa, K. Chadha, Y. He, and C. Aranas, “Laser powder bed fusion of M789 maraging steel on Cr–Mo N709 steel: Microstructure, texture, and

- mechanical properties,” *Mater. Sci. Eng. A*, vol. 839, p. 142827, Apr. 2022, doi: 10.1016/j.msea.2022.142827.
- [8] K. Chadha, Y. Tian, P. Bocher, J. G. Spray, and C. Aranas, “Microstructure Evolution, Mechanical Properties and Deformation Behavior of an Additively Manufactured Maraging Steel,” *Materials*, vol. 13, no. 10, p. 2380, May 2020, doi: 10.3390/ma13102380.
- [9] K. Chadha, Y. Tian, J. Spray, and C. Aranas, “Effect of Annealing Heat Treatment on the Microstructural Evolution and Mechanical Properties of Hot Isostatic Pressed 316L Stainless Steel Fabricated by Laser Powder Bed Fusion,” *Metals*, vol. 10, no. 6, p. 753, Jun. 2020, doi: 10.3390/met10060753.
- [10] J. Samei et al., “A hybrid additively manufactured martensitic-maraging stainless steel with superior strength and corrosion resistance for plastic injection moulding dies,” *Addit. Manuf.*, vol. 45, p. 102068, Sep. 2021, doi: 10.1016/j.addma.2021.102068.
- [11] B. Blakey-Milner, “Metal additive manufacturing in aerospace: A review,” p. 33, 2021.
- [12] T. G. Gutowski, M. S. Branham, J. B. Dahmus, A. J. Jones, A. Thiriez, and D. P. Sekulic, “Thermodynamic Analysis of Resources Used in Manufacturing Processes,” *Environ. Sci. Technol.*, vol. 43, no. 5, pp. 1584–1590, Mar. 2009, doi: 10.1021/es8016655.
- [13] J. P. Oliveira, A. D. LaLonde, and J. Ma, “Processing parameters in laser powder bed fusion metal additive manufacturing,” *Mater. Des.*, vol. 193, p. 108762, Aug. 2020, doi: 10.1016/j.matdes.2020.108762.

- [14] K. S. Prakash, T. Nancharaih, and V. V. S. Rao, “Additive Manufacturing Techniques in Manufacturing -An Overview,” *Mater. Today Proc.*, vol. 5, no. 2, pp. 3873–3882, 2018, doi: 10.1016/j.matpr.2017.11.642.
- [15] V. V. Popov and A. Fleisher, “Hybrid additive manufacturing of steels and alloys,” *Manuf. Rev.*, vol. 7, p. 6, 2020, doi: 10.1051/mfreview/2020005.
- [16] C. Li, Z. Y. Liu, X. Y. Fang, and Y. B. Guo, “Residual Stress in Metal Additive Manufacturing,” *Procedia CIRP*, vol. 71, pp. 348–353, 2018, doi: 10.1016/j.procir.2018.05.039.
- [17] S. Jayanath and A. Achuthan, “A computationally efficient hybrid model for simulating the additive manufacturing process of metals,” *Int. J. Mech. Sci.*, vol. 160, pp. 255–269, Sep. 2019, doi: 10.1016/j.ijmecsci.2019.06.007.
- [18] A. Yadollahi and N. Shamsaei, “Additive manufacturing of fatigue resistant materials: Challenges and opportunities,” *Int. J. Fatigue*, vol. 98, pp. 14–31, May 2017, doi: 10.1016/j.ijfatigue.2017.01.001.
- [19] A. S. Johnson, S. Shuai, N. Shamsaei, S. M. Thompson, and L. Bian, “Fatigue Behavior and Failure Mechanisms of Direct Laser Deposited Inconel 718,” p. 13.
- [20] S. Mohd Yusuf, S. Cutler, and N. Gao, “Review: The Impact of Metal Additive Manufacturing on the Aerospace Industry,” *Metals*, vol. 9, no. 12, p. 1286, Nov. 2019, doi: 10.3390/met9121286.
- [21] A. Seidel et al., “Added value by hybrid additive manufacturing and advanced manufacturing approaches,” *J. Laser Appl.*, vol. 30, no. 3, p. 032301, Aug. 2018, doi: 10.2351/1.5040632.

- [22] K. Wei, X. Zeng, F. Li, M. Liu, and J. Deng, “Microstructure and Mechanical Property of Ti-5Al-2.5Sn/Ti-6Al-4V Dissimilar Titanium Alloys Integrally Fabricated by Selective Laser Melting,” *JOM*, vol. 72, no. 3, pp. 1031–1038, Mar. 2020, doi: 10.1007/s11837-019-03988-6.
- [23] K. Chen et al., “Selective laser melting 316L/CuSn10 multi-materials: Processing optimization, interfacial characterization and mechanical property,” *J. Mater. Process. Technol.*, vol. 283, p. 116701, Sep. 2020, doi: 10.1016/j.jmatprotec.2020.116701.
- [24] Z. H. Liu, D. Q. Zhang, S. L. Sing, C. K. Chua, and L. E. Loh, “Interfacial characterization of SLM parts in multi-material processing: Metallurgical diffusion between 316L stainless steel and C18400 copper alloy,” *Mater. Charact.*, vol. 94, pp. 116–125, Aug. 2014, doi: 10.1016/j.matchar.2014.05.001.
- [25] S. L. Sing, L. P. Lam, D. Q. Zhang, Z. H. Liu, and C. K. Chua, “Interfacial characterization of SLM parts in multi-material processing: Intermetallic phase formation between AlSi10Mg and C18400 copper alloy,” *Mater. Charact.*, vol. 107, pp. 220–227, Sep. 2015, doi: 10.1016/j.matchar.2015.07.007.
- [26] S. Shakerin, M. Sanjari, B. S. Amirkhiz, and M. Mohammadi, “Interface engineering of additively manufactured maraging steel-H13 bimetallic structures,” *Mater. Charact.*, vol. 170, p. 110728, Dec. 2020, doi: 10.1016/j.matchar.2020.110728.
- [27] C. V. S. Murthy, A. G. Krishna, and G. M. Reddy, “Dissimilar Welding of Maraging Steel (250) and 13-8 Mo Stainless Steel by GTCAW, LBW and EBW Processes,” *Trans. Indian Inst. Met.*, vol. 72, no. 9, pp. 2433–2441, Sep. 2019, doi: 10.1007/s12666-019-01695-z.

- [28] R. Palad, Y. Tian, K. Chadha, S. Rodrigues, and C. Aranas, “Microstructural features of novel corrosion-resistant maraging steel manufactured by laser powder bed fusion,” *Mater. Lett.*, vol. 275, p. 128026, Sep. 2020, doi: 10.1016/j.matlet.2020.128026.
- [29] E. V. Pereloma, A. Shekhter, M. K. Miller, and S. P. Ringer, “Ageing behaviour of an Fe–20Ni–1.8Mn–1.6Ti–0.59Al (wt%) maraging alloy: clustering, precipitation and hardening,” *Acta Mater.*, vol. 52, no. 19, pp. 5589–5602, Nov. 2004, doi: 10.1016/j.actamat.2004.08.018.
- [30] H. Leitner, M. Schober, and R. Schnitzer, “Splitting phenomenon in the precipitation evolution in an Fe–Ni–Al–Ti–Cr stainless steel,” *Acta Mater.*, vol. 58, no. 4, pp. 1261–1269, Feb. 2010, doi: 10.1016/j.actamat.2009.10.030.
- [31] Y. Li et al., “A new 1.9GPa maraging stainless steel strengthened by multiple precipitating species,” *Mater. Des.*, vol. 82, pp. 56–63, Oct. 2015, doi: 10.1016/j.matdes.2015.05.042.
- [32] A. Basak and S. Das, “Epitaxy and Microstructure Evolution in Metal Additive Manufacturing,” *Annu. Rev. Mater. Res.*, vol. 46, no. 1, pp. 125–149, Jul. 2016, doi: 10.1146/annurev-matsci-070115-031728.
- [33] T. W. Nelson, “Nature and Evolution of the Fusion Boundary in Ferritic-Austenitic Dissimilar Weld Metals, Part 1 Nucleation and Growth,” p. 9.
- [34] S. Shakerin, A. Hadadzadeh, B. S. Amirkhiz, S. Shamsdini, J. Li, and M. Mohammadi, “Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique,” *Addit. Manuf.*, vol. 29, p. 100797, Oct. 2019, doi: 10.1016/j.addma.2019.100797.

- [35] S. Das, “Physical Aspects of Process Control in Selective Laser Sintering of Metals,” *Adv. Eng. Mater.*, vol. 5, no. 10, pp. 701–711, Oct. 2003, doi: 10.1002/adem.200310099.
- [36] C. Böhm, M. Werz, and S. Weihe, “Dilution Ratio and the Resulting Composition Profile in Dissimilar Laser Powder Bed Fusion of AlSi10Mg and Al99.8,” *Metals*, vol. 10, no. 9, p. 1222, Sep. 2020, doi: 10.3390/met10091222.
- [37] M. Niu, G. Zhou, W. Wang, M. B. Shahzad, Y. Shan, and K. Yang, “Precipitate evolution and strengthening behaviour during aging process in a 2.5 GPa grade maraging steel,” *Acta Mater.*, vol. 179, pp. 296–307, Oct. 2019, doi: 10.1016/j.actamat.2019.08.042.
- [38] V. K. Vasudevan, S. J. Kim, and C. M. Wayman, “Precipitation reactions and strengthening behaviour in 18 Wt Pct nickel maraging steels,” *Metall. Trans. A*, vol. 21, no. 10, pp. 2655–2668, Oct. 1990, doi: 10.1007/BF02646061.
- [39] Y. Xiao, Y. Yang, S. Wu, J. Chen, D. Wang, and C. Song, “Microstructure and Mechanical Properties of AlSi10Mg Alloy Manufactured by Laser Powder Bed Fusion Under Nitrogen and Argon Atmosphere,” *Acta Metall. Sin. Engl. Lett.*, vol. 35, no. 3, pp. 486–500, Mar. 2022, doi: 10.1007/s40195-021-01354-7.
- [40] Y. Song, H. Jiang, L. Zhang, S. Li, J. Zhao, and J. He, “A Model Describing Solidification Microstructure Evolution in an Inoculated Aluminum Alloys,” *Acta Metall. Sin. Engl. Lett.*, vol. 34, no. 6, pp. 861–871, Jun. 2021, doi: 10.1007/s40195-020-01154-5.

- [41] T. Bhardwaj and M. Shukla, “Effect of laser scanning strategies on texture, physical and mechanical properties of laser sintered maraging steel,” *Mater. Sci. Eng. A*, vol. 734, pp. 102–109, Sep. 2018, doi: 10.1016/j.msea.2018.07.089.
- [42] S.-S. Rui et al., “Correlations between two EBSD-based metrics Kernel Average Misorientation and Image Quality on indicating dislocations of near-failure low alloy steels induced by tensile and cyclic deformations,” *Mater. Today Commun.*, vol. 27, p. 102445, Jun. 2021, doi: 10.1016/j.mtcomm.2021.102445.
- [43] W. Sha, “Thermodynamic calculations for precipitation in maraging steels,” *Mater. Sci. Technol.*, vol. 16, no. 11–12, pp. 1434–1436, Nov. 2000, doi: 10.1179/026708300101507415.
- [44] K. Hagihara, T. Nakano, and Y. Umakoshi, “Plastic deformation behaviour in Ni₃Ti single crystals with D0₂₄ structure,” *Acta Mater.*, vol. 51, no. 9, pp. 2623–2637, May 2003, doi: 10.1016/S1359-6454(03)00060-0.
- [45] M. Schmidt and K. Rohrbach, *ASM_Metals_HandBook_Volume_4_-_Heat_Treating.pdf*, vol. 4, 24 vols. in *Heat Treating*, vol. 4. ASM International, 1994.
- [46] E. I. Galindo-Nava, W. M. Rainforth, and P. E. J. Rivera-Díaz-del-Castillo, “Predicting microstructure and strength of maraging steels: Elemental optimization,” *Acta Mater.*, vol. 117, pp. 270–285, Sep. 2016, doi: 10.1016/j.actamat.2016.07.020.
- [47] V. G. Gavriluk, V. M. Nadutov, S. P. Oshkaderov, J. Pietikäinen, and K. Ullakko, “Phase and magnetic transformations in the Fe-Ni-C alloy at low temperatures,” *Phys. Met. Metallogr.*, vol. 70, pp. 120–124, 1990.

- [48] X. Zhou et al., “Effect of Pre-deformation on Nanoscale Precipitation and Hardness of a Maraging Stainless Steel,” *Acta Metall. Sin. Engl. Lett.*, vol. 35, no. 12, pp. 2089–2100, Dec. 2022, doi: 10.1007/s40195-022-01440-4.
- [49] P. Kürsteiner, M. B. Wilms, A. Weisheit, P. Barriobero-Vila, E. A. Jäggle, and D. Raabe, “Massive nanoprecipitation in an Fe-19Ni- x Al maraging steel triggered by the intrinsic heat treatment during laser metal deposition,” *Acta Mater.*, vol. 129, pp. 52–60, May 2017, doi: 10.1016/j.actamat.2017.02.069.
- [50] J. Tian et al., “A New Maraging Stainless Steel with Excellent Strength–Toughness–Corrosion Synergy,” *Materials*, vol. 10, no. 11, p. 1293, Nov. 2017, doi: 10.3390/ma10111293.
- [51] T. LeBrun, T. Nakamoto, K. Horikawa, and H. Kobayashi, “Effect of retained austenite on subsequent thermal processing and resultant mechanical properties of selective laser melted 17–4 PH stainless steel,” *Mater. Des.*, vol. 81, pp. 44–53, Sep. 2015, doi: 10.1016/j.matdes.2015.05.026.
- [52] D. H. Ping, M. Ohnuma, Y. Hirakawa, Y. Kadoya, and K. Hono, “Microstructural evolution in 13Cr–8Ni–2.5Mo–2Al martensitic precipitation-hardened stainless steel,” *Mater. Sci. Eng. A*, vol. 394, no. 1–2, pp. 285–295, Mar. 2005, doi: 10.1016/j.msea.2004.12.002.
- [53] S. D. Erlach et al., “Comparison of NiAl precipitation in a medium carbon secondary hardening steel and C-free PH13-8 maraging steel,” *Mater. Sci. Eng. A*, vol. 429, no. 1–2, pp. 96–106, Aug. 2006, doi: 10.1016/j.msea.2006.05.071.
- [54] Y. Tian, R. Palad, L. Jiang, T. Dorin, K. Chadha, and C. Aranas, “The effect of heat treatments on mechanical properties of M789 steel fabricated by laser powder bed

fusion,” J. Alloys Compd., vol. 885, p. 161033, Dec. 2021, doi:
10.1016/j.jallcom.2021.161033.

5. Analysis of dynamic recrystallization through austenite grain reconstruction of additively manufactured martensitic M789 steel

In this chapter, the hot deformation behaviour of M789 is extensively explored. Experimental data is utilized to develop physical materials models that accurately capture the hot deformation behaviour of M789. Through parent grain reconstruction, complemented with shape factor analysis, the dynamic recrystallization phenomenon in M789 is assessed. Additionally, the active mechanism for deformation is defined in this work.

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*Kudakwashe Nyamuchiwa served as the lead investigator of this work and was primarily responsible for writing this manuscript and performing the experimental work. Co-authors contributed to defining the problem, analysis of results and finalizing the manuscript.

5.1 Abstract

This work examines the hot compression behaviour of additively manufactured M789 steel via laser powder bed fusion (LPBF) at temperatures ranging from 850 °C to 1050 °C and strain rates between 0.01 s⁻¹ and 1 s⁻¹. Combined corrections for friction and deformation heating were applied to the flow curves to acquire accurate stress-strain data, followed by phenomenological and physical-based constitutive modelling. In particular, the application of Sellars and Tegart hyperbolic sine model, the Kocks-Mecking model and the Johnson-Mehl-Avrami-Kolmogorov equation facilitated the development of physical material models that accurately depict the behaviour of LPBF-fabricated M789 steel, highlighting the inadequacy of traditional models, such as the modified Johnson-Cook model, in capturing the complexities and metallurgical phenomena associated with high-temperature deformation. The activation energy for the deformation of M789 steel was determined to be lower than that of conventional M250 steel, indicating an accelerated recrystallization process at elevated temperatures. The study also introduces a novel quantitative analysis of dynamically recrystallized grains via combined austenite parent grain reconstruction (based on the Kurdjumov-Sachs orientation relationship combined with the Markov clustering technique) and shape factor measurements, confirming the occurrence of dynamic recrystallization (DRX). The findings suggest that DRX more readily occurs at higher strain rates and higher deformation temperatures, whereas at lower temperatures, a lower strain rate is preferred to generate a higher fraction of DRX grains along with finer grains. Additionally, the work indicates that specific variants from the Kurdjumov-Sachs orientation relationship are favoured during the solidification process in LPBF, which seem to disappear when the alloy is subjected to its austenitization temperature.

5.2 Introduction

Maraging steels are a specialized class of high-strength alloys, characterized by their minimal carbon content and extensive alloying [1,2]. These steels are recognized for their exceptional weldability and formability, rendering them ideally suited for applications within the aerospace, die, and tool manufacturing sectors [3]. The martensitic microstructure of these steels is notably enhanced through post-aging, characterized by a dispersion of fine intermetallic compounds, including $\text{Ni}_3(\text{Ti},\text{Mo},\text{Al})$, Fe_2Mo , and Fe_7Mo_6 [4]. Traditional production methods for maraging steels primarily involve casting, facilitated by the vacuum arc remelting process [5]. However, the growing demands for enhanced production efficiency and the capacity to accommodate complex designs have triggered the adoption of additive manufacturing (AM) techniques as a viable alternative [6]. Notably, AM is recognized for its ability to create distinct microstructures, deviating from those produced through conventional methodologies, attributable to its intricate thermal profiles [7].

Based on the literature, significant work has been conducted to understand the thermomechanical responses of conventional maraging steels. Studies on M300 steel, for example, have indicated that an intermediate temperature range (900-1000 °C) at high strain rates is crucial for dynamic recrystallization [5], contrasting with the slower strain rates and higher temperatures commonly reported [8,9]. M350 steel exhibits similar behaviour under these conditions but with more refined austenite grains, measuring approximately 3 μm [10]. In contrast, M250 steel does not demonstrate dynamic recrystallization below 1000 °C, likely due to Ni and Mo segregation impeding nuclei formation [11]. On the other hand, research on AM-fabricated maraging steels, particularly

regarding their hot deformation behaviour, is still limited. A study on 18Ni300 maraging steel at temperatures between 900 °C and 1200 °C found significant dynamic recrystallization with parent grains averaging around 9 µm, and an activation energy of 379 kJ/mol, comparable to that of conventional counterparts (391 kJ/mol) [12]. Other research on the thermomechanical behaviour of AM alloys has been conducted on Ti-6Al-4V [13] and Al-Si-Cu alloys [14]. However, these works lack an in-depth analysis of mechanical and microstructural behaviour during high-temperature deformation. With the increasing use of AM technology, especially in high-temperature environments, it is important to study how maraging steels, which are commonly used in AM, behave under these conditions. While there is extensive literature on the hot deformation behaviour of conventional steels, studies on AM steels, particularly maraging steels such as M789, are limited. M789 steel, strategically designed for the laser powder bed fusion (LPBF) process, exhibits mechanical and corrosion resistance properties comparable to conventional M250 and PH17-4 steels at room temperature [15,16]. The tensile strength of M789 steel, which is about 1800 MPa, along with 9% ductility after aging, makes it suitable for plastic injection moulding applications. However, the high-temperature properties of M789 steel remain unexplored, which is the focus of this study.

Although high-temperature testing is of great interest, the impact of adiabatic heating and friction during hot compression is crucial [17,18]. These factors are particularly significant in identifying critical points related to softening phenomena like recrystallization and recovery, as well as in establishing parameters for constitutive modelling [19]. During deformation, isothermal conditions are often assumed, but higher Zener-Hollomon (Z) parameters can lead to significant temperature deviations due to adiabatic heating [20].

Correcting temperature is essential for accurate flow stress measurements and for identifying critical values for dynamic softening mechanisms. Similarly, friction during testing influences the stress responses in materials [17]. Various theoretical approaches have been explored to correct friction, such as using Siebel-type compression specimens and the upper bound theorem [17,21,22]. However, despite the availability of such literature, the combined correction for adiabatic heating and frictional stress during hot compressions is rarely performed. Moreover, there are no reports of temperature correction based on the actual measured sample temperature during deformation to account for other external factors.

During hot deformation, predicting flow stress and dynamic recrystallization (DRX) kinetics under different deformation conditions is essential for the application and extended application of steels [5]. This involves using models to describe the respective flow stress and to understand the combined effect of strain hardening and associated softening mechanisms during plastic deformation [23]. These predictive models can be classified into phenomenological and physical-based models [24]. In the former, flow stress is predicted based on experimental observations, which are related to an empirical relationship. A major drawback of these models is the lack of a physical background to justify the fitted experimental results [24]. For example, in the Johnson-Cook model [25], the effects of strain rate, temperature softening, and strain hardening are considered independent. In the Fields-Backofen model [26], flow stress is related to the strain hardening exponent and the strain rate sensitivity. Its reliability is limited when softening mechanisms are experienced during deformation. Some modifications to this model have been implemented by introducing an exponential function that accounts for the softening

ratio [27]. Regardless, models such as the Johnson-Cook, Voce-Kocks [28,29], and Fields-Backofen model can be implemented over limited strain rates and temperatures with reasonable accuracy.

Phenomenological material constitutive models do not accurately capture the effects of dislocation evolution and microstructural changes, especially in the high strain rate and temperature regime [30]. A model first proposed by Bergström [31], later changed to the modified Kocks-Mecking model [32,33], has been used to model recovery behaviour based on the rate change of dislocation density with respect to strain, represented as the finite difference between work hardening and recovery [20]. Similarly, the Zerilli-Armstrong (ZA) model [34] is based on changes in dislocation mechanics, particularly relating the progression of dislocation density over obstacles to the applied strain. It incorporates the combined effects of thermal softening, work hardening, and strain hardening [34,35]. However, it operates under a constant work hardening premise for bcc-type metals, where dislocations overcome Peierls-Nabarro barriers [36]. This approach is problematic since the strain hardening behaviours of such metals are often dependent on strain rate and temperature [37]. Moreover, the constitutive parameters of the ZA model tend to lose physical meaning at elevated temperatures and strain rates [38]. To capture the kinetics of softening due to DRX, the Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation [39] has been applied successfully to predict flow stress evolution under the influence of softening mechanisms [40,41]. It measures the exponential time rate dependence of nuclei formation, relating the natural logarithm of the recrystallized fraction to the time exponent [42,43]. Despite being applied to predict the evolution of recrystallized volume fraction and grain size, it fails to account for randomly sized grains. During deformation processes, the

dislocation volume increases, resulting in a non-uniform distribution of stored deformation energy in randomly sized grains (duplex grain effect) [44].

The current study addresses gaps in existing knowledge regarding the high-temperature properties of AM materials, as previously described. It specifically focuses on M789 steel fabricated via LPBF. To enhance the accuracy of high-temperature stress-strain data, a correction method that combines the effects of temperature and friction has been proposed and implemented. Utilizing the corrected data, a range of material models, including both phenomenological and physical approaches, were established. The mechanical behaviour of the steel was correlated with microstructural changes observed before and after deformation by reconstructing austenite grains and conducting grain shape factor analysis. This approach enables a quantitative evaluation of recrystallized grains. The research presents a reliable method for acquiring high-temperature stress-strain data and conducting microstructural analysis, considering various external factors that may influence the accuracy of the data.

5.3 Methodology

5.3.1 Material preparation

Powders of M789 steel (with sizes in the range of 15-45 μm) were processed using an EOS M290 LPBF machine. An energy density of 75-85 J/mm^3 , laser power of 260-280 W, scanning speed of 1050-1200 m/s, and layer thickness of 40 μm were utilized. The details of the printing parameters are available in an earlier work by the present authors [15,45]. Printed M789 steel coupons were machined into cylindrical compression samples with an aspect ratio of 1.5 (i.e., diameters and lengths of 6mm and 9mm, respectively), with the compression axis aligned along the building direction (BD).

5.3.2 Mechanical Testing and Material characterization

The hot deformation tests were performed using a Gleeble 563 system and covered a temperature range from 850 °C to 1050 °C at 50 °C intervals, with strain rates from 0.01 s⁻¹ to 1 s⁻¹, up to a true strain of 0.7. The heating of the samples was accomplished through Joule heating, while deformation was performed using a servo-hydraulic system. Graphite sheets with nickel paste were placed between the compression anvils and the specimen to minimize friction, and a K-type thermocouple was spot-welded at the centre of each specimen to precisely record the temperature. Each sample was heated at a rate of 3.3 °C/s to 1150 °C and soaked for 30 seconds in an argon atmosphere. The sample temperature was then adjusted to the testing temperature at a similar rate and soaked for 30 seconds to eliminate any thermal gradient before deformation, as shown in **Fig. 5.1a**.

After the hot deformation of M789 steel samples, stress-strain data from the Gleeble system were then used as input to develop material constitutive models using MATLAB and Python codes. In addition to hot compression tests, dilatometric experiments were also carried out using the Gleeble 563 equipped with a crosswise transducer, employing a heating and cooling cycle at a rate of 0.4 - 0.48 °C/s from room temperature to 1050 °C.

After the mechanical data analysis, the deformed M789 samples were air-cooled and cut along the deformation axis (**Fig. 5.1b**). The samples were then polished using standard metallographic preparation techniques, starting with rough polishing using SiC papers up to 1 µm diamond paste. Additionally, final polishing was performed using 0.02 µm colloidal silica to prepare the samples for electron backscatter diffraction (EBSD) analysis, which was carried out using a Thermo Scientific Scios 2 DualBeam system. The EBSD analysis focused on a 200×200 µm² observation area, applying an accelerating voltage of

20kV and a step size of 0.04 μm , achieving at least 92% indexing. Low-angle grain boundaries were identified by misorientation angles between 2° and 15° ; on the other hand, misorientation angles above 15° were labelled as high-angle grain boundaries. All analyses were conducted on region III, as illustrated in **Fig. 5.1b**. For the EBSD data, the reconstruction of prior austenite grains was performed via MTEX, employing an iterative process that leveraged the Kurdjumov-Sachs (K-S) orientation relationship (OR) [46], combined with a Markov clustering technique [47]. Due to various intrinsic and extrinsic factors, the experimental austenite-martensite OR tends to vary from the rational K-S orientation relationship. The actual OR was initially aligned with the K-S relationship. This OR value was then iteratively refined to converge at the best fit between the mean grain boundary misorientations and the assigned OR [47].

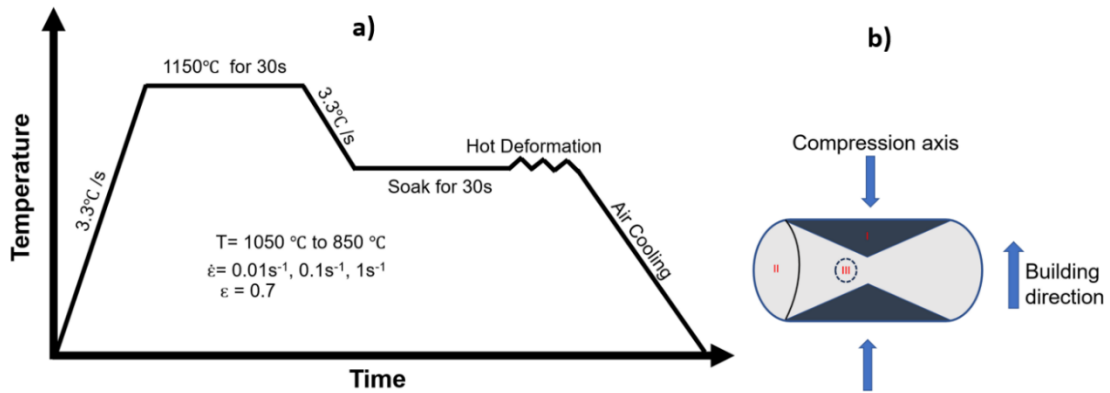


Figure 5.1. a) Thermomechanical schedule employed for M789 steel samples, and b) an image showing the region where the microstructural analysis was performed for all the samples (i.e., region III).

5.3.3 Effect of deformation heating and friction on the flow curves

5.3.3.1 Friction correction

The flow stress data presented in the article includes the raw experimental data and data corrected for friction effects. This correction is crucial for the precise estimation of the corresponding flow stress of M789 steel. Friction significantly influences the flow stress properties, as evidenced by specimen barreling after deformation [17]. During compression, friction occurs at the surface of the specimen in contact with the anvils, leading to the specimen sticking. Under such conditions, Poisson contraction cannot be achieved, and the material of the specimen accumulates towards the centre. Consequently, this results in inhomogeneous deformation, which overestimates the recorded flow stress values [18]. The influence of friction is expected to increase with rising strains and strain rates, where friction is expected to transition from a dynamic to a static state [48]. Hence, the values of flow stress were corrected using a friction factor, which accounts for the barreling of the specimen, as described in the following equation [49]:

$$\delta\sigma_f = \left[\frac{\sigma_k}{1 + (2/3\sqrt{3})m (r_0/h_0)\exp(1.5\varepsilon)} \right]_{\dot{\varepsilon}} \quad (5.1)$$

where σ_k is the measured flow stress, r_0/h_0 is the ratio of the initial radius and height, respectively, ε is the corresponding true strain, and m is the friction coefficient given by [49]:

$$m = \left[\frac{(R_a/h)b}{(4/\sqrt{3}) - (2b/3\sqrt{3})} \right]_{\dot{\varepsilon}} \quad (5.2)$$

where R_a/h is the ratio of the specimen barreling radius and height after deformation, and b is the barreling factor derived from the following equation [49]:

$$b = 4[(R_a - R_t)/R_a]/[h/(h_0 - h)] \quad (5.3)$$

The presence of friction was a notable factor, elevating the flow stress by 2 to 11%, predominantly observed under high-temperature conditions. At elevated temperatures, the lubricant tends to dry up, causing the specimen to adhere to the anvils and leading to increased friction.

5.3.3.2 Temperature correction

At the highest strain rate of 1 s^{-1} , data from the Gleeble indicated a deviation from isothermal conditions due to the increased internal energy changes in the specimens [20]. Given the brevity of the test, the equipment used cannot adjust the temperature quickly enough to maintain the testing temperature (i.e., within less than a second). Assuming an adiabatic condition in the system, the energy derived from the internal changes is reflected in the deviation from the test temperature. Observations from the experiments indicate that at low strain rates, specifically 0.01 and 0.1 s^{-1} , the system has sufficient time to adjust the temperature to maintain the test conditions. However, this is not the case at high strain rates, as demonstrated, for example, in **Fig. 5.2** for the test at $1050 \text{ }^\circ\text{C}$ and a 1 s^{-1} condition. During deformation, the temperature is observed to vary, with a maximum increase of about $20 \text{ }^\circ\text{C}$ observed at a strain of 0.35 . This adiabatic heating leads to significant flow softening. As a result, the obtained flow stress data may give a false impression of softening that does not correspond to preset isothermal conditions. Isothermal flow stress data is crucial for accurate modelling of flow stress behaviour; hence, a correction was

implemented. Temperature correction is based on the degree of conversion of plastic work into heat and is often thermodynamically derived from inherent material properties, such as mean flow stress, specific heat capacity, and density [20]. However, the initial theoretical approach to determine temperature deviation does not account for in-situ specimen temperature or the effects of external temperature control from the PID system [20]. A key aspect of this work was to correct the temperature based on in-situ temperature measurements recorded directly from the specimen itself; this approach accounts for any heat loss from the system due to various external factors. Initially, these measurements were compared to theoretical predictions, and it was observed that they underestimated the actual temperature deviations during deformation. The actual temperature of the specimen was experimentally determined based on the thermal-strain profiles and was used to accurately calculate the flow stresses using the following formulation [20]:

$$\delta\sigma_i = \left[\frac{\partial\sigma}{\partial(1/T)} \right]_{\dot{\epsilon}, \delta\epsilon_i} \left[\frac{1}{T_{iso} + \delta T} - \frac{1}{T_{iso}} \right] \quad (5.4)$$

where $\beta = \left[\frac{\partial\sigma}{\partial(1/T)} \right]_{\dot{\epsilon}, \delta\epsilon_i}$, δT is the change in temperature and T_{iso} isothermal test temperature (all corrections are considered for constant strain and strain rate).

5.4 Results

A dilatometric curve for the complete heating and cooling cycle of M789 steel is shown in **Fig. 5.3**, with inflections indicating the phase transformation temperatures. Precipitation was observed at the first inflection point (581 °C), indicated by the contraction of the alloy. Energy is utilized to form precipitates within the solute matrix until they dissolve at 671

°C [50]. Precipitation is preceded by the formation of a two-phase region between temperatures of 671 °C and 707 °C, consisting of martensite and austenite phases. The occurrence of a two-phase region is derived from the initial precipitation, where the partitioning of Ni into Ni-rich and Ni-lean regions occurs [51]. The Ni-rich regions tend to have a lower austenite start temperature, resulting in the coexistence of martensite and austenite. The A_{e3} and martensite transformation temperatures were identified at 758 °C and 203 °C, respectively. These results agree with thermodynamic calculations carried out using MatCalc (not shown here), showing a two-phase region between 650 °C and 675 °C, and an austenite finish temperature of 753 °C. It is important to note that the sample from dilatometry, labeled here as sample SLC, is analyzed later.

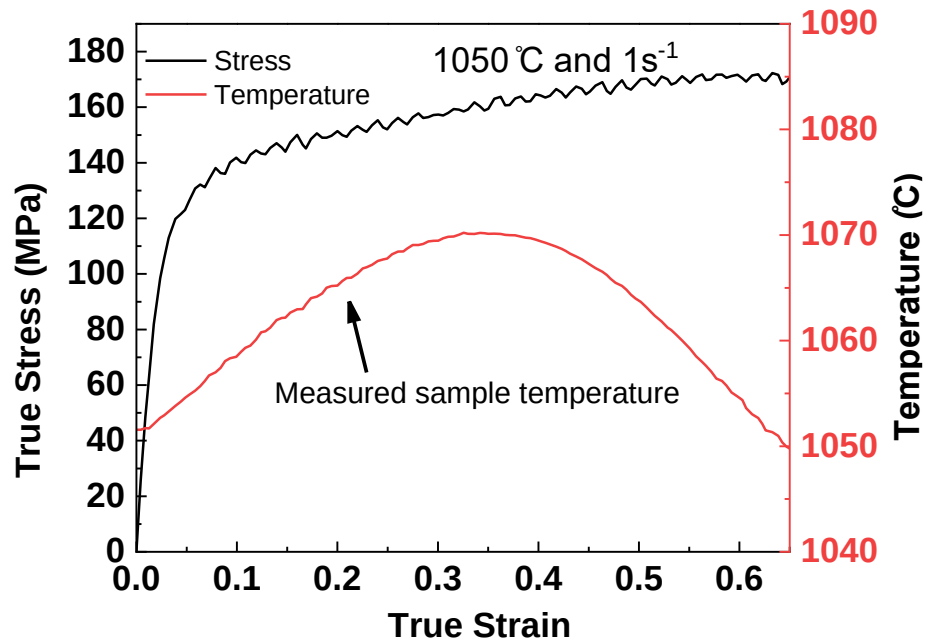


Figure 5.2. Temperature variation as a result of applied stress from hot compression tests at 1050 °C and 1 s⁻¹.

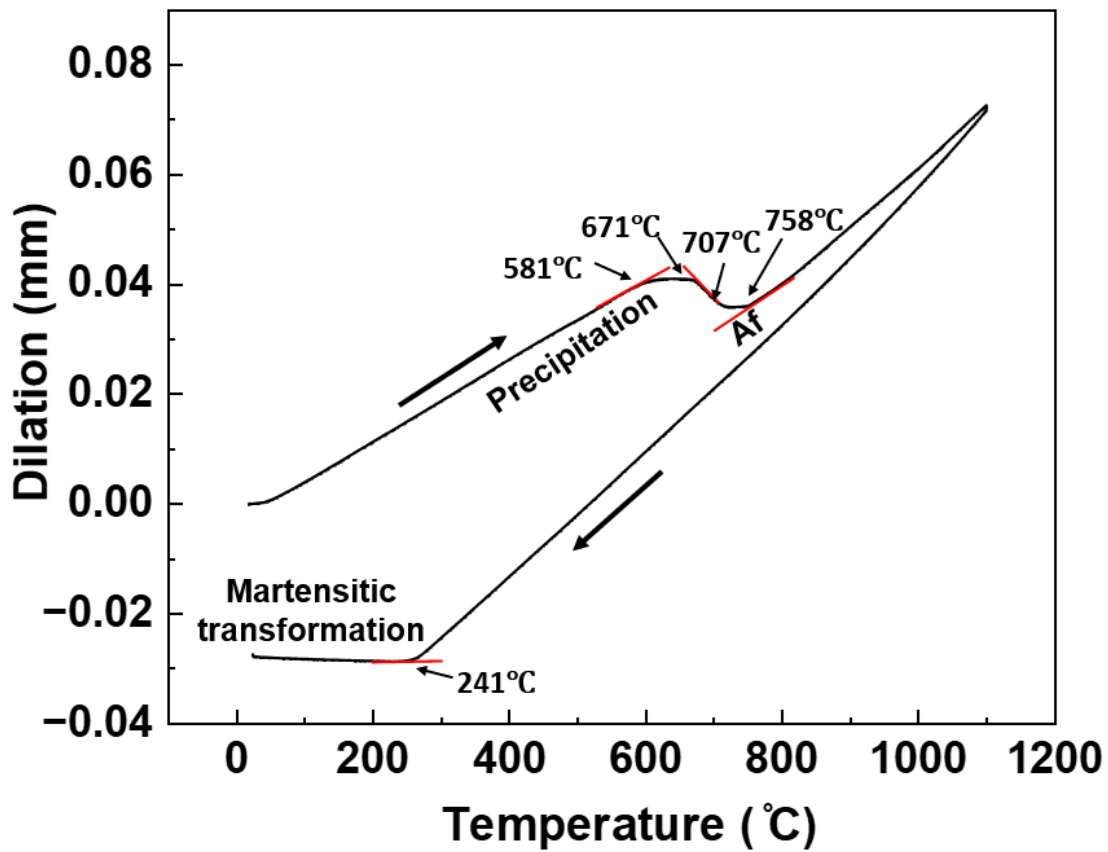


Figure 5.3. Dilatometry curve for LPBF-fabricated M789 steel conducted at a heating and cooling rate of 0.40 and 0.48 °C·s⁻¹, respectively.

The experimental true stress-strain curves for M789 steel under various deformation conditions are illustrated in **Fig. 5.4a-c**, showcasing different strain rates. These hot deformation tests were conducted within a fully austenitic region, as confirmed by the dilatometry results. Generally, the curves reveal an initial rapid increase in flow stress, attributed to work hardening resulting from the accumulation of dislocations at the onset of deformation. This initial increase is followed by a gradual reduction in the rate of flow stress increase with increasing strain, indicating material softening. The flow curves presented in **Fig. 5.4** illustrate a consistent, monotonic decrease in flow stress with rising

temperature across all deformation conditions, highlighting the impact of friction on flow stress.

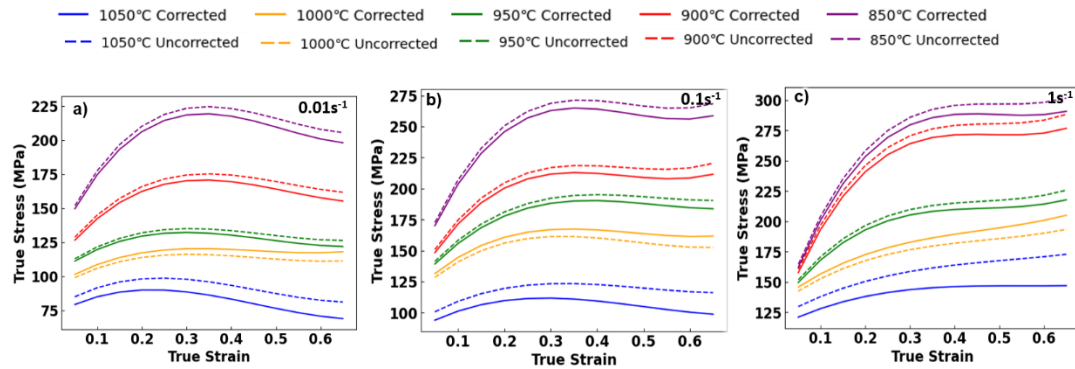


Figure 5.4. Friction corrected (solid line) vs. uncorrected (dashed line) values of flow stress at strain rates of: a) 0.01 s^{-1} , b) 0.1 s^{-1} and c) 1 s^{-1} .

In **Fig. 5.5**, the independent effects of friction and temperature on the experimental flow stress are presented. From **Fig. 5.5a**, it is evident that the friction-corrected flow stress data exhibits increasing work hardening behaviour. However, upon applying temperature correction in **Fig. 5.5b**, notable softening behaviour is observed after a strain of 0.4. During the early phases of deformation, work hardening predominates as the primary metallurgical phenomenon. However, competing softening mechanisms such as dynamic recovery and recrystallization induce the annihilation and reordering of dislocations, countering and offsetting the initial high rate of work hardening [5,52]. This effect is seen in the flow stress patterns that gradually recede after reaching a peak value. As the interaction between work hardening and softening mechanisms intensifies, an equilibrium is reached, resulting in steady-state flow stress conditions. Notably, a drop in temperature is observed in **Fig. 5.5b** after a true strain of 0.35, when corrected to the isothermal testing temperature. This correction lowers the measured flow stress, corresponding to the temperature differences

from the reference temperature. The flow curve thus depicts some softening and a decrease in the work hardening rate. At lower testing temperatures of 850 °C and 900 °C, the effects are minimal below a strain of 0.35, suggesting that at lower strains, the impact of internal changes in the material can be considered negligible under these conditions. It is important to note that at very high strains, a drop in flow stress after peak strength is not always evident due to the dominance of work hardening [52].

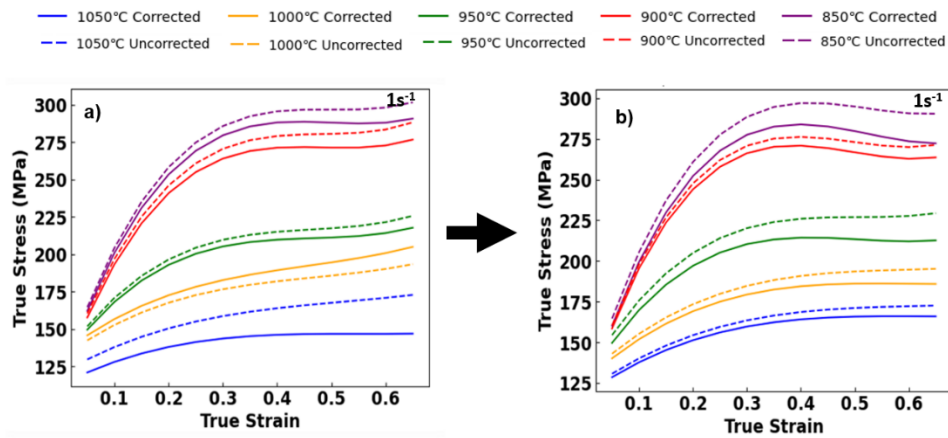


Figure 5.5. Flow stress curves after application of: a) friction correction only, and b) friction and temperature corrections. The uncorrected data is shown as dashed line, while the corrected data is presented in solid lines.

5.5 Physical Parameters Related to Hot Working

The corrected flow stress values have been incorporated into constitutive models to assess their suitability for modelling the hot deformation behaviour of LPBF-fabricated M789 steel. Details of these models are presented in the following sub-sections.

5.5.1 Modified Johnson-Cook

The modified Johnson-Cook model was utilized to overcome the limitations of the original Johnson-Cook model, which does not consider the combined effects of temperature, strain,

and strain rate. This model specifically accounts for the coupled effects of temperature and strain rate, highlighting the enhanced effects of strain rates at elevated temperatures [53]. Material constants were determined at a reference temperature and strain rate of 850 °C and 0.01 s⁻¹, respectively. The modified Johnson-Cook flow stress equation is expressed as follows [53]:

$$\sigma = (A_1 + B_1\varepsilon + B_2\varepsilon^2)(1 + C_1\ln\dot{\varepsilon}^*)e^{(\lambda_1 + \lambda_2 \ln\dot{\varepsilon}^*)(T - T_{ref})} \quad (5.5)$$

where A_1 , B_1 , B_2 , C_1 , λ_1 and λ_2 are material constants, ε is the true strain, and $\dot{\varepsilon}^*$ is the dimensionless strain rate derived from the ratio of the strain rate ($\dot{\varepsilon}$) and the reference strain rate ($\dot{\varepsilon}_{ref}$). The values T and T_{ref} are the actual temperature and the reference temperature, respectively.

At the reference temperature and strain rate, the material constants A_1 , B_1 , and B_2 describe the flow stress curve up to the yield point. These constants are related according to the following equation:

$$\sigma = (A_1 + B_1\varepsilon + B_2\varepsilon^2) \quad (5.6)$$

The coefficients A_1 , B_1 , and B_2 were determined by conducting a second-order polynomial fit at reference strain rate and temperature (850 °C and 0.01 s⁻¹) with their values evaluated from the corrected stress-strain data as 68.76 MPa, 79.69 MPa, and -123.89 MPa, respectively. The constants C_1 , λ_1 and λ_2 were determined from the slopes of the following linear plots; $(\sigma / A_1 + B_1\varepsilon + B_2\varepsilon^2)$ vs $\ln\dot{\varepsilon}^*$ for C_1 , and $\ln(\sigma / (A_1 + B_1\varepsilon + B_2\varepsilon^2)(1 + C_1\ln\dot{\varepsilon}^*))$ vs T . Their values were 0.816363, 0.004673 and -0.0003, respectively. **Fig. 5.6** shows a comparison between the results predicted by the modified Johnson-Cook model

and the corresponding experimental data across five different temperatures (850-1050 °C) and three distinct strain rates (0.01-1 s⁻¹).

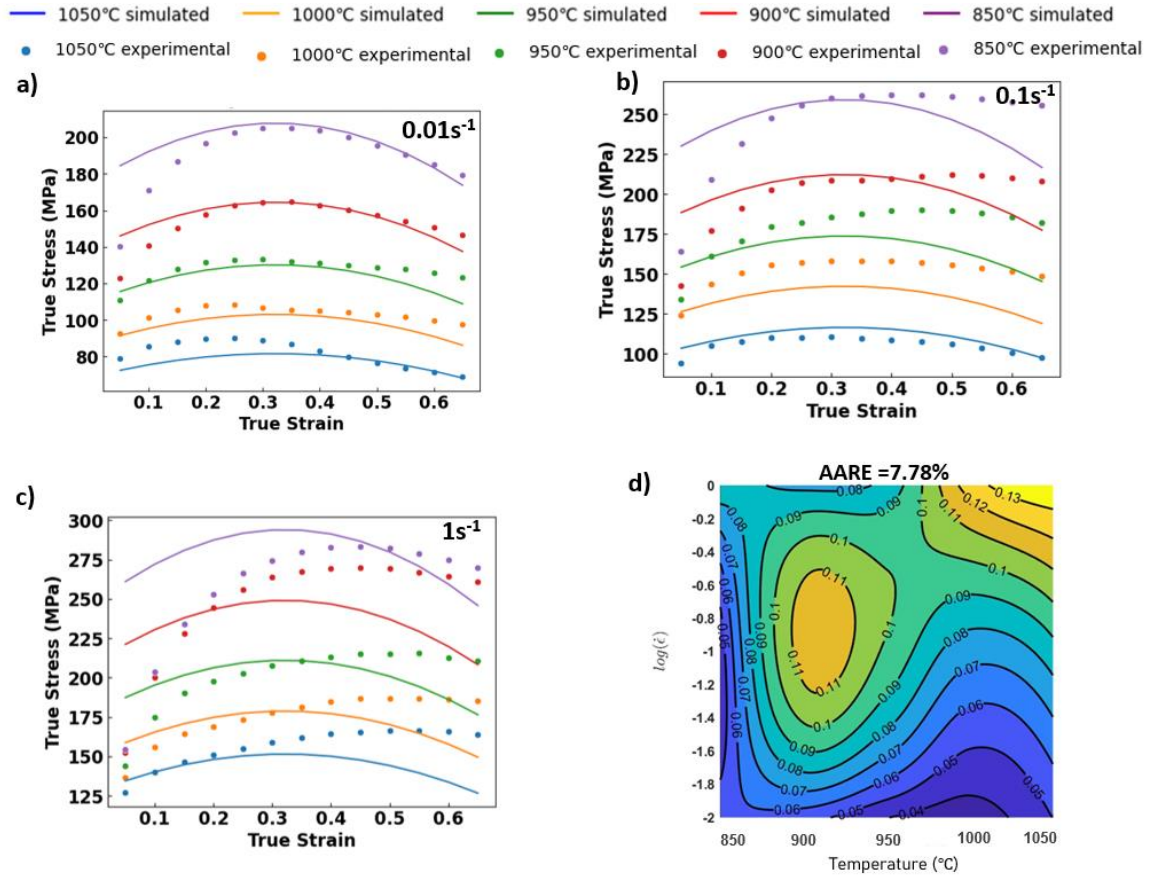


Figure 5.6. Modified Johnson-Cook constitutive model showing the experimental and simulated flow curves at strain rates of: a) 0.01 s⁻¹, b) 0.1 s⁻¹, and c) 1 s⁻¹. d) The contour plot of average absolute relative error is also presented.

At a strain rate of 0.01 s⁻¹, good fitting is particularly observable for true strain values greater than 0.30, as depicted in **Fig. 5.6a**. However, as the strain rate increases to 0.1 s⁻¹ and 1 s⁻¹, a noticeable decrease in the quality of fit beyond a strain of 0.30 becomes apparent (**Fig. 5.6b-c**). Despite the high R² value of 87.75% at these higher strain rates (**Fig. 5.6c**), the model increasingly shows unreliability, as evidenced by the poor fitting with the

experimental values. **Fig. 5.6d** illustrates the average absolute relative error (AARE) between the predicted and experimental flow stress under the studied experimental conditions. Considering the combined effects of all strain rates and temperatures, the model exhibits significant errors, about 12% at high strain rates and temperatures. Conversely, higher temperatures at slower strain rates demonstrate lower AARE values, approximately 5%. The overall error computed across all strain rates and temperatures is 7.78%. The AARE gradually increases with the strain rate and temperature, indicating that the model best predicts the flow stress within a narrow domain around the reference temperature and strain rate.

5.5.2 Sellars and Tegart hyperbolic sine model

The effects of temperature and strain rate were evaluated using the hyperbolic sine equation derived from steady-state conditions of creep as proposed by Sellars and Tegart [54] which is in the form:

$$\dot{\epsilon} = AF(\sigma)e^{(-Q/RT)} \quad (5.7)$$

$$F(\sigma) = \begin{cases} \sigma^n & \alpha\sigma < 1 \\ e^{(\beta\sigma)} & \alpha\sigma > 1.2 \\ [\sinh(\alpha\sigma)]^n & \text{for all } \sigma \end{cases} \quad (5.8)$$

$$Z = \dot{\epsilon}e^{(Q/RT)} = A[\sinh(\alpha\sigma)]^n \quad (5.9)$$

where Z is the Zenner-Holloman parameter [55] which expresses a temperature compensated strain rate. The predicted flow stress can be described as function of the Z parameter as:

$$\sigma = \frac{1}{\alpha} \ln \left\{ \left(\frac{z}{A} \right)^{\frac{1}{n}} + \left[\left(\frac{z}{A} \right)^{\frac{2}{n}} + 1 \right]^{\frac{1}{2}} \right\} \quad (5.10)$$

where, $\dot{\epsilon}$ is the strain rate, σ is the flow stress, T is the absolute temperature and Q is the hot deformation activation energy. A , α , and β are material constants, and n is the stress exponent. The evaluation of these constants was based on the peak stresses obtained directly from the flow stress curves. Generally, the value of the adjustable scaling factor α is estimated by β/n by plotting $\ln \dot{\epsilon}$ vs $\ln \sigma$ at different temperatures. However, the stress exponent value obtained through this method was considered suboptimal. To address this, the approach developed by Uvira and Jonas was employed [56]. It involves varying the scaling factor over a narrow range, resulting in a variation of n . The best suited value of $\alpha = 0.015 \text{ MPa}^{-1}$ was selected due to its minimum standard deviation (3.64 %) in n over the calculated range. Q was obtained by multiplying the gradient of $\ln(\sinh(\alpha\sigma))$ vs $(1/T)$ by the values of n and R , where R is the real gas constant.

Under the studied conditions, the activation energy at peak stress for M789 steel was calculated to be approximately 415 kJ/mol. This value is lower than that calculated for conventional M250 steel, which is 428.8 kJ/mol, but higher than that of 17-4PH steel, with an activation energy of 373 kJ/mol.

The above analysis resulted in the following constitutive equation:

$$Z = \dot{\epsilon} e^{(414937/RT)} = 2.9 * 10^{13} [\sinh(0.015\sigma)]^{3.42} \quad (5.11)$$

The onset of dynamic softening was determined by observing the work hardening rate (θ) and the rate of change of work hardening ($d\theta/d\epsilon$) using the second derivative method with a 9th order polynomial fitting [57]. An example of this analysis is presented in **Fig. 5.7**,

which shows the respective peak stress (σ_p), steady stress (σ_{ss}), and critical stress (σ_c). Softening mechanisms, such as DRX, are initiated at a specific critical stress, with a corresponding critical strain (ϵ_c) [57,58]. After reaching this critical condition, the stress peaks at σ_p , where the trade-off between softening and work hardening occurs, particularly between the unrecrystallized and recrystallized grains.

Progressively, a steady-state stress (σ_{ss}) independent of strain is achieved, signifying the most energetically favoured balance between the competing mechanisms [57]. Considering the boundary testing conditions, the critical stress (σ_c) for the onset of recrystallization at 1050 °C and 1 s⁻¹ was evaluated to be about 165 MPa at a critical strain of 0.44. This value is the lowest at 1050 °C and 0.01 s⁻¹, with a critical stress and strain of 85 MPa and 0.10, respectively. The peak stress at 1050 °C and 1 s⁻¹ was observed to be almost double that at 1050 °C and 0.01 s⁻¹, with stresses of 168 MPa ($\epsilon=0.52$) and 91 MPa ($\epsilon=0.24$), respectively. Conversely, at the lower temperature of 850 °C and a strain rate of 1 s⁻¹, the highest σ_c was observed at 274 MPa ($\epsilon=0.3$). It is worth noting that σ_c could not be fully distinguished from the peak stress (σ_p) as they had almost the same value. At 850 °C and 0.01 s⁻¹, σ_c and σ_p were evaluated at 190 MPa (0.16) and 206 MPa ($\epsilon=0.36$), respectively.

The characteristic points of flow curves were plotted against the Zener-Holloman parameter in **Fig. 5.8** to determine the dependence of σ_p and ϵ_c on the temperature-compensated strain rate. The σ_c and σ_p show a stronger linear dependence on the Zener-Holloman parameter with a coefficient of determination, R^2 of 0.86 and 0.91, respectively. On the other hand, the ϵ_c and ϵ_p values have an R^2 value of 0.154 and 0.41, respectively.

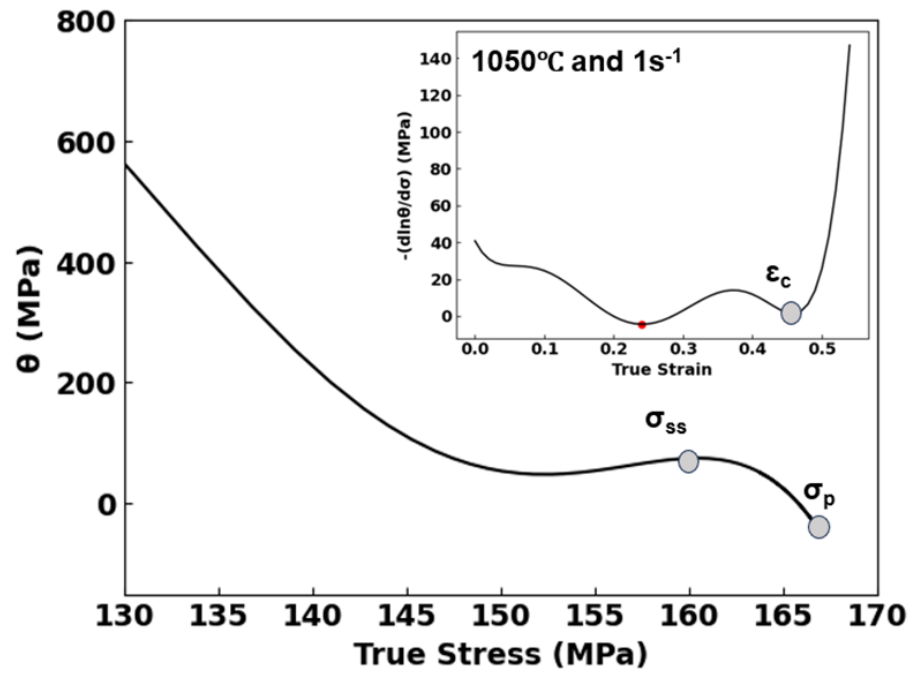


Figure 5.7. Application of the double differentiation method to identify the critical points associated with DRX.

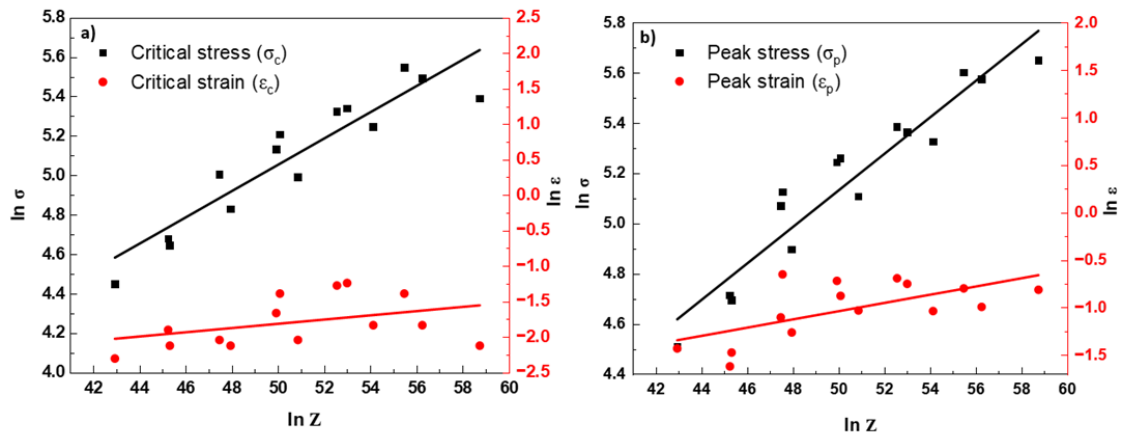


Figure 5.8. Dependence of a) σ_c and ϵ_c , and b) σ_p and ϵ_p on the Zener-Holloman parameter.

5.5.3 Kocks-Mecking (KM) and JMAK kinetic models

The Kocks-Mecking (KM) model [32] was utilized to model the flow stress behaviour of M789 steel, aiming to interpret its recovery behaviour as a result of the competing mechanisms of strain hardening and recovery. The rate of strain hardening is determined by the balance between the storage and rearrangement of dislocations. The rate of change in dislocation density (ρ) with plastic strain ($d\varepsilon$) is represented by the following relationship [20,31,32]:

$$\frac{d\rho}{d\varepsilon} = h - r\rho \quad (5.14)$$

where h is an athermal work hardening rate constant related to dislocation storage, and r is a constant that serves as an indicator of the strain rate and temperature effects [20]. This particular constant quantifies the rate of recovery change at a fixed temperature and strain rate, independent of strain [32]. Based on this relationship, the associated flow stress during work hardening up to the saturation flow stress (σ_{sat}), where $d\rho/d\varepsilon$ approaches zero, is described by the equations [43]:

$$\sigma = (\sigma_{sat}^2 - ((\sigma_{sat}^2 - \sigma_0^2)\exp(-r\varepsilon))^{0.5} \quad (5.15)$$

$$\sigma \frac{d\sigma}{d\varepsilon} = 0.5(\sigma_{sat}^2 - r\sigma^2) \quad (5.16)$$

Equation 5.16 presents a linear relationship of $\sigma \frac{d\sigma}{d\varepsilon}$ with σ^2 . Hence, the values of the recovery parameter r , and σ_{sat} can be obtained from the slope and intercept of the plotted equation before the critical point associated with DRX. Based on these values, the flow stress curves related to the initial work hardening of regions that have not undergone recrystallization can be determined. The recovery curves at the boundary of the testing regime are presented in **Fig. 5.9**. Observations indicate that the work hardening curves of

the unrecrystallized regions show a work hardening rate comparable to the experimental conditions (after the yield point), particularly at 1050 °C and 1 s⁻¹, and 850 °C and 1 s⁻¹, as shown in **Fig. 5.9a-b**, respectively. Despite the prediction of recovery between 1050 °C and 850 °C at 1 s⁻¹, some degree of softening is observed where the flow stress does not continuously increase with strain. From the model prediction (1050°C and 1s⁻¹), the experimental curve shows higher strain hardening compared to the simulated curve with an absolute average relative difference of about 4%. In contrast, deviations from the experimental flow stress curve are observed approximately after the yield point. This observation suggests that under these conditions, recovery is the dominant softening mechanism. At lower strain rates (0.01 s⁻¹) at 850 °C and 1050 °C, some degree of softening predicted by the model is suggestive of recrystallization.

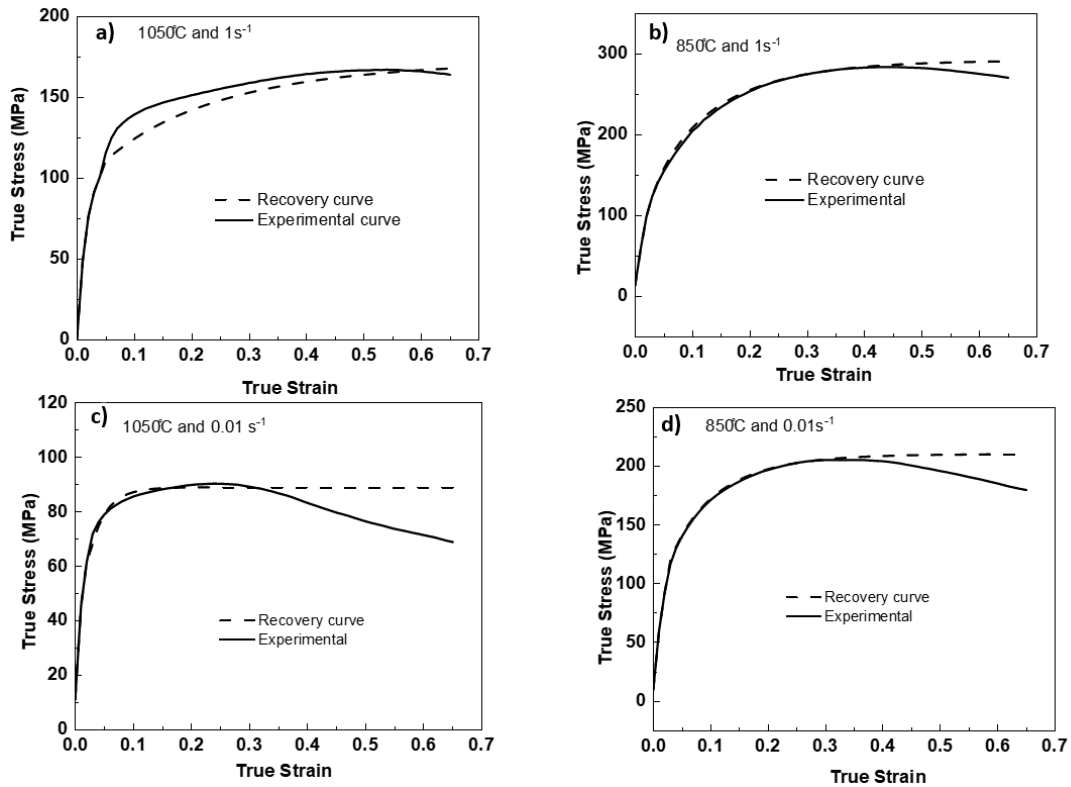


Figure 5.9. Predicted recovery curves at a) 1050°C and 0.01s⁻¹, b) 1050°C and 1s⁻¹, c) 850°C and 0.01s⁻¹, and 850°C and 1s⁻¹ conditions.

Further analysis was conducted using the Johnson-Mehl-Avrami-Kolmogorov (JMAK) plot to understand the recrystallization kinetics of M789 steel. A plot of $\ln(-\ln(1-X))$ and $\ln(\epsilon - \epsilon_c)/\dot{\epsilon}$ is presented in **Fig. 5.10**. The gradient and intercept of these plots provide the rate constant n and k , respectively, which satisfy this relationship. The fractional softening (X) was determined by the formula $(\sigma_p - \sigma)/(\sigma_p - \sigma_{ss})$. A plot of the average n values at varying strain rates is shown in **Fig. 5.11**. The relationship between the average values of n and the corresponding strain rate demonstrates a linear relationship, described by the equation: $n = 0.5173 \ln \dot{\epsilon} + 6.7772$. The value of k was determined from the intercepts and was observed to vary with Z parameter.

Thus, the JMAK equation can be represented as follows [39,43]:

$$X = 1 - e^{(-k(\epsilon - \epsilon_c)/\dot{\epsilon})^n} \quad (5.17)$$

Based on these constants, the simulated curves were compared against the experimental data as shown in **Fig. 5.12**. A notable agreement between the experimental and predicted flow stress using the JMAK equation was observed, with the differences in the AARE between the experimental data and the JMAK model being less than 1.5%. However, this trend diminishes at the 1050 °C and 1 s⁻¹ condition, where an error of about 4% is observed.

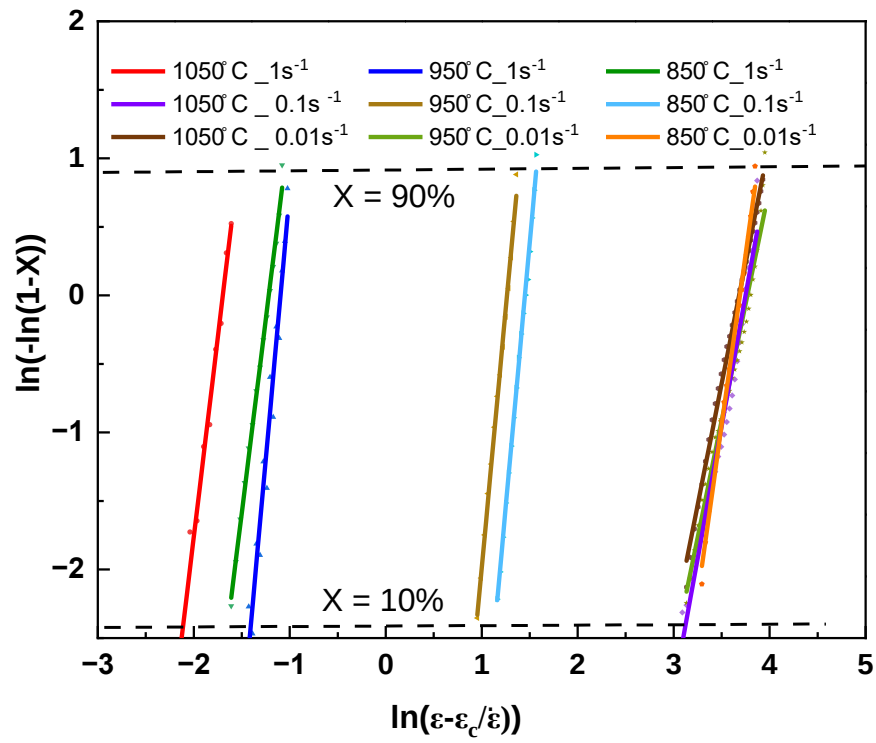


Figure 5.10. The Johnson-Mehl-Avrami-Kolmogorov (JMAK) plots at different testing temperatures and strain rates.

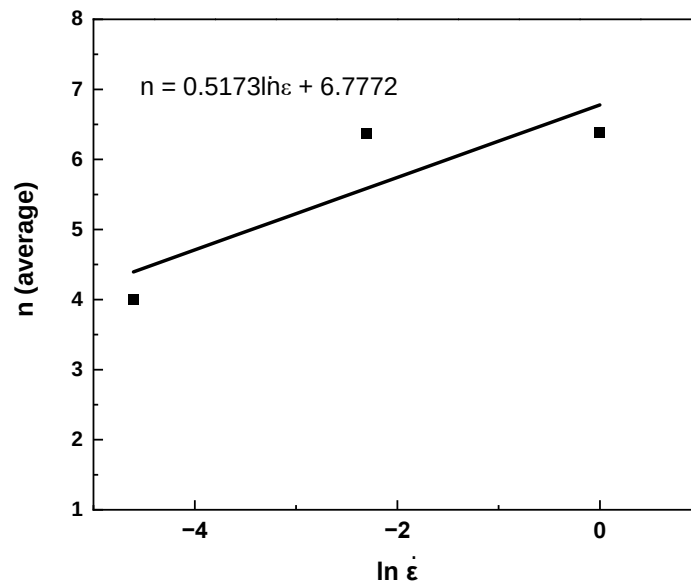


Figure 5.11. Linear relationship between average slope (n) and strain rate.

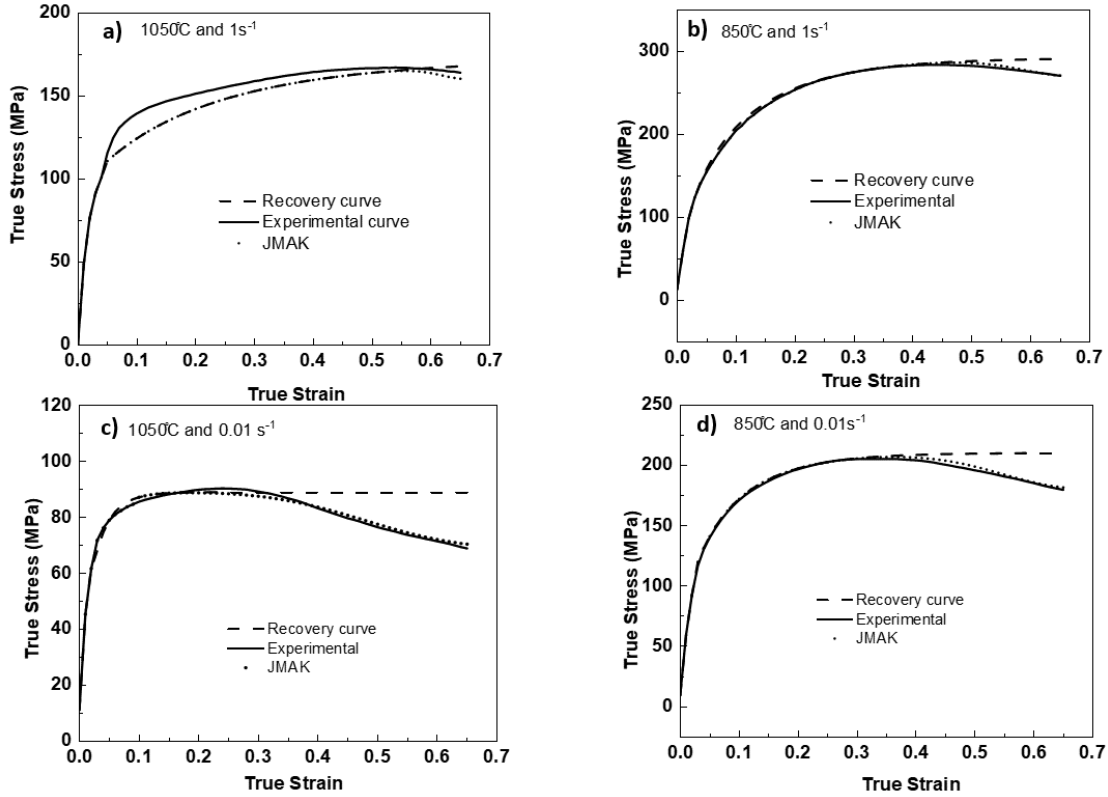


Figure 5.12. Recovery and DRX curves a) 1050 °C and 0.01 s⁻¹, b) 1050 °C and 1 s⁻¹, c) 850 °C and 0.01 s⁻¹, and 850 °C and 1 s⁻¹.

5.5.4 Microstructural analysis

To further analyze the recrystallized grains and support the stress-strain analysis, the microstructure of M789 steel under hot deformed conditions was examined at temperatures of 850 °C, 950 °C, and 1050 °C, and strain rates ranging from 0.01 s⁻¹ to 1 s⁻¹. The band contrast images are presented in **Fig. 5.13**. From **Fig. 5.13a**, several fine grains are evident in samples deformed at 1050 °C and 1 s⁻¹, suggesting a high degree of recrystallization occurred during deformation above the A_{e3} temperature. In contrast, at a slower strain rate of 0.01 s⁻¹ (**Fig. 5.13b**), significant grain growth is observed, with elongated grains oriented at 45 degrees to the compression axis. Intermediate temperatures and strain rates (i.e.,

950 °C and 0.1 s⁻¹, respectively) show less significant growth but significant lath refinement (**Fig. 5.13c**).

For the 850 °C and 1 s⁻¹ deformed samples (**Fig. 5.13d**), the crystallographic laths appear to be larger compared to those at a strain rate of 0.01 s⁻¹ (**Fig. 5.13e**), and this is confirmed by the crystallographic block sizes given in **Fig. 5.14**, where the weighted martensite block widths are 2.54 µm and 1.4 µm, respectively. Significant lath growth within the prior austenite grains can be observed in the printed condition (labelled as AP), with block sizes of about 5 µm observed, possibly due to epitaxial growth and remelting during subsequent layers; this observation contrasts with the expected behaviour during the fast-cooling rates of the printed sample. The effects of strain and temperature are significant in samples deformed at 1050 °C and 1 s⁻¹ condition, where increased lath refinement was observed with a weighted block width of less than 1 µm. The formation of small block widths inside prior austenite grains indicates grain refinement during deformation in the austenite phase.

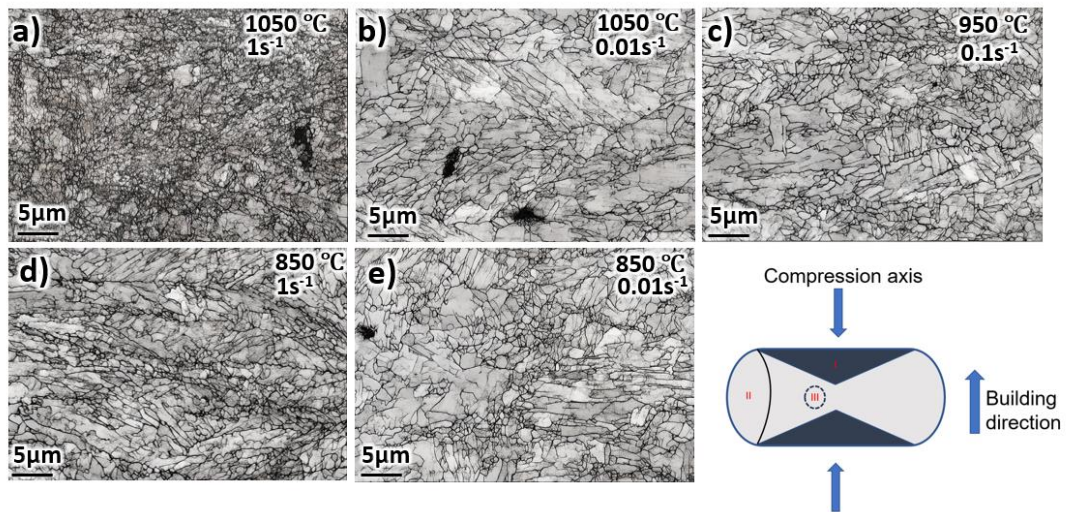


Figure 5.13. Microstructure of deformed M789 steel at temperatures and strain rates of: a) 1050 °C and 1 s⁻¹, b) 1050 °C and 0.01 s⁻¹, c) 950 °C and 0.1 s⁻¹, d) 850 °C and 1 s⁻¹, and e) 850 °C and 0.01 s⁻¹.

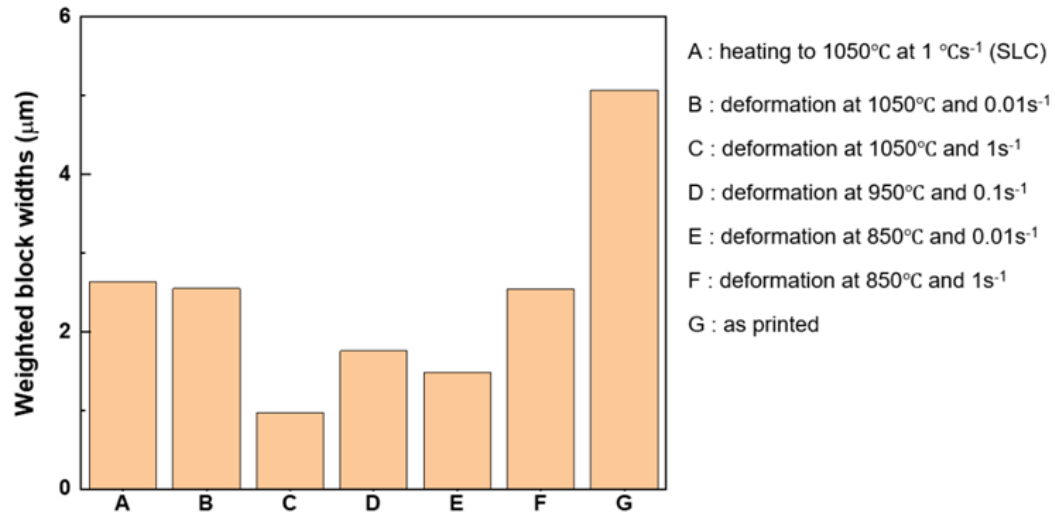


Figure 5.14. Martensite block size comparison chart. (SLC – heating and cooling rate of $1^{\circ}\text{C}\cdot\text{s}^{-1}$).

5.6 Discussion

5.6.1 Materials Modelling

The modified Johnson Cook model from **Fig. 5.6** exhibited high deviations in its flow stress predictions (AARE=7.78%) compared to the JMAK model in **Fig. 5.12** (AARE = 4%). The observed large deviations can be attributed to the constant coefficient of strain hardening, C_1 , which was maintained constant in this study and did not account for the possibility of strain rate softening [59,60]. This presents limitations in the prediction of flow stress since this model does not account for physical changes that occur within the material. In comparison, the use of KM and JMAK physical models accounts for the effect of work hardening and fractional softening based on dislocation motion [32,43]. However, the reliability of both the KM and JMAK models appears constrained when applied to high temperatures and strain rates in this hot working regime, exhibiting masking of strain softening behaviours. Nonetheless, the KM and JMAK models exhibit better predictability

of flow stress compared to the modified Johnson-Cook, as indicated by the poorer prediction of softening, especially towards the critical points. As seen from **Fig. 5.8** the critical stress (σ_c) and peak stress (σ_p) exhibit a strong linear dependence on the Z parameter. Specifically, the characteristic stress tends to increase as the Z value decreases. Within the studied temperature and strain rate regime, the ratios of both critical to peak stress (σ_c/σ_p) and critical to peak strain (ϵ_c/ϵ_p) were evaluated to be approximately 0.9 and 0.5, respectively. The observed value of σ_c/σ_p is in the same range as reported in a previous study [43]. This investigation suggests that for dynamic recrystallization to occur in LPBF-fabricated M789 steel, the normalized critical strain (ϵ_c/ϵ_p) must reach a value of about 0.5. The calculated critical strains for DRX are in the range of 0.1-0.5.

The accurate determination of the scaling factor(α) and stress exponent (n) was crucial for this study. Past studies suggest that α should fall in the range between 0.01-0.02 MPa-1 [23,61], and in this study, it was expanded to 0.005-0.02 MPa-1. The lower bound of 0.005 MPa-1 was used to accommodate the initial estimation of n (β/n). With a more accurate α value the average n value (3.42) was computed by calculating the gradient of $\ln \dot{\epsilon}$ vs $\ln(\sinh(\alpha\sigma))$. The obtained n value can be associated with plastic deformation whose rate is predominantly governed by dislocation sliding, which is common for Class A alloy types. The presence of solute atoms (Ni, Cr, Mo, Ti) impedes the glide and velocity of dislocations, making it the rate determining process [62]. Additionally, the disorder caused by dislocation motion forms new interfacial energies that act as barriers to dislocation motion [63]. Alternatively, due to the high homologous temperature range ($T/T_m=0.65-0.76$), the contribution of dislocation climb, and the diffusion of vacancies cannot be ignored, contributing to glissile dislocations.

The stress exponent (n) obtained from the method proposed by Uvira and Jonas [56] in the current study derives its meaning from the empirical Mukherjee-Bird-Dorn relationship [64]. This relationship describes high-temperature crystalline plasticity in the creep steady state regime, characterized by a constant strain rate. Here, ' n ' is the normalized stress exponent that describes the mechanism involved during deformation [64]. Under the high-temperature conditions studied for M789, dislocation motion is expected to proceed through the creation of vacancies in neighbouring slip planes, facilitating the progression of dislocations and resulting in plastic deformation [65]. An ' n ' value of 1 indicates Newtonian type mechanisms, where the strain rate exhibits a linear relationship with the applied stress [66]. Under such conditions, three main mechanisms are possible: Nabarro-Herring [67,68] and Coble [69] creep mechanisms, which are diffusion-based, and the Harper-Dorn mechanism [70], which is dislocation-based. The distinction between these mechanisms is also associated with the inverse grain size exponent (p) of 2, 3, and 0, respectively. It is evident from the stated p values that the strain rate in the Coble creep mechanism [69] is more sensitive to grain size than in the Nabarro-Herring creep mechanism [67,68]. Conversely, the Harper-Dorn mechanism demonstrates strain rate independence from grain size.

In the Nabarro-Herring type mechanism, diffusion occurs through the lattice or grain interior, whereas, in the Coble mechanism, grain boundary diffusion occurs via the grain boundaries [71,72]. An ' n ' value of 2 is associated with grain boundary sliding phenomena, where the response of grain boundaries to an applied stress involves the relative movement of grains along a common boundary or within a narrow zone near the boundary [73,74]. Depending on the accommodating mechanism, whether diffusion or glide and climb, grain

boundary sliding is referred to as Lifshitz grain boundary sliding [73] and Rachinger sliding [74], respectively. In all cases, boundary sliding accommodation is necessary to prevent void formation at the grain boundary regions [73,74].

For an 'n' value of 3, an alloy-type creep behaviour is expected, where dislocation climb dominates over glide, making the latter the rate-determining step [65,75]. Cottrell et al. [62] suggested that reduced glide could be attributed to a drag force associated with solute atoms segregation caused by dislocation motion, and the speed of dislocation motion is influenced by the rate of diffusion of solute atoms. Moreover, Fisher [63] proposed that the disruption of short-range orders in the solid solution creates new interfacial energy barriers to dislocation motion. At n values ranging from 4 to 5, the rate-determining mechanism is considered to be dislocation climb. The values near 3 are also associated with climb-controlled dislocation motion, especially at elevated temperatures [65,76]. For higher n values, greater than 5, deformation is described as a power law breakdown [77–79], where changes in the strain rate are no longer constant at higher stress and creep rates vary with stress exponentially. Sherby et al. [77] proposed that the occurrence of vacancy diffusion due to vacancy supersaturation at these high stresses determines the rate-controlling mechanism.

5.6.2 Analysis of recrystallized grains through austenite grain reconstruction

Recrystallization typically occurs in the austenite phase [3,12], and to gain insight into the austenitic microstructure before phase transformation, parent grain reconstruction was carried out to observe the microstructural evolution. The dilatometry results presented in **Fig. 5.3** confirmed that all deformations occurred in a fully austenitic phase field. An

optimized K-S relationship, featuring minimal disorientation between the theoretical and experimental orientation relationships in the child-to-child linkage, was adopted [47].

The resulting reconstruction is presented in **Figs. 5.15-5.16**, with the grain weighted diameter and area presented in **Fig. 17**. In line with the martensite lath sizes (**Figs. 5.13, 5.14**), large equiaxed grains are observed in the SLC sample heated to 1050 °C. The child and parent phases for the SLC sample are shown in **Figs. 5.15a** and **5.15b**. Slow heating results in significant growth as seen in **Fig. 5.15b**, and this is reflected in the transformed martensite after reaching the martensite start temperature (241°C). The prior austenite grains measured about 24 μm with the largest weighted area of 204 μm^2 (**Fig. 5.17**). Conversely, a deformation strain rate of 1 s^{-1} at the same heating temperature (1050 °C) led to grain refinement, as illustrated in **Fig. 5.15d** (parent reconstruction from child grains in **Fig. 5.15c**), with the prior austenite grains measuring about 8 μm and a weighted area size of 23 μm^2 (**Fig. 5.17**). The grain refinement in **Fig. 5.15f** (reconstructed from **Fig. 5.15e**) displays coarser grains than those in **Fig. 5.15d**.

Observations from **Fig. 5.15** are compared to the child and parent phases associated with tests at 950 °C and 0.1 s^{-1} (**Figs. 5.16a** and **5.16b**), 850 °C and 1 s^{-1} (**Figs. 5.16c** and **5.16d**), and 850 °C and 0.01 s^{-1} (**Figs. 5.16e** and **5.16f**). Comparing the microstructures from **Figs. 5.15** and **5.16**, the sample deformed at 1050 °C and 1 s^{-1} condition confirms the occurrence of recrystallization due to significant grain refinement compared to other conditions presented in **Figs. 5.16b, 5.16d, and 5.16f**. It is often reported that dynamically recrystallized grains require slow strain rates to allow for nucleation and growth [8]. However, the trend observed in M789 steel appears to contradict this, suggesting that to acquire finer grains, higher strain rates may increase the chance of obtaining finer grains.

At the lower temperature of 850 °C, some degree of grain refinement was observed at the slower strain rate of 0.01 s⁻¹ (**Fig. 5.16f**) compared to the higher strain rate of 1 s⁻¹ (**Fig. 5.16d**). The grain diameter and area were evaluated to be 27 µm and 153 µm², respectively. Significant grain growth is not anticipated at this temperature and strain rate (**Fig. 5.16d**), especially when compared to the condition of a slower strain rate (**Fig. 5.16f**). However, **Fig. 5.16d** shows evidence of pancaking of grains under the high flow stress conditions experienced during deformation. This grain distortion is evident from the calculated average shape factor of 3, the highest among the studied conditions. Under high flow stress conditions, grains tend to adopt the most favourable geometry by spreading perpendicular to the applied load.

Fig. 5.17 illustrates the recrystallized fraction of austenite grains during deformation, quantified by the shape factor parameter obtained from the EBSD data. A grain shape factor threshold of 2 was applied to distinguish between recrystallized and non-recrystallized grains. The presence of grains with a shape factor of less than 2, as observed in the as-printed state (**Fig. 5.17a**), exceeds that of the SLC sample (**Fig. 5.17b**). The occurrence of these grains in the as-printed condition is likely due to the rapid cooling of specific nuclei that do not have time to grow or possibly the entrapment of delta ferrite during rapid cooling, rather than recrystallization [80].

As depicted in **Fig. 5.18**, the grain sizes in the as-printed sample are significantly larger (i.e., by 60%) than those reported for the 1050 °C and 1 s⁻¹ condition. This suggests that the observed grains were most likely formed from rapid nucleation and limited growth during printing. About 46% of the grains in the deformed samples at 1050 °C and 1 s⁻¹ have a shape factor of less than two, with a total average shape factor of 2 (**Fig. 5.17c**). These

grains are predominantly located at the boundaries of larger austenite grains and tend to be engulfed during growth at slower strain rates (**Fig. 5.17d**). For the deformed samples at 850 °C and 1 s⁻¹ (**Fig. 5.17e**), 73% of the grains have a shape factor above 2, whereas, at 850°C and 0.01 s⁻¹, the proportion of grains reduces to about 53% (**Fig. 5.17f**).

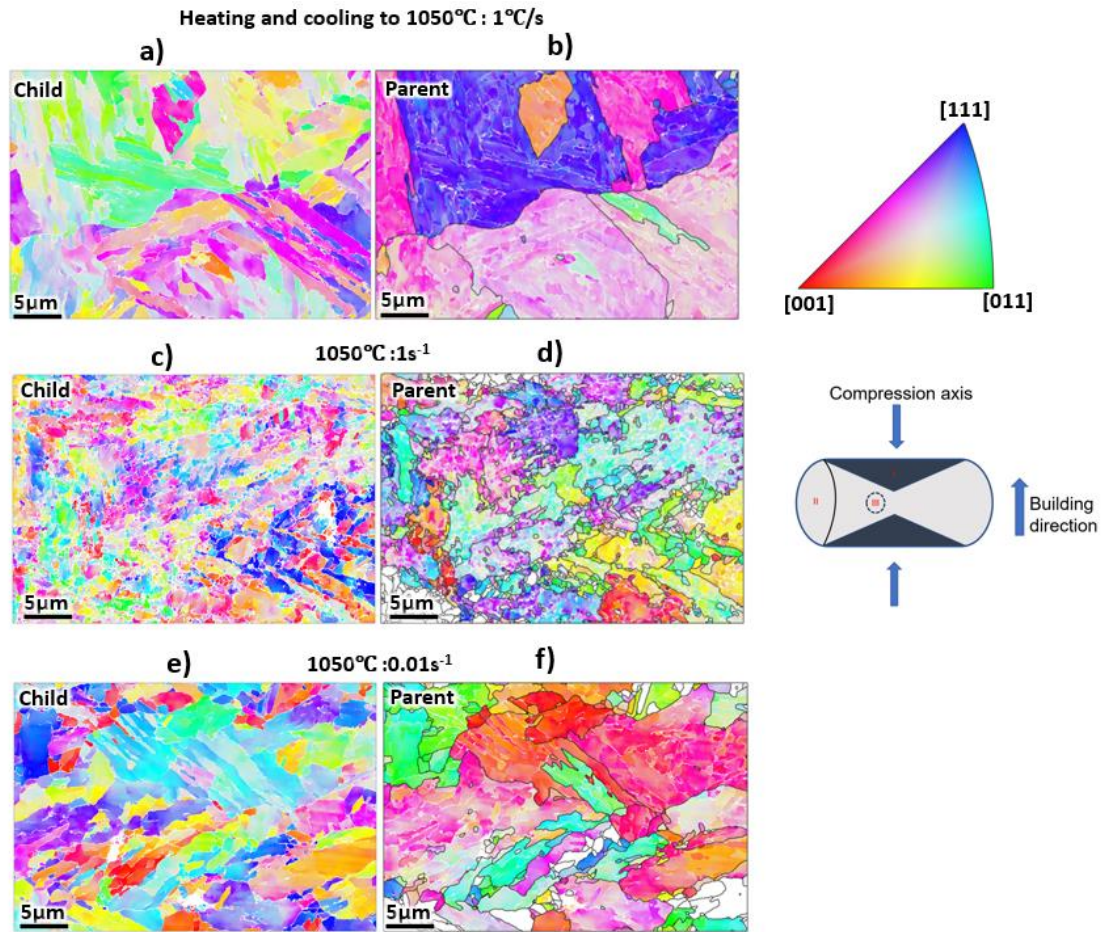


Figure 5.15. Inverse pole figure maps for child and parent phase for: a,b) SLC sample, c,d) deformed sample at 1050 °C and 1 s⁻¹, and e,f) deformed sample at 1050 °C and 0.01 s⁻¹.

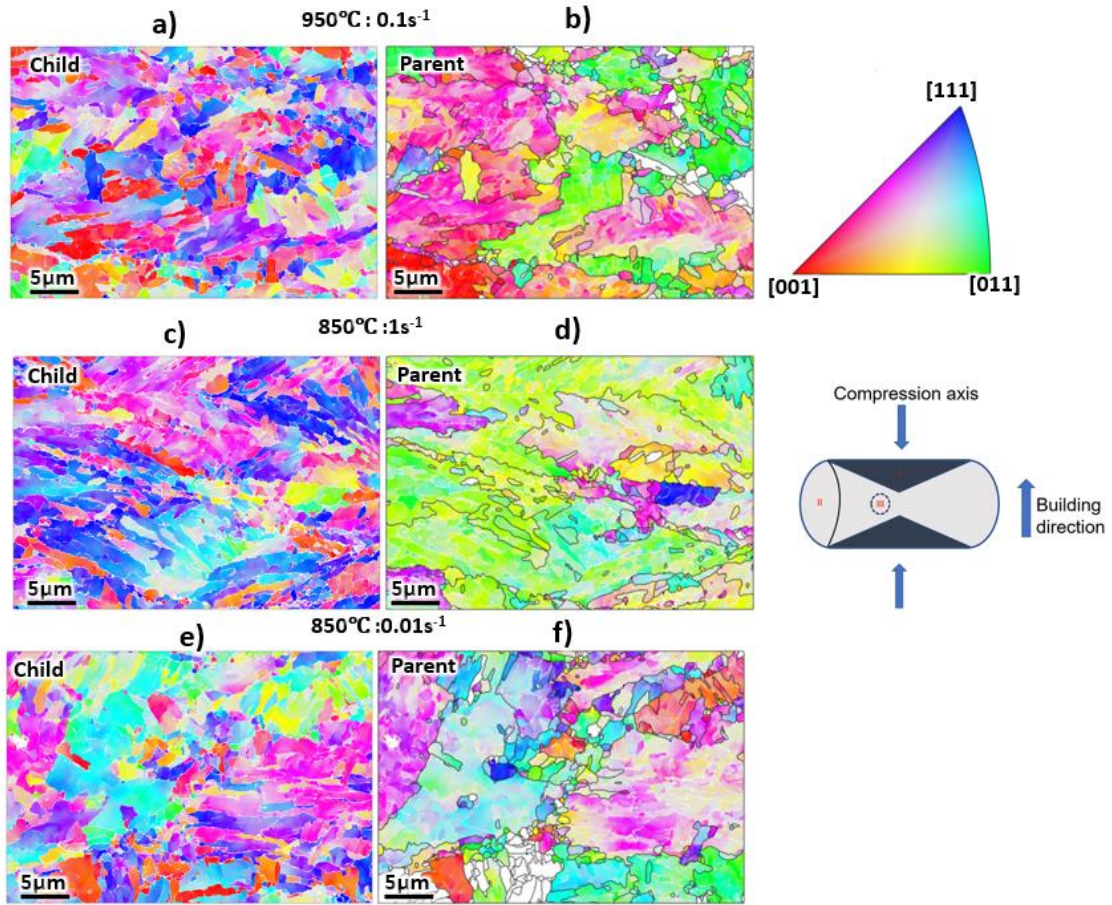


Figure 5.16. Inverse pole figure maps for child and parent phases for M789 steel samples deformed at: a,b) 950 °C and 0.1 s⁻¹, c,d) 850 °C and 1 s⁻¹, and e,f) 850 °C and 0.01 s⁻¹.

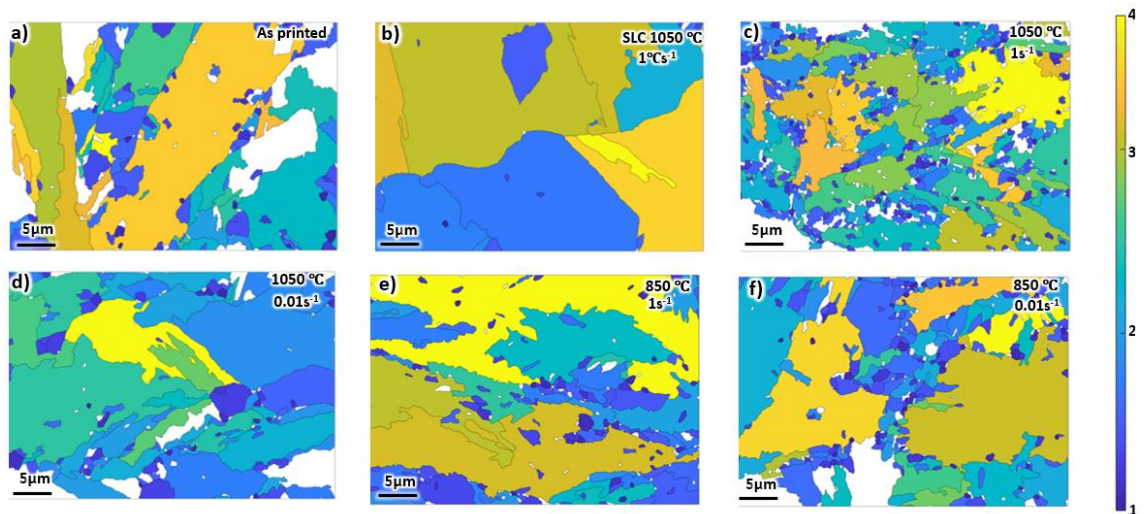


Figure 5.17. Quantification of recrystallized fraction of austenite grains using grain shape factor for the following conditions of M789 steel samples: a) as-printed state, b) after heating/cooling at 1 °C-s⁻¹, c) deformed at 1050 °C and 1 s⁻¹, d) deformed at 1050 °C and 0.01 s⁻¹, e) deformed at 850 °C and 1 s⁻¹, and f) deformed at 850 °C and 0.01 s⁻¹.

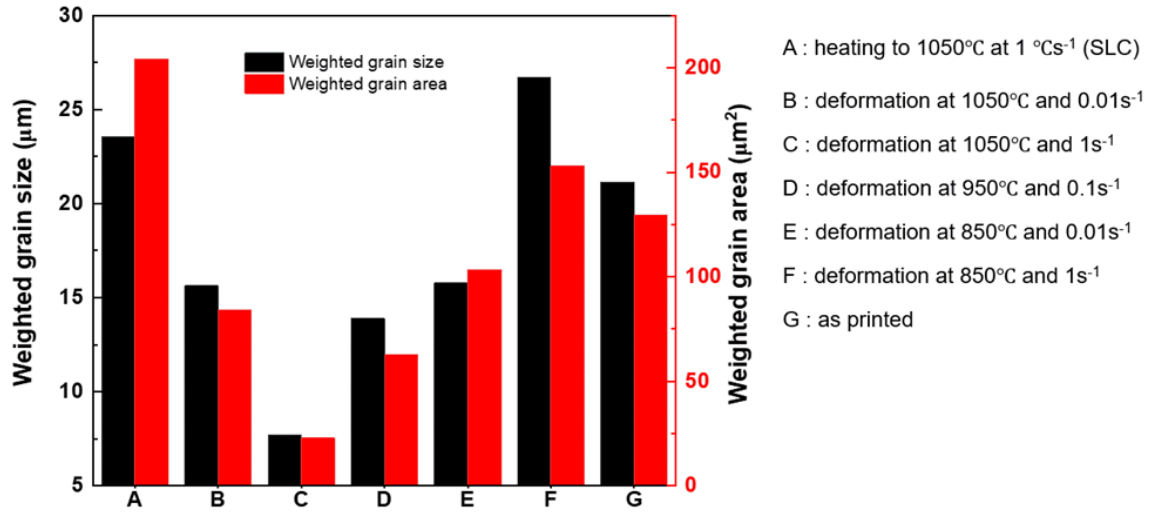


Figure 5.18. Grain size comparison between different test conditions. (SLC – heating and cooling rate of 1°C-s⁻¹, AP-as-printed).

A summary table displaying the percentage of grains with a shape factor of less than 2, identified as recrystallized grains, is presented in **Table 5.1**. This data is combined with the results of Johnson-Mehl-Avrami-Kolmogorov modelling, as illustrated in **Fig. 5.10**. This analysis leads to a graphical representation of the relationship between the fraction of recrystallized grains and strain, shown in **Fig. 5.19**. For simplicity, the effect of dynamic recovery is neglected in this plot.

The line plots reveal the kinetics of fractional softening due to dynamic recrystallization at 850°C and 1050 °C. At 1050 °C, the differential softening slope ($dX/d\varepsilon$) between low and high strain rates is 6.60 and 9.95, respectively, indicating a difference of 3.35. This significant DRX occurrence at higher strain rates (from $\varepsilon=0.5$) facilitates the formation of smaller grains, evidenced by a weighted grain diameter of 7.71 μm . Conversely, at lower

strain rates, prolonged deformation at relatively high temperatures results in significant grain growth, leading to a grain diameter of 15.63 μm .

At 850 °C, consistent with the literature and previous discussions, a lower strain rate is shown to favour a higher fraction of DRX grains, along with finer grains. From **Fig. 5.19**, the difference in the softening slope between low and high strain rates is relatively minor, at 1.08, approximately three times lower than the results from the 1050°C tests; thus, the grain sizes are not significantly influenced by dynamic fractional softening. However, the extended deformation time at lower strain rates promotes the formation of a higher fraction of DRX grains, a topic elaborated on later in this section.

The dynamic progression of the fraction of DRX grains can be traced from line A, B, C, and D in **Fig. 5.19**, corresponding to applied strains of 0.3, 0.4, 0.5, and 0.6, respectively. Utilizing the percentage of DRX grains (i.e., after an applied strain of 0.65) obtained from EBSD analysis, specifically the shape factor data, the estimated DRX grains are documented in **Table 5.2**. The evolution of DRX grains associated with different test conditions is evident, aligning with DRX critical strains depicted in **Fig. 5.8**. This analysis is crucial for predicting grain evolution during hot working processes.

Table 5.1. Estimated fraction of recrystallized grains using weighted shape factor.

Testing condition	Grain diameter (μm)	Grains with shape factor of <2 (%)	Average shape factor	Softening slope ($dX/d\varepsilon$)
1050 °C, 1 s^{-1}	7.71	46.04	2.02	9.85
1050 °C, 0.01 s^{-1}	15.63	43.53	1.91	6.60
850 °C, 1 s^{-1}	26.66	26.93	2.70	4.02
850 °C, 0.01 s^{-1}	15.79	46.75	2.02	2.94

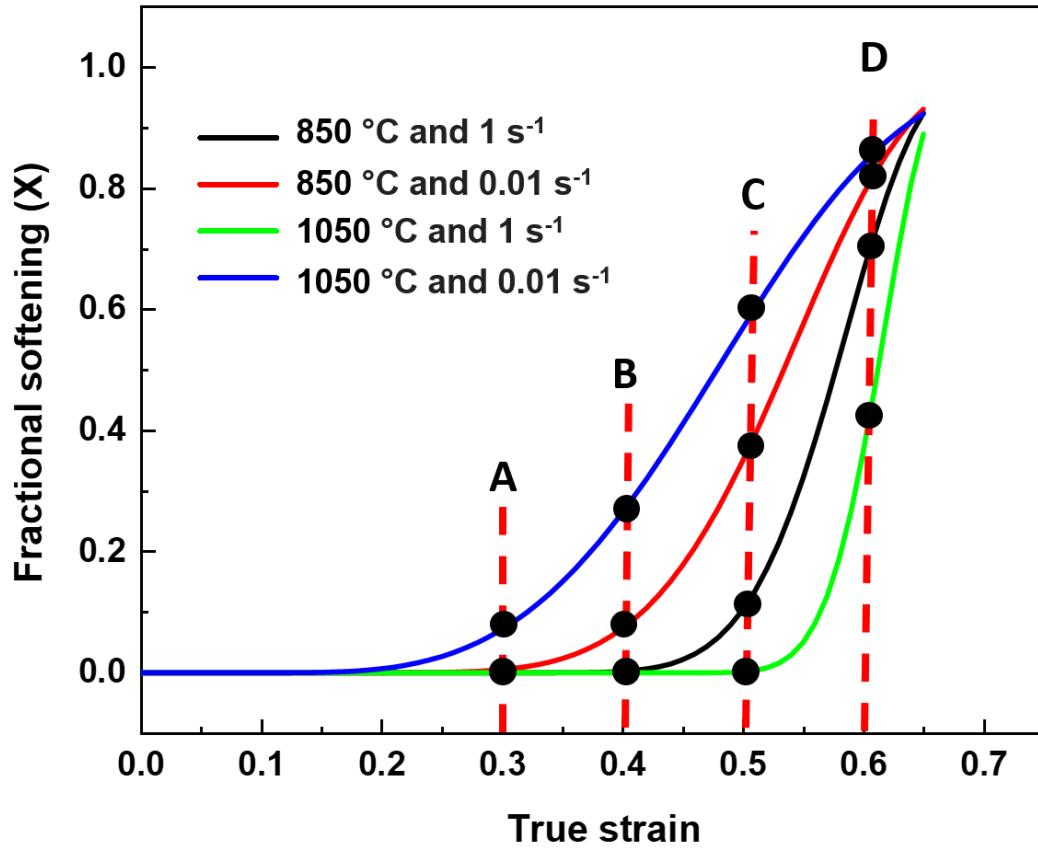


Figure 5.19. The dependence of fractional softening on applied strain based on Johnson-Mehl-Avrami-Kolmogorov model of Fig. 5.10.

Table 5.2. Evolution of DRX grains based on shape factor data from Table 5.1.

Testing condition	DRX grains from EBSD at $\varepsilon=0.7$ (%)	Projected DRX grains based on JMAK plot (in %)			
		A ($\varepsilon=0.30$)	B ($\varepsilon=0.40$)	C ($\varepsilon=0.50$)	D ($\varepsilon=0.60$)
1050 °C, 1 s ⁻¹	46.04	0.0	0.0	0.0	19.7
1050 °C, 0.01 s ⁻¹	43.53	3.2	12.0	25.9	37.5
850 °C, 1 s ⁻¹	26.93	0.0	0.0	3.2	19.1
850 °C, 0.01 s ⁻¹	46.75	0.0	3.7	17.7	38.2

In the studied material, the degree of recrystallization at high temperatures (1050 °C) is enhanced at high strain rates, whereas at lower temperatures (850 °C), recrystallization is more pronounced at slower strain rates. This phenomenon is proposed to occur due to discontinuous dynamic recrystallization (DDRX); wherein strain-free grains nucleate at

regions dense with dislocations [8,9]. The formation of new high grain boundaries from strain-hardened austenite in the maraging alloy is often impeded by alloying elements such as Mo, Co, and Ti, with this effect being more pronounced at lower temperatures [81].

As observed in both the child and parent phases from EBSD analysis, there is a concentration of nucleated grains along grain boundaries in the deformed microstructure that bulges from pre-existing grain boundaries. The occurrence of these fine equiaxed grains along the boundaries can be associated with the stacking fault energy (SFE) of M789 steel. M789 steel has a low SFE of about 33 mJ/m² at room temperature, which increases to about 250 mJ/m² at 1050 °C. Metals with low SFE tend to exhibit a high degree of dislocation dissociation into partial dislocations, which are energetically more favourable [82]. The presence of these partial dislocations retards dynamic recovery by inhibiting dislocation climb and cross-slip, and they tend to accumulate at the grain boundaries as statistically stored dislocations (SSDs) [83]. Moreover, at the high strain rate used in this study, the accumulating dislocations increase the stored energy necessary for recrystallization. In contrast, while the effect of high temperature on dislocation mobility and annihilation cannot be ignored, the brevity of the compression test at 1 s⁻¹ suggests that dislocation pile-up at boundaries is predominant. A significant number of dislocations are proposed to still exist, providing enough stored energy for recrystallization.

However, in tests at 1050 °C and 0.01 s⁻¹ (**Figs. 5.15e** and **5.15f**), where the specimen was exposed to high temperature for 70 seconds, the degree of recrystallization along the grain boundaries was reduced, thereby increasing the rate of recovery and growth. This trend is opposite to the common observation that high temperatures and slow strain rates favour increased grain boundary mobility and the annihilation of dislocations [5,9]. Dynamic

recovery, characterized by the rearrangement and annihilation of dislocations, consumes stored energy to form low-energy configurations [30]. The existence of rapid dynamic recovery at 1050 °C and 0.01 s⁻¹ can be associated with the annihilation of dislocations necessary to sustain DDRX. This is reflected in the high SFE at 1050 °C, which favours dislocation glide, climb, and cross-slip [84].

5.6.3 Texture analysis and variant selection during hot deformation

The connection between deformation and solidification texture was examined using inverse pole figures (IPF) maps with reference to the building and compression directions. A texture component of <111> parallel to the building direction, similar to the as-printed condition [15,16,45], is observed for all deformation conditions (**Figs. 5.20a, 5.20c, and 5.20d** for tests at 850 °C and 1 s⁻¹, 850 °C and 0.01 s⁻¹, and 1050 °C and 0.01 s⁻¹, respectively), except in the case of the deformed sample at 1050 °C and 1 s⁻¹ (**Fig. 5.20b**). Cubic metals ideally solidify with a preferred <100> orientation, where grains with this family of directions align parallel to the heat flux, outgrowing grains with differing orientations [85]. However, in LPBF, the scanning strategy primarily aims to randomize grain orientation by changing the heat flow direction between successive layers [86], often producing a texture-free material. Nonetheless, the printing of M789 steel reportedly produces a weak <111> texture parallel to the build direction [16].

A variant selection analysis is displayed in **Fig. 5.21** using the K-S relationship. Notably, in most samples tested in this work (specifically in **Figs. 5.21a, 5.21b, and 5.21d**), all variants appear except for the test at 850 °C and 0.01 s⁻¹, and the as-printed state, where certain variants disappear (see **Fig. 5.21c and 5.21e**). The phenomenological theory governing austenite-martensite transformation suggests that, in the absence of variant

selection, all 24 variants are equally likely to form [87,88]. The absence of these variants may be attributed to the effects of residual stress, which influences the energy balance within the grain structure resulting in the selection of variants [87,89], warranting further investigation. Variants are favoured when the interaction energy that supplies the mechanical driving force supplements to the overall transformation driving force with the goal of minimizing the strain energy [90,91]. In contrast, the higher strain rate condition (at 850 °C and 1 s⁻¹, see **Fig. 5.21a**) shows no specific variant selection despite experiencing the highest deformation stress. Despite the lack of variant selection, the test at 850 °C and 1 s⁻¹ condition exhibits the strongest texture component near <111> with a maximum intensity of 3.5 (**Fig. 5.20a**). In the parent grain, this texture component is about 2, and it intensifies during transformation to martensite. This observation may be due to preferential martensite transformation along localized stress regions [92].

Recrystallization followed by phase transformation produces a more random texture in the test at 1050 °C and 1 s⁻¹, with a near <123> parallel to the building direction, as seen in **Fig. 5.20b**. With reconstruction, the texture of the deformed austenite can be identified, as presented in **Fig. 5.22**. Compressed FCC structures have a preferred <110> deformation texture [93], which is prevalent in all conditions (**Figs. 5.22a, 5.22c, and 5.22d** for tests at 850 °C and 1 s⁻¹, 850 °C and 0.01 s⁻¹, and 1050 °C and 0.01 s⁻¹, respectively), except in the highly recrystallized condition in **Fig. 5.22b** for the deformed sample at 1050 °C and 1 s⁻¹. Under high flow stress conditions, the crystal lattice reorients itself by aligning the normal to the active slip plane with the compression direction. This rotation occurs near <110>, resulting in an overall rotation towards the <111> direction upon transformation to martensite according to the K-S OR relationship.

Texture analysis in the SLC condition, where the degree of recrystallization is negligible (**Fig. 5.17b**), was done to isolate texture evolution via deformation or residual cooling strain. In the absence of deformation, strain, and recrystallization, a strong texture developed near the $\langle 111 \rangle$ and $\langle 113 \rangle$ directions parallel to the BD (with intensities of 2.5 and 4, respectively). This suggests that for the test at 1050 °C and 1 s^{-1} , the observed random texture is due to recrystallization. The recrystallized condition shows a randomized texture with a weak $\langle 123 \rangle$ with an intensity of 1.4, associated with the nucleation of dislocation-free grains with randomized orientations. Furthermore, for the test at 850 °C and 0.01 s^{-1} , where the degree of recrystallization is evident, the most favourable texture is near the $\langle 123 \rangle$ direction (**Fig. 5.22c**), similar to the weak texture developed in the test at 1050 °C and 1 s^{-1} . However, due to the higher flow stress at 850 °C compared to 1050 °C, a weak $\langle 011 \rangle$ is seen from **Fig. 5.22c**, associated with the preferred deformation texture in FCC material. For the test at 1050 °C and 0.01 s^{-1} , the texture component is midway between the preferred $\langle 001 \rangle$ and $\langle 011 \rangle$, with the deformation texture having a stronger component than the solidification texture. At high temperatures and low strain rates, cubic systems exhibit a texture shift towards the $\langle 100 \rangle$ crystallographic direction. Extended time under these conditions promotes grain recovery and growth, with dynamic recovery emerging as the primary softening mechanism [33].

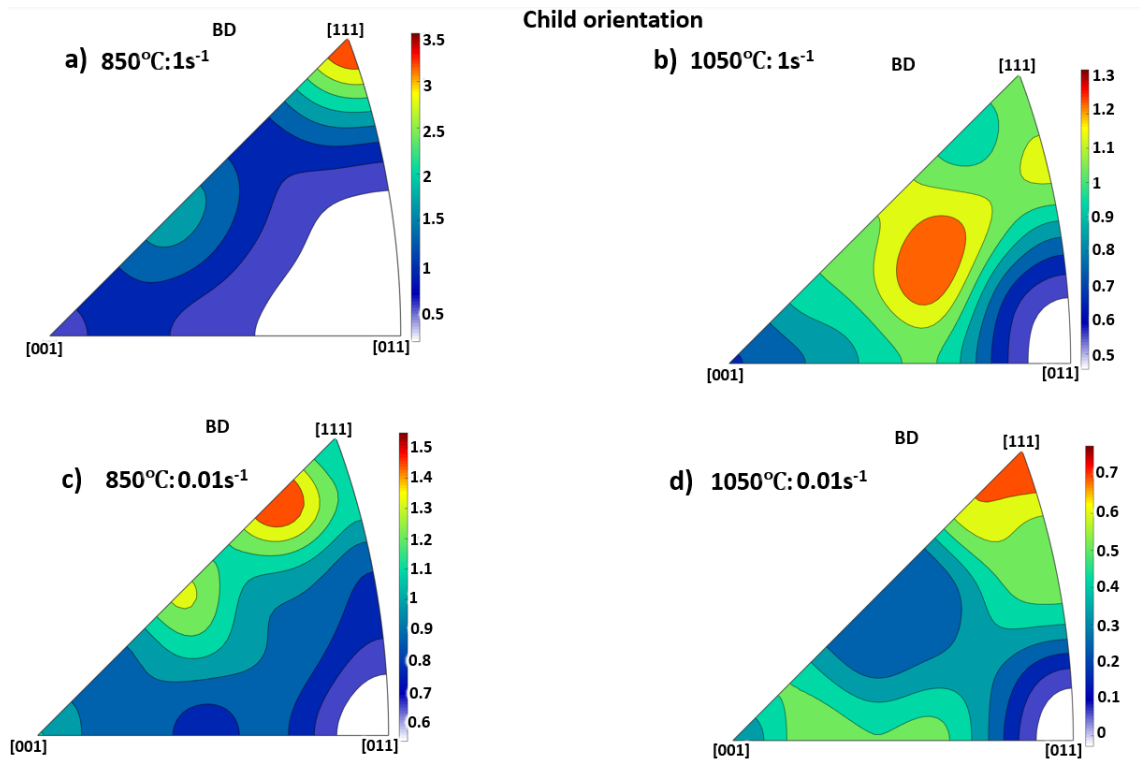
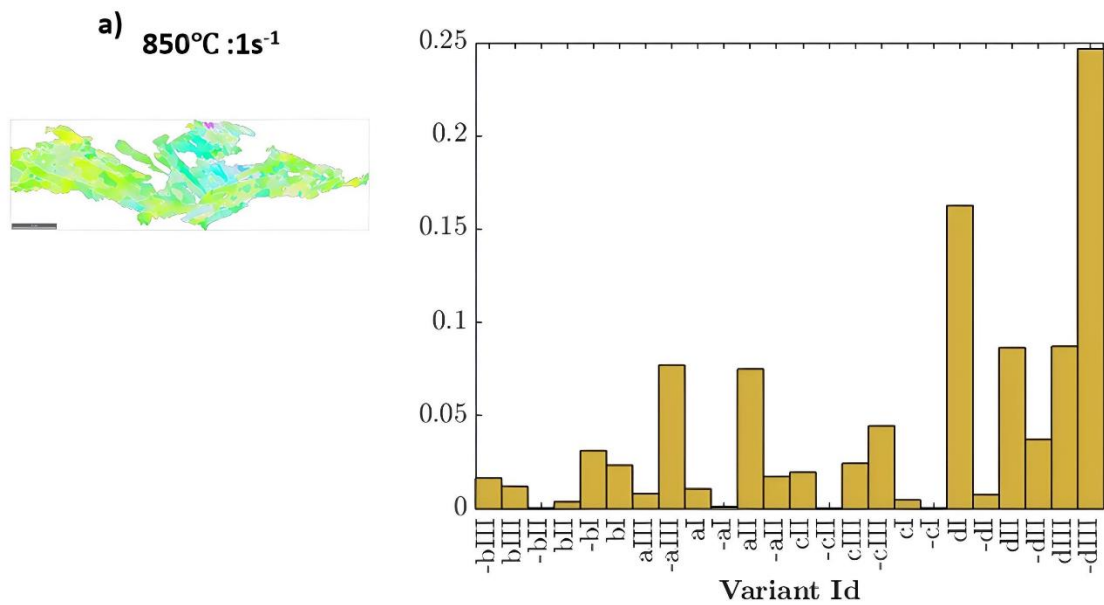
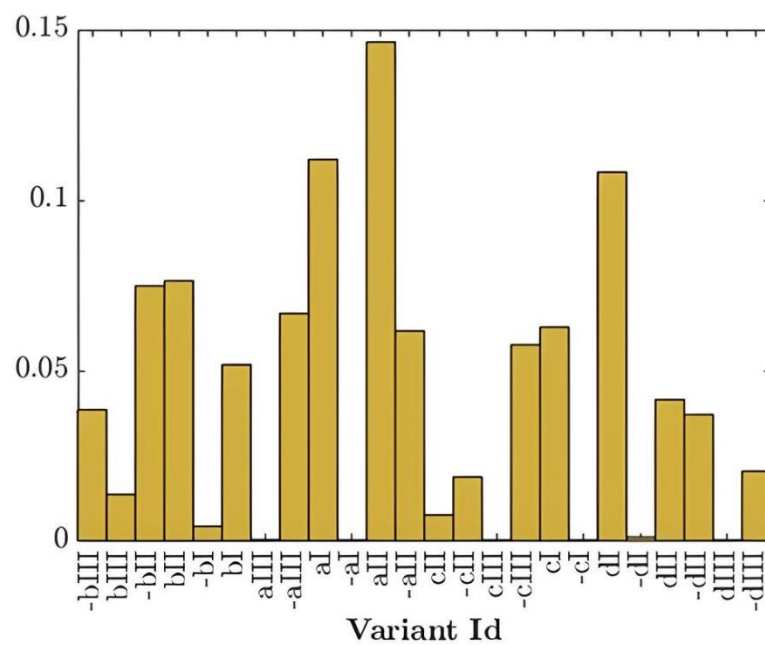
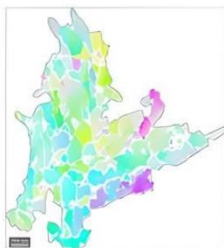


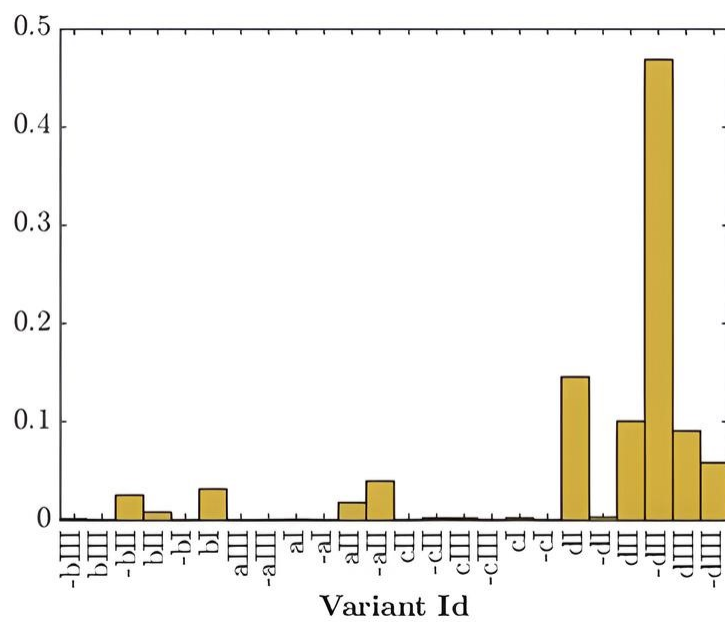
Figure 5.20. Child orientation with respect to the build direction for tests at: a) 850 °C and 1 s⁻¹, b) 1050 °C and 1 s⁻¹, c) 850 °C and 0.01 s⁻¹, and 1050 °C and 0.01 s⁻¹.



b) 1050°C :1s⁻¹



c) 850°C :0.01s⁻¹



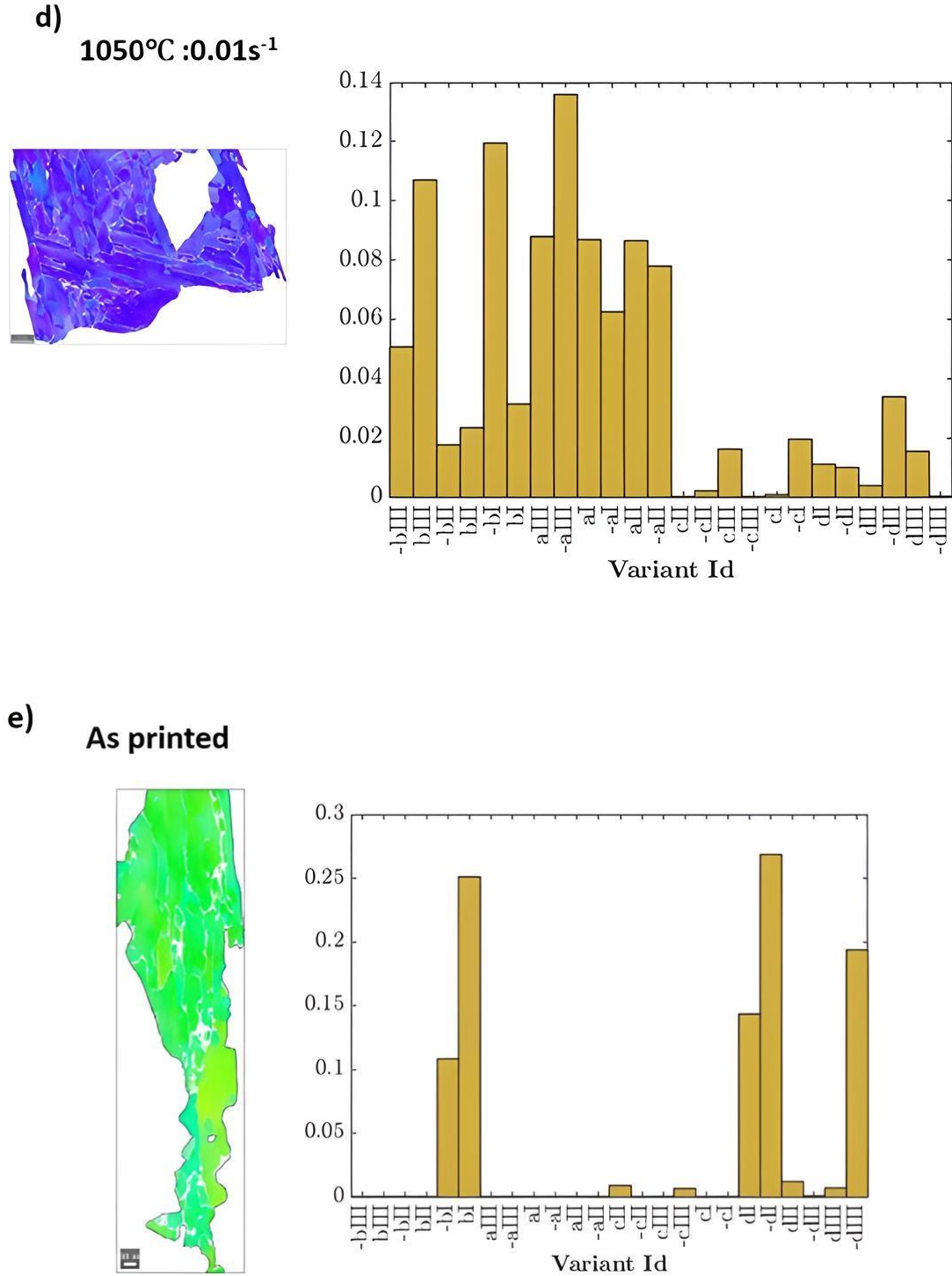


Figure 5.21. Variant selection using KS relationship for samples deformed at: a) 850°C and 1s^{-1} , b) 1050°C and 1s^{-1} , c) 850°C and 0.01s^{-1} , d) 1050°C and 0.01s^{-1} , and e) as-printed condition (undeformed).

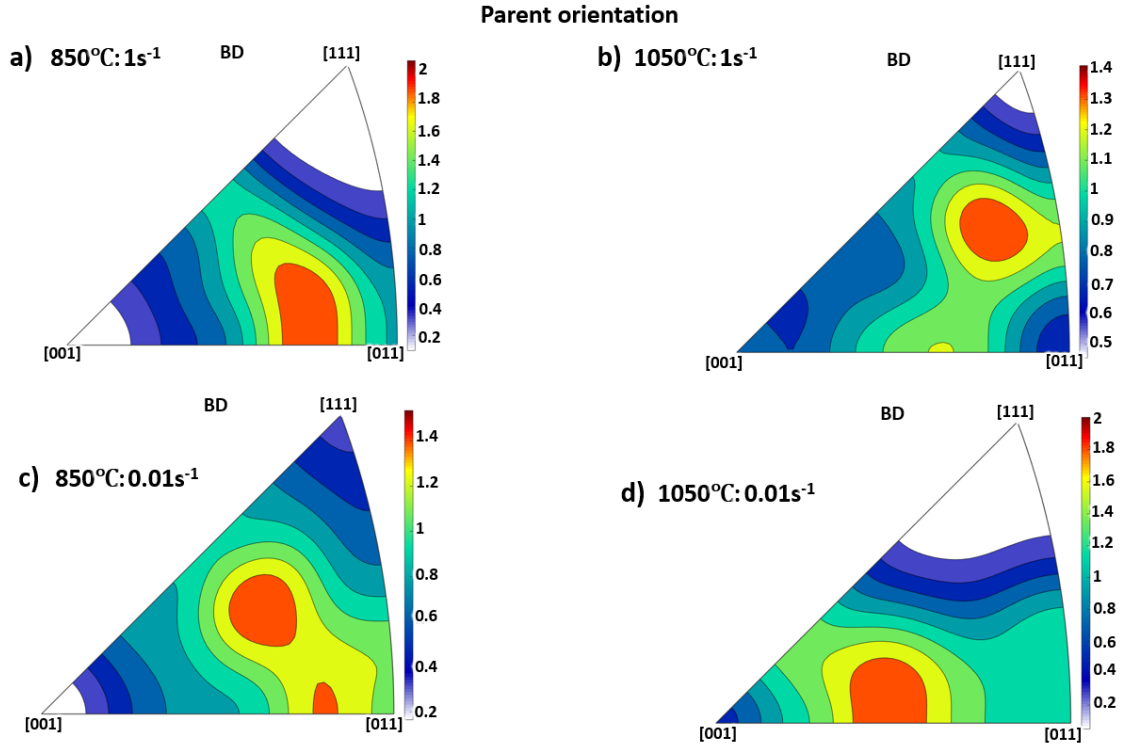


Figure 5.22. Parent orientation for deformed samples at: a) 850 °C and 1 s⁻¹, b) 1050 °C and 1 s⁻¹, c) 850 °C and 0.01 s⁻¹, and d) 1050 °C and 0.01s⁻¹.

5.7 Conclusion

In this study, the high-temperature properties of LPBF-fabricated M789 steel were investigated across a temperature range of 850-1050 °C and strain rates from 0.01 to 1 s⁻¹. Based on mechanical and microstructural analyses of undeformed and deformed samples, the following conclusions were drawn:

1. Uncorrected stress-strain data from high-temperature compression tests can result in inaccuracies of up to 10% due to friction and adiabatic heating effects. This work demonstrates effective methods for correcting these inaccuracies. Friction correction is achieved by measuring the dimensions of the material post-deformation, while temperature correction is conducted through dynamic

monitoring of the sample's temperature increase during the test. Combining these approaches allows for the calculation of accurate high-temperature stress-strain data, leading to improved accuracy in measuring mechanical responses at elevated temperatures.

2. Utilizing the corrected data, constitutive material models were developed. The Kocks-Mecking model and the Johnson-Mehl-Avrami-Kolmogorov equation proved to be the most accurate, with the lowest AAREs of 4%. These models effectively captured the high-temperature stress-strain response of LPBF-fabricated M789 steel within the tested temperature and strain rate range.
3. The activation energy for the deformation of M789 steel was determined to be lower than that of its closest conventional counterpart, M250 steel. This indicates unique deformation characteristics, potentially making it easier to recrystallize under high-temperature conditions for the LPBF-fabricated steel. Recrystallization in M789 steel was observed to occur readily at 1050 °C under high strain rates (1 s^{-1}), and at 850 °C under low strain rates (0.01 s^{-1}), with grains refined to as small as 8 μm . Effective analysis of dynamically recrystallized grains in martensitic steels can be achieved through the grain reconstruction of the parent austenite phase from the child phase, complemented by shape factor analysis.
4. The stress exponent value of 3.42 obtained in this study indicates that dislocation climb is the predominant rate-controlling mechanism governing plastic deformation within the tested range.
5. In the microstructural examination of as-printed and deformed samples at 850 °C and a strain rate of 0.01 s^{-1} , it was observed that specific variants from the K-S OR

are favoured during the solidification process in LPBF. These variants seem to disappear when the samples are subjected to austenitization temperatures and subsequently cooled to form martensitic grains.

5.8 References

- [1] C. Turk, H. Zunko, C. Aumayr, H. Leitner, and M. Kapp, ‘Advances in Maraging Steels for Additive Manufacturing’, BHM Berg- Hüttenmänn. Monatshefte, vol. 164, no. 3, pp. 112–116, Mar. 2019, doi: 10.1007/s00501-019-0835-z.
- [2] J. Pasco, Y. Tian, K. Chadha, L. Jiang, T. Dorin, and C. Aranas Jr, ‘Unusual interface phase transformation during continuous additive manufacturing of maraging steel and Co–30Cr–7Mo alloy’, Mater. Sci. Eng. A, vol. 881, p. 145336, Aug. 2023, doi: 10.1016/j.msea.2023.145336.
- [3] M. Nageswara Rao and S. V. S. Narayana Murty, ‘Hot Deformation of 18 % Ni Maraging Steels: A Review’, Mater. Perform. Charact., vol. 8, no. 5, p. 20190027, May 2019, doi: 10.1520/MPC20190027.
- [4] G. H. D. O. Freitas and C. A. S. D. Oliveira, ‘Effect of Hot Deformation on Microstructure, Hardness and Precipitation Kinetics in a C350 Maraging Steel Modified by Titanium Addition’, Mater. Res., vol. 21, no. 6, Sep. 2018, doi: 10.1590/1980-5373-mr-2018-0120.
- [5] S. S. Ansari, J. Mukhopadhyay, and S. V. S. N. Murty, ‘Analysis of Stress-Strain Curves to Predict Dynamic Recrystallization During Hot Deformation of M300 Grade Maraging Steel’, J. Mater. Eng. Perform., vol. 30, no. 8, pp. 5557–5567, Aug. 2021, doi: 10.1007/s11665-021-05780-7.

- [6] B. Blakey-Milner et al., ‘Metal additive manufacturing in aerospace: A review’, *Mater. Des.*, vol. 209, p. 110008, Nov. 2021, doi: 10.1016/j.matdes.2021.110008.
- [7] O. Abdulhameed, A. Al-Ahmari, W. Ameen, and S. H. Mian, ‘Additive manufacturing: Challenges, trends, and applications’, *Adv. Mech. Eng.*, vol. 11, no. 2, p. 168781401882288, Feb. 2019, doi: 10.1177/1687814018822880.
- [8] K. K. Alaneme and E. A. Okotete, ‘Recrystallization mechanisms and microstructure development in emerging metallic materials: A review’, *J. Sci. Adv. Mater. Devices*, vol. 4, no. 1, pp. 19–33, Mar. 2019, doi: 10.1016/j.jsamd.2018.12.007.
- [9] K. Huang and R. E. Logé, ‘A review of dynamic recrystallization phenomena in metallic materials’, *Mater. Des.*, vol. 111, pp. 548–574, Dec. 2016, doi: 10.1016/j.matdes.2016.09.012.
- [10] S. Syed Ansari, K. V. A. Chakravarthi, S. V. S. Narayana Murty, B. Nageswara Rao, and J. Mukhopadhyay, ‘Hot Workability and Microstructure Control through the Analysis of Stress–Strain Curves during Hot Deformation of M350 Grade Maraging Steel’, *Mater. Perform. Charact.*, vol. 8, no. 5, p. 20190030, May 2019, doi: 10.1520/MPC20190030.
- [11] H. Lee et al., ‘Influence of Segregation on Microstructure and Hot Workability of Grade 250 Maraging Steel’, *Met. Mater. Int.*, vol. 27, no. 4, pp. 691–704, Apr. 2021, doi: 10.1007/s12540-020-00771-0.
- [12] B. Tomiczek, P. Snopiński, W. Borek, M. Król, A. R. Gutiérrez, and G. Matula, ‘Hot Deformation Behaviour of Additively Manufactured 18Ni-300 Maraging Steel’, *Materials*, vol. 16, no. 6, p. 2412, Mar. 2023, doi: 10.3390/ma16062412.

- [13] M. Bambach et al., ‘On the hot deformation behaviour of Ti-6Al-4V made by additive manufacturing’, *J. Mater. Process. Technol.*, vol. 288, p. 116840, Feb. 2021, doi: 10.1016/j.jmatprotec.2020.116840.
- [14] A. S. J. Al-Zubaydi et al., ‘The hot deformation behaviour of laser powder bed fusion deposited Al–Si–Cu alloy processed by high-pressure torsion’, *J. Mater. Sci.*, vol. 57, no. 43, pp. 20402–20418, Nov. 2022, doi: 10.1007/s10853-022-07847-9.
- [15] Y. Tian, K. Nyamuchiwa, K. Chadha, Y. He, and C. Aranas, ‘Laser powder bed fusion of M789 maraging steel on Cr–Mo N709 steel: Microstructure, texture, and mechanical properties’, *Mater. Sci. Eng. A*, vol. 839, p. 142827, Apr. 2022, doi: 10.1016/j.msea.2022.142827.
- [16] K. Nyamuchiwa, Y. Tian, K. Chadha, L. Jiang, T. Dorin, and C. Aranas, ‘Precipitation Behaviour at the Interface of an Additively Manufactured M789–N709 Hybrid Alloy’, *Acta Metall. Sin. Engl. Lett.*, May 2023, doi: 10.1007/s40195-023-01558-z.
- [17] P. Christiansen, P. A. F. Martins, and N. Bay, ‘Friction Compensation in the Upsetting of Cylindrical Test Specimens’, *Exp. Mech.*, vol. 56, no. 7, pp. 1271–1279, Sep. 2016, doi: 10.1007/s11340-016-0164-z.
- [18] J. Obiko, ‘Friction correction of flow stress-strain curve in the upsetting process’, *IOP SciNotes*, vol. 2, no. 1, p. 014401, Mar. 2021, doi: 10.1088/2633-1357/abdd96.
- [19] D. Gomez-Marquez, E. Ledesma-Orozco, R. Hino, E. Aguilera-Gomez, G. Korpała, and U. Prah, ‘Numerical study on the hot compression test for bulk metal forming application’, *SN Appl. Sci.*, vol. 4, no. 8, p. 220, Aug. 2022, doi: 10.1007/s42452-022-05093-x.

- [20] A. Laasraoui and J. J. Jonas, 'Prediction of steel flow stresses at high temperatures and strain rates', *Metall. Trans. A*, vol. 22, no. 7, pp. 1545–1558, Jul. 1991, doi: 10.1007/BF02667368.
- [21] S. Ataya and T. Emde, 'Friction Coefficients on Compression Testing of AA6060 and 42CrMo4 with Different Lubrication Conditions'. Feb. 01, 2014. doi: 10.1002/9781118888438.ch64.
- [22] M. Cook and E. C. Larke, 'Resistance of copper and copper alloys to homogeneous deformation in compression', 1945. [Online]. Available: <https://api.semanticscholar.org/CorpusID:136722227>
- [23] H. J. McQueen and N. D. Ryan, 'Constitutive analysis in hot working', *Mater. Sci. Eng. A*, vol. 322, no. 1–2, pp. 43–63, Jan. 2002, doi: 10.1016/S0921-5093(01)01117-0.
- [24] Y. C. Lin and X.-M. Chen, 'A critical review of experimental results and constitutive descriptions for metals and alloys in hot working', *Mater. Des.*, vol. 32, no. 4, pp. 1733–1759, Apr. 2011, doi: 10.1016/j.matdes.2010.11.048.
- [25] G. R. Johnson and W. H. Cook, 'A Constitutive Model And Data For Metals', p. 7.
- [26] D. Fields and W. Backofen, 'Determination of strain hardening characteristics by torsion testing', in *Proc. ASTM*, 1957, pp. 1259–1272.
- [27] Y. Q. Cheng, H. Zhang, Z. H. Chen, and K. F. Xian, 'Flow stress equation of AZ31 magnesium alloy sheet during warm tensile deformation', *J. Mater. Process. Technol.*, vol. 208, no. 1–3, pp. 29–34, 2008.
- [28] E. Voce, 'The relationship between stress and strain for homogeneous deformation', *J. Inst. Met.*, vol. 74, pp. 537–562, 1948.

- [29] U. F. Kocks, ‘Laws for work-hardening and low-temperature creep’, 1976.
- [30] F. Montheillet, D. Piot, N. Matougui, and M. L. Fares, ‘A Critical Assessment of Three Usual Equations for Strain Hardening and Dynamic Recovery’, *Metall. Mater. Trans. A*, vol. 45, no. 10, pp. 4324–4332, Sep. 2014, doi: 10.1007/s11661-014-2388-9.
- [31] Y. Bergström, ‘A dislocation model for the stress-strain behaviour of polycrystalline α -Fe with special emphasis on the variation of the densities of mobile and immobile dislocations’, *Mater. Sci. Eng.*, vol. 5, no. 4, pp. 193–200, 1970.
- [32] Y. Estrin and H. Mecking, ‘A unified phenomenological description of work hardening and creep based on one-parameter models’, *Acta Metall.*, vol. 32, no. 1, pp. 57–70, Jan. 1984, doi: 10.1016/0001-6160(84)90202-5.
- [33] H. Mecking, ‘Strain hardening and dynamic recovery’, in *Dislocation Modelling of Physical Systems*, Elsevier, 1981, pp. 197–211. doi: 10.1016/B978-0-08-026724-1.50026-1.
- [34] F. J. Zerilli and R. W. Armstrong, ‘Dislocation-mechanics-based constitutive relations for material dynamics calculations’, *J. Appl. Phys.*, vol. 61, no. 5, pp. 1816–1825, Mar. 1987, doi: 10.1063/1.338024.
- [35] F. J. Zerilli, ‘Dislocation mechanics-based constitutive equations’, *Metall. Mater. Trans. A*, vol. 35, no. 9, pp. 2547–2555, Sep. 2004, doi: 10.1007/s11661-004-0201-x.
- [36] K. Ono and A. W. Sommer, ‘Peierls-Nabarro hardening in the presence of point obstacles’, *Metall. Trans.*, vol. 1, pp. 877–884, 1970.

- [37] R. Liang and A. S. Khan, ‘A critical review of experimental results and constitutive models for BCC and FCC metals over a wide range of strain rates and temperatures’, *Int. J. Plast.*, vol. 15, no. 9, pp. 963–980, Jan. 1999, doi: 10.1016/S0749-6419(99)00021-2.
- [38] G. Z. Voyiadjis and F. H. Abed, ‘Microstructural based models for bcc and fcc metals with temperature and strain rate dependency’, *Mech. Mater.*, vol. 37, no. 2–3, pp. 355–378, Feb. 2005, doi: 10.1016/j.mechmat.2004.02.003.
- [39] M. Avrami, ‘Kinetics of phase change. II transformation-time relations for random distribution of nuclei’, *J. Chem. Phys.*, vol. 8, no. 2, pp. 212–224, 1940.
- [40] A. M. Jorge Junior and O. Balancin, ‘Prediction of steel flow stresses under hot working conditions’, *Mater. Res.*, vol. 8, no. 3, pp. 309–315, Sep. 2005, doi: 10.1590/S1516-14392005000300015.
- [41] A. M. Jorge, W. Regone, and O. Balancin, ‘Effect of competing hardening and softening mechanisms on the flow stress curve modelling of ultra-low carbon steel at high temperatures’, *J. Mater. Process. Technol.*, vol. 142, no. 2, pp. 415–421, Nov. 2003, doi: 10.1016/S0924-0136(03)00620-4.
- [42] Y. Zhang, Q. Fan, X. Zhang, Z. Zhou, Z. Xia, and Z. Qian, ‘Avrami Kinetic-Based Constitutive Relationship for Armco-Type Pure Iron in Hot Deformation’, *Metals*, vol. 9, no. 3, p. 365, Mar. 2019, doi: 10.3390/met9030365.
- [43] J. J. Jonas, X. Queleennec, L. Jiang, and É. Martin, ‘The Avrami kinetics of dynamic recrystallization’, *Acta Mater.*, vol. 57, no. 9, pp. 2748–2756, May 2009, doi: 10.1016/j.actamat.2009.02.033.

- [44] M. K. Razali and M. S. Joun, ‘A new approach of predicting dynamic recrystallization using directly a flow stress model and its application to medium Mn steel’, *J. Mater. Res. Technol.*, vol. 11, pp. 1881–1894, Mar. 2021, doi: 10.1016/j.jmrt.2021.02.026.
- [45] R. Palad, Y. Tian, K. Chadha, S. Rodrigues, and C. Aranas, ‘Microstructural features of novel corrosion-resistant maraging steel manufactured by laser powder bed fusion’, *Mater. Lett.*, vol. 275, p. 128026, Sep. 2020, doi: 10.1016/j.matlet.2020.128026.
- [46] G. Kurdjumov and G. Sachs, ‘Oberden mechanismus det stahlhirtung’, *Z Phys.*, vol. 64, p. 225, 1930.
- [47] F. Niessen, T. Nyysönen, A. A. Gazder, and R. Hielscher, ‘Parent grain reconstruction from partially or fully transformed microstructures in MTEX’, *J. Appl. Crystallogr.*, vol. 55, no. 1, pp. 180–194, Feb. 2022, doi: 10.1107/S1600576721011560.
- [48] Y. P. Li, E. Onodera, H. Matsumoto, and A. Chiba, ‘Correcting the Stress-Strain Curve in Hot Compression Process to High Strain Level’, *Metall. Mater. Trans. A*, vol. 40, no. 4, pp. 982–990, Apr. 2009, doi: 10.1007/s11661-009-9783-7.
- [49] R. Ebrahimi and A. Najafizadeh, ‘A new method for evaluation of friction in bulk metal forming’, *J. Mater. Process. Technol.*, vol. 152, no. 2, pp. 136–143, Oct. 2004, doi: 10.1016/j.jmatprotec.2004.03.029.
- [50] L. G. D. Carvalho, M. S. Andrade, R. L. Plaut, F. M. Souza, and A. F. Padilha, ‘A dilatometric study of the phase transformations in 300 and 350 maraging steels during continuous heating rates’, *Mater. Res.*, vol. 16, no. 4, pp. 740–744, May 2013, doi: 10.1590/S1516-14392013005000069.

- [51] R. Kapoor, L. Kumar, and I. S. Batra, ‘A dilatometric study of the continuous heating transformations in 18wt.% Ni maraging steel of grade 350’, *Mater. Sci. Eng. A*, vol. 352, no. 1–2, pp. 318–324, Jul. 2003, doi: 10.1016/S0921-5093(02)00934-6.
- [52] Z. Chen et al., ‘Thermo-mechanics Driven Dynamic Recrystallization Behavior and Mechanism in High Strength Martensitic Stainless Steel’, *Metall. Mater. Trans. A*, vol. 54, no. 9, pp. 3503–3518, Sep. 2023, doi: 10.1007/s11661-023-07106-9.
- [53] Y. C. Lin, X.-M. Chen, and G. Liu, ‘A modified Johnson–Cook model for tensile behaviours of typical high-strength alloy steel’, *Mater. Sci. Eng. A*, vol. 527, no. 26, pp. 6980–6986, Oct. 2010, doi: 10.1016/j.msea.2010.07.061.
- [54] C. M. Sellars and W. J. McTegart, ‘On the mechanism of hot deformation’, *Acta Metall.*, vol. 14, no. 9, pp. 1136–1138, Sep. 1966, doi: 10.1016/0001-6160(66)90207-0.
- [55] C. Zener and J. H. Hollomon, ‘Effect of Strain Rate Upon Plastic Flow of Steel’, *J. Appl. Phys.*, vol. 15, no. 1, pp. 22–32, Jan. 1944, doi: 10.1063/1.1707363.
- [56] J. Uvira and J. Jonas, ‘Hot compression of armco iron and silicon steel’, *Trans Met. Soc AIME*, vol. 242, p. 9, Dec. 1967.
- [57] C. Ghosh, V. V. Basabe, and J. J. Jonas, ‘Determination of the Critical Strains for the Initiation of Dynamic Transformation and Dynamic Recrystallization in Four Steels of Increasing Carbon Contents’, *Steel Res. Int.*, vol. 84, no. 5, pp. 490–494, May 2013, doi: 10.1002/srin.201200188.
- [58] E. I. Poliak and J. J. Jonas, ‘Initiation of Dynamic Recrystallization in Constant Strain Rate Hot Deformation’, *ISIJ Int.*, vol. 43, no. 5, pp. 684–691, 2003, doi: 10.2355/isijinternational.43.684.

- [59] Y. Zhao, J. Sun, J. Li, Y. Yan, and P. Wang, ‘A comparative study on Johnson-Cook and modified Johnson-Cook constitutive material model to predict the dynamic behaviour laser additive manufacturing FeCr alloy’, *J. Alloys Compd.*, vol. 723, pp. 179–187, Nov. 2017, doi: 10.1016/j.jallcom.2017.06.251.
- [60] H. Zhu and H. Ou, ‘Constitutive modelling of hot deformation behaviour of metallic materials’, *Mater. Sci. Eng. A*, vol. 832, p. 142473, Jan. 2022, doi: 10.1016/j.msea.2021.142473.
- [61] H. J. McQueen, S. Yue, N. D. Ryan, and E. Fry, ‘Hot working characteristics of steels in austenitic state’, *J. Mater. Process. Technol.*, vol. 53, no. 1–2, pp. 293–310, Aug. 1995, doi: 10.1016/0924-0136(95)01987-P.
- [62] A. H. Cottrell and A. M. Jawson, ‘Distribution of solute atoms round a slow dislocation’, *R. Soc. Publ.*, 1949, [Online]. Available: <http://royalsocietypublishing.org>
- [63] J. C. Fisher, ‘On the strength of solid solution alloys’, *Acta Metall.*, vol. 2, no. 1, pp. 9–10, Jan. 1954, doi: 10.1016/0001-6160(54)90087-5.
- [64] A. K. Mukherjee, J. E. Bird, and J. E. Dorn, ‘Experimental correlations for high-temperature creep’, 1968.
- [65] J. Weertman, ‘Theory of steady-state creep based on dislocation climb’, *J. Appl. Phys.*, vol. 26, no. 10, pp. 1213–1217, 1955.
- [66] T. G. Langdon and F. A. Mohamed, ‘A new type of deformation mechanism map for high-temperature creep’, *Mater. Sci. Eng.*, vol. 32, no. 2, pp. 103–112, Feb. 1978, doi: 10.1016/0025-5416(78)90029-0.

- [67] F. R. N. Nabarro, Report of a conference on the strength of solids Physical Society. London, 1948.
- [68] C. Herring, 'Diffusional Viscosity of a Polycrystalline Solid', J. Appl. Phys., vol. 21, no. 5, pp. 437–445, May 1950, doi: 10.1063/1.1699681.
- [69] R. L. Coble, 'A Model for Boundary Diffusion Controlled Creep in Polycrystalline Materials', J. Appl. Phys., vol. 34, no. 6, pp. 1679–1682, Jun. 1963, doi: 10.1063/1.1702656.
- [70] J. Harper and J. E. Dorn, 'Viscous creep of aluminum near its melting temperature', Acta Metall., vol. 5, no. 11, pp. 654–665, Nov. 1957, doi: 10.1016/0001-6160(57)90112-8.
- [71] F. R. N. Nabarro, 'Steady-state diffusional creep', Philos. Mag., vol. 16, no. 140, pp. 231–237, 1967.
- [72] P. M. Hazzledine and J. H. Schneibel, 'Theory of coble creep for irregular grain structures', Acta Metall. Mater., vol. 41, no. 4, pp. 1253–1262, Apr. 1993, doi: 10.1016/0956-7151(93)90176-S.
- [73] I. M. Lifshitz, 'On the theory of diffusion-viscous flow of polycrystalline bodies', Sov Phys JETP-USSR, vol. 17, no. 4, p. 909, 1963.
- [74] W. A. Rachinger, 'Relative grain translations in the plastic flow of aluminum', J Inst Met., vol. 81, 1952.
- [75] W. Blum and P. Eisenlohr, 'Dislocation mechanics of creep', 11th Int. Conf. Creep Fract. Eng. Mater. Struct. CREEP 2008, vol. 510–511, pp. 7–13, Jun. 2009, doi: 10.1016/j.msea.2008.04.110.

- [76] M. E. Kassner, ‘New Developments in Understanding Harper–Dorn, Five-Power Law Creep and Power-Law Breakdown’, *Metals*, vol. 10, no. 10, 2020, doi: 10.3390/met10101284.
- [77] O. D. Sherby and P. M. Burke, ‘Mechanical behaviour of crystalline solids at elevated temperature’, *Prog. Mater. Sci.*, vol. 13, pp. 323–390, 1968.
- [78] M. E. Kassner and M.-T. Pérez-Prado, ‘Five-power-law creep in single phase metals and alloys’, *Prog. Mater. Sci.*, vol. 45, no. 1, pp. 1–102, Jan. 2000, doi: 10.1016/S0079-6425(99)00006-7.
- [79] A. K. Mukherjee, ‘An examination of the constitutive equation for elevated temperature plasticity’, *Mater. Sci. Eng. A*, vol. 322, no. 1, pp. 1–22, Jan. 2002, doi: 10.1016/S0921-5093(01)01115-7.
- [80] T. Allam, K. G. Pradeep, P. Köhnen, A. Marshal, J. H. Schleifenbaum, and C. Haase, ‘Tailoring the nanostructure of laser powder bed fusion additively manufactured maraging steel’, *Addit. Manuf.*, vol. 36, p. 101561, Dec. 2020, doi: 10.1016/j.addma.2020.101561.
- [81] T. S. Dolotova, V. I. Kucheryavyi, and N. V. Ul’yanova, ‘Effect of alloying elements on structural recrystallization of maraging stainless steels’, *Met. Sci. Heat Treat.*, vol. 18, no. 9, pp. 786–789, Sep. 1976, doi: 10.1007/BF00704120.
- [82] H. Bahmanpour et al., ‘Effect of stacking fault energy on deformation behaviour of cryo-rolled copper and copper alloys’, *Mater. Sci. Eng. A*, vol. 529, pp. 230–236, Nov. 2011, doi: 10.1016/j.msea.2011.09.022.
- [83] S. S. S. Kumar, T. Raghu, P. P. Bhattacharjee, G. A. Rao, and U. Borah, ‘Work hardening characteristics and microstructural evolution during hot deformation of a

- nickel superalloy at moderate strain rates', *J. Alloys Compd.*, vol. 709, pp. 394–409, 2017, doi: <https://doi.org/10.1016/j.jallcom.2017.03.158>.
- [84] S. R. Das et al., 'On the mechanism of cross-slip induced dislocation substructure formation in an high-Mn steel', *Materialia*, vol. 15, p. 101042, Mar. 2021, doi: [10.1016/j.mtla.2021.101042](https://doi.org/10.1016/j.mtla.2021.101042).
- [85] H. L. Wei, J. Mazumder, and T. DebRoy, 'Evolution of solidification texture during additive manufacturing', *Sci. Rep.*, vol. 5, no. 1, p. 16446, Nov. 2015, doi: [10.1038/srep16446](https://doi.org/10.1038/srep16446).
- [86] R. Kannan and P. Nandwana, 'Texture evolution during processing and post-processing of maraging steel fabricated by laser powder bed fusion', *Sci. Rep.*, vol. 12, no. 1, p. 6396, Apr. 2022, doi: [10.1038/s41598-022-09977-1](https://doi.org/10.1038/s41598-022-09977-1).
- [87] S. K. Giri, A. Durgaprasad, K. V. Manikrishna, C. R. Anoop, S. Kundu, and I. Samajdar, 'Exploring the origin of variant selection through martensite-austenite reconstruction', *Philos. Mag.*, vol. 99, no. 6, pp. 699–717, Mar. 2019, doi: [10.1080/14786435.2018.1552030](https://doi.org/10.1080/14786435.2018.1552030).
- [88] J. Pasco, Y. He, A. Keshavarzkermani, and C. Aranas, 'Texture Preference and Variant Analysis of Martensite Formation in Laser Powder Bed Fusion', in *TMS Annual Meeting & Exhibition*, Springer, 2024, pp. 1808–1816.
- [89] T. D. Truong, G. Asala, O. T. Ola, O. A. Ojo, and A. G. Odeshi, 'Effects of additive manufacturing process parameters and heat treatment on texture evolution and variant selection during austenite-martensite transformation in 18%Ni-M350 maraging steel', *Mater. Charact.*, vol. 204, p. 113190, Oct. 2023, doi: [10.1016/j.matchar.2023.113190](https://doi.org/10.1016/j.matchar.2023.113190).

- [90] S. Kundu and H. K. D. H. Bhadeshia, ‘Transformation texture in deformed stainless steel’, *Scr. Mater.*, vol. 55, no. 9, pp. 779–781, Nov. 2006, doi: 10.1016/j.scriptamat.2006.07.021.
- [91] N. F. Viana, C. D. S. Nunes, and H. F. G. De Abreu, ‘The variant selection in the transformation from austenite to martensite in samples of maraging-350 steel’, *J. Mater. Res. Technol.*, vol. 2, no. 4, pp. 298–302, Oct. 2013, doi: 10.1016/j.jmrt.2013.03.017.
- [92] Y. Tian, R. Palad, and C. Aranas, ‘Microstructural evolution and mechanical properties of a newly designed steel fabricated by laser powder bed fusion’, *Addit. Manuf.*, vol. 36, p. 101495, Dec. 2020, doi: 10.1016/j.addma.2020.101495.
- [93] K. H. Oh, J. S. Jeong, Y. M. Koo, and D. N. Lee, ‘The evolution of the rolling and recrystallization textures in cold-rolled Al containing high Mn austenitic steels’, *Mater. Chem. Phys.*, vol. 161, pp. 9–18, Jul. 2015, doi: 10.1016/j.matchemphys.2015.04.019.

6. Summary and Conclusion

6.1 Summary from published papers

This study aimed to explore the feasibility of applying a newly developed Co-free LPBF-M789 steel alloy, specifically designed for the LPBF process, in a functional hybrid application with wrought N709 steel. To gain insights into the current progress in hybrid alloy, a comprehensive review paper was published entitled “Recent Progress in Hybrid Additive Manufacturing of Metallic Materials.” This paper identified the current gap in the literature regarding hybrid alloys through combined conventional and additive manufacturing techniques. All the succeeding papers below were planned based on the assessment from the review paper.

The paper entitled “Laser powder bed fusion of M789 maraging steel on Cr-Mo N709 steel: microstructure, texture, and mechanical properties” explored the feasibility of printing LPBF-M789 on conventional N709 steel to determine its practical usability. Printing was successfully achieved with no defects at the interface using specified processing parameters. The resulting hybrid metal was evaluated for quality and suitability by determining its mechanical and microstructure properties. Hardness values across the two, materials M789 and N709, were assessed and complimented by tensile testing to evaluate the strength of the hybrid alloy, especially at the joining interface. The effects of direct aging were also examined to understand how mechanical properties and phase stability are affected by employing a non-conventional heat treatment.

To further understand the strength at the interface, the paper entitled “Precipitation behaviour at the interface of an additively manufactured M789-N709 hybrid alloy” has been published. In this work, the nature of the obtained interface from the previous paper

was investigated with a focus on identifying the primary strengthening mechanism. This involved quantifying the size of the interface and conducting compositional analysis across and to determine phase evolution. Thermodynamic and kinetic simulations alongside experimental analysis were performed to quantify and predict the phase evolution at the interface leading to the proposal of a primary mechanism.

Finally, the last paper entitled “Analysis of dynamic recrystallization through austenite grain reconstruction of additively manufactured martensitic M789 steel” addresses the gap in of lack of information on the high temperature behaviour of additively manufactured M789 steel, a relatively new commercial alloy that is targeted to be employed as a hybrid material in this work. Key phase transition temperatures were identified, and the properties of LPBF-fabricated M789 steel were explored to understand its high-temperature performance under extreme environments. The microstructural and textural evolution of this material under varying temperature (1050°C-850°C), and strain rates (1s^{-1} - 0.01s^{-1}) was studied, establishing the dynamic recrystallization behaviour of M789 steel. Moreover, physical models that describe the deformation behaviour under dynamic recovery and recrystallization for LPBF steel were developed to help extend the practical usability of this emerging alloy.

6.2 Conclusion

In the first part of the work, the mechanical and microstructural properties of the LPBF M789-N709 hybrid material were assessed to determine the feasibility of practical use of this new material. Based on the experimental and simulation results, the following conclusions were drawn:

- A strong metallurgical bond is formed between the M789 and N709 steels by the LPBF process, as evidenced by the fact that the material fractured either in the M789 section (as-printed) or in the N709 section (direct aging) during tensile testing, not on the interface.
- The fracture of the as-printed material on the M789 part during the tensile test may be attributed to the strengthening of the β -NiAl precipitates in the wrought N709 section before direct aging and η -phase after aging which strengthened the M789 section.
- The hardness of the M789 section after heat treatment was considerably increased, due to precipitation strengthening, while that of the base N709 section is essentially not changed.
- The manufactured M789-N709 hybrid material appears to have a randomly oriented martensitic structure after the LPBF process, with a very weak $\langle 111 \rangle // \text{BD}$ texture (maximum intensity of 1.73) before aging and after direct aging (maximum intensity of 1.34).

In the second part of the work, the elemental concentration profile and precipitation behaviour of M789-N709 hybrid system, with emphasis on the interface region, were assessed using various characterization techniques to further understand its microstructural evolution during post-processing heat treatment. Based on the results, the following conclusions were drawn:

- The EBSD analysis on M789, interface and N709 regions of the hybrid alloy system revealed that the as-printed material contains significant dislocation density, as

depicted by the measured low angle grain boundaries and Kernel average misorientations. After direct aging, the observed low angle grain boundaries, and Kernel average misorientations slightly decreased compared to that of the as-printed sample. This result is due to the low aging temperature that limits the complete removal of dislocations and the absence of solution heat treatment. Moreover, the crystallographic texture from EBSD taken from M789, interface and N709 regions suggests that a random arrangement of grains was generated, which promotes the isotropic behaviour of the material.

- An interface with a thickness of approximately 300 μm was generated after printing M789 alloy on wrought N709. The interface is characterized by the variation of Ti from 0 wt.% in the N709 region to about 1 wt.% in the M789 region. This variation results in different sizes and amounts of $\text{ETA-Ni}_3(\text{Ti,Al})$ strengthening precipitates, with a maximum fraction of precipitate of about 5% and a radius of 5 nm (near the M789 region).
- Thermodynamic and kinetic simulations were performed based on the measured chemistry of the EPMA; the results showed that $\text{ETA-Ni}_3(\text{Ti,Al})$ precipitates with sizes of approximately 1 nm started to form in the region 150 μm away from the N709 region. Moreover, the precipitate significantly increases with Ti additions of up to 0.6 wt.%. Beyond this threshold, the increase in the amount of precipitation is gradual.
- The thermodynamic and kinetic simulation performed in the interface of the M789-N709 interface is validated by EDS, EPMA and APT results. The measured precipitate size is consistent with the simulation results. Two morphologies were

detected: elongated and spherical. These precipitates contribute to the robustness of the interface.

In the third and final phase of the work, the high-temperature properties of LPBF-fabricated M789 steel were investigated across a temperature range of 850-1050 °C and strain rates from 0.01 to 1 s⁻¹. Based on mechanical and microstructural analyses of undeformed and deformed samples, the following conclusions were drawn:

- Uncorrected stress-strain data from high-temperature compression tests can result in inaccuracies of up to 10% due to friction and adiabatic heating effects. This work demonstrates effective methods for correcting these inaccuracies. Friction correction is achieved by measuring the dimensions of the material post-deformation, while temperature correction is conducted through dynamic monitoring of the sample's temperature increase during the test. Combining these approaches allows for the calculation of accurate high-temperature stress-strain data, leading to improved accuracy in measuring mechanical responses at elevated temperatures.
- Utilizing the corrected data, constitutive material models were developed. The Kocks-Mecking model and the Johnson-Mehl-Avrami-Kolmogorov equation proved to be the most accurate, with the lowest AAREs of 4%. These models effectively captured the high-temperature stress-strain response of LPBF-fabricated M789 steel within the tested temperature and strain rate range.
- The activation energy for the deformation of M789 steel was determined to be lower than that of its closest conventional counterpart, M250 steel. This indicates unique deformation characteristics, potentially making it easier to recrystallize

under high-temperature conditions for the LPBF-fabricated steel. Recrystallization in M789 steel was observed to occur readily at 1050 °C under high strain rates (1 s^{-1}), and at 850 °C under low strain rates (0.01 s^{-1}), with grains refined to as small as 8 μm . Effective analysis of dynamically recrystallized grains in martensitic steels can be achieved through the grain reconstruction of the parent austenite phase from the child phase, complemented by shape factor analysis.

- The stress exponent value of 3.42 obtained in this study indicates that dislocation climb is the predominant rate-controlling mechanism governing plastic deformation within the tested range.
- In the microstructural examination of as-printed and deformed samples at 850 °C and a strain rate of 0.01 s^{-1} , it was observed that specific variants from the K-S OR are favoured during the solidification process in LPBF. These variants seem to disappear when the samples are subjected to austenitization temperatures and subsequently cooled to form martensitic grains.

7. Contributions to Original Knowledge

This study marks the pioneering investigation into the feasibility of applying LPBF M789 to N709 as a hybrid material. It represents the first endeavour to combine the newly developed LPBF-M789 from voestalpine Additive Manufacturing Centre with a hybrid base to create a novel material. By doing so, this research provides valuable insights into the extended application and suitability of this material, crucial for mitigating the high costs associated with additive manufacturing in industry and broadening the utility of M789 to meet industry needs. The work demonstrates that high mechanical properties can be achieved by applying a non-conventional heat treatment protocol. By minimizing the printed build stock to functional regions while utilizing a cheaper substrate for the remainder, this approach offers potential cost savings without compromising performance. Furthermore, this research conducted high-temperature dynamic testing on M789 for the first time, revealing its recrystallization behaviour. This information is essential for comprehending the material's performance as it becomes increasingly prominent in industrial applications. Understanding the high-temperature behaviour also holds significance for potential further processing techniques such as hot isostatic pressing and forging post LPBF printing.

One of the main challenges in the LPBF process is the relatively slow building rate compared to other manufacturing processes. Addressing this challenge requires consideration of factors such as layer thickness, which affects building rate and introduces porosity issues. The findings of this study offer insights into mitigating these challenges through secondary processes like hot isostatic pressing or forging. These secondary processes subject the material to dynamic straining at elevated temperatures similar to

those investigated in this study, making the obtained information invaluable for designing optimal experimental conditions that yield superior properties.

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Publications

- **K. Nyamuchiwa**, J. Pasco, T. McCarthy, A. Keshavarzkermani, T. Wang, and C. Aranas, Analysis of Dynamic Recrystallization through Austenite Grain Reconstruction of Additively Manufactured Martensitic M789 Steel, Mater. Sci. Eng. A 897, 146345 (2024).

- **K. Nyamuchiwa**, A. Keshavarzkermani, and C. Aranas, Effects of Friction and Deformation Heating on Additively Manufactured M789 Steel During Hot Compression Tests, TMS Annual Meeting & Exhibition (2024), 172–180.
- H. Bongao, J. Pasco, T. McCarthy, **K. Nyamuchiwa**, and C. Aranas, Softening Mechanisms in Additively Manufactured 420 Stainless Steel at Elevated Temperatures, TMS Annual Meeting & Exhibition (2024), 244–253.
- F. Zvavamwe, M. Paek, **K. Nyamuchiwa**, and C. Aranas Jr, Effect of Vanadium on the Mechanical and Microstructural Properties of Medium-Mn Steels, TMS Annual Meeting & Exhibition (2024), 1412–1417.
- **K. Nyamuchiwa**, Y. Tian, K. Chadha, L. Jiang, T. Dorin, and C. Aranas, Precipitation Behaviour at the Interface of an Additively Manufactured M789–N709 Hybrid Alloy, Acta Metall. Sin. Engl. Lett. 36(8) (2023) 1353-1370.
- **K. Nyamuchiwa**, R. Palad, J. Panlican, Y. Tian, and C. Aranas Jr, Recent Progress in Hybrid Additive Manufacturing of Metallic Materials, Appl. Sci. 13, (2023) 8383.
- **K. Nyamuchiwa**, Y. Tian, K. Chadha, and C. Aranas, Laser Powder Bed Fusion of M789 Steel on Wrought N709 Steel Substrate, in Proceedings of the 61st Conference of Metallurgists (2023), 9–14.
- C. Aranas Jr, **K. Nyamuchiwa**, K. Chadha, J. Pasco, and T. McCarthy, Thermomechanical Response of an Additively Manufactured Hybrid Alloy by Means of Powder Bed Fusion, Key Eng. Mater. 964, (2023) 117.

- Y. Tian, **K. Nyamuchiwa**, K. Chadha, Y. He, and C. Aranas, Laser Powder Bed Fusion of M789 Maraging Steel on Cr–Mo N709 Steel: Microstructure, Texture, and Mechanical Properties, Mater. Sci. Eng. A 839, (2022) 142827.
- K. Chadha, Y. Tian, **K. Nyamuchiwa**, J. Spray, and C. Aranas, Austenite Transformation during Deformation of Additively Manufactured H13 Tool Steel, Mater. Today Commun. 33, (2022) 104332.
- **K. Nyamuchiwa**, Y. Tian, K. Chadha, Y. L. He, and C. Aranas Jr, Interfacial Properties of Additively Manufactured M789 Steel on Wrought N709 Alloy, Defect and Diffusion Forum 421 (2022), 15–19.
- **K. Nyamuchiwa**, Z. Lei, and C. Aranas, Cybersecurity Vulnerabilities in Off-Site Construction, Appl. Sci. 12, (2022) 5037.
- **K. Nyamuchiwa**, M. A. H. Gepreel, A. S. Hamada, and K. Nakamura, Microstructure and Mechanical Properties Change with Cold Deformation of the Biomedical Ti-17Nb-6Ta-3Zr Alloy, Key Eng. Mater. 780, (2018) 15.

Conference Presentations

- Kudakwashe Nyamuchiwa, ASEAN Conference on Additive Manufacturing, (ACAM 2021), Interfacial Properties of Additively Manufactured M789 Steel on Wrought N709 Alloy, October 28-29, 2021, Online
- Kudakwashe Nyamuchiwa, Holistic Innovation in Additive Manufacturing in Additive Manufacturing, (NSERC HI-AM), Laser Powder Bed Fusion of M789 Steel on Wrought N709 Steel Substrate, June 21-22, 2022, Montreal, Quebec, Canada

- Kudakwashe Nyamuchiwa, Conference of Metallurgist (COM 2022), Laser Powder Bed Fusion of M789 Substrate on Wrought Steel Substrate, August 21-24, 2022, Montreal, Quebec, Canada
- Kudakwashe Nyamuchiwa, TMS 2024, Effects of Friction and Deformation Heating on Additively Manufactured M789 Steel During Hot Compression Tests, March 3-7, 2024, Orlando, Florida, USA
- Kudakwashe Nyamuchiwa, TMS 2024, Effects of Vanadium on the Mechanical and Microstructural Properties of Medium-Mn Steels, March 3-7, 2024, Orlando, Florida, USA