

**HYBRID ADDITIVELY MANUFACTURED TOOL STEEL BIMETALS
PRODUCED USING LASER POWDER BED FUSION**

By

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Abstract

This study attempts to broaden our knowledge on the fabrication of tool steel bimetal using powder-based additive manufacturing techniques. It mainly involves the fabrication of bimetal for tool and dies applications in the automotive industry along with broad microstructural and mechanical characterizations, and heat treatment procedures. Two conventionally made alloys, including hot work tool steel H13 and cold work tool steel P20, were selected as the conventionally made substrates. Maraging steel (MS1) powder was used as the feedstock material and deposited on the substrates via the laser powder bed fusion (LPBF) technique. Two bimetallic systems were fabricated and designated as MS1-H13 and MS1-P20 hybrid steels in the as-built condition.

Comprehensive investigations were conducted on characterizing the morphological and microstructural aspects of these two hybrid steels as well as their mechanical properties. Scanning electron microscope (SEM), X-ray diffraction (XRD), energy dispersive spectrometer (EDS), electron probe microanalysis (EPMA), electron backscatter diffraction (EBSD), focus ion milling (FIB), and transmission electron microscope (TEM) were utilized as advanced diagnosis techniques to identify the features of hybrid steels at different microscopic scales. Various mechanical tests were conducted at either micro- or macro-scales to determine the mechanical performance of the hybrid steels in service conditions. Microhardness tests, uniaxial tensile measurements, fractography, digital image correlation (DIC) along with various bending tests were carried out for this purpose.

Outstanding results were achieved for both hybrid steels. The PBF technique led to the production of a defect-free interface between the substrates and deposited MS1 powder. Cracks, delamination, and porosities were not observed, and a very sound joint was built

between both sides of the hybrid steels. The additive side of hybrid steels showed finer morphology, higher hardness, and superior strength in comparison with the substrate side. Both hybrid steels exhibited high mechanical strength and did not fail at the interface, making the hybrid steels suitable for practical use. However, heterogeneity was found in the microstructural and mechanical properties of the as-built hybrid steels since two dissimilar alloys were involved.

The heterogeneity across the interface area might impair the efficiency of hybrid steels in working conditions. Therefore, I tried to mitigate this effect on the additively manufactured hybrid steels via post-processing heat treatment. Various heat treatment cycles were designed, performed, and evaluated on the as-built samples. For MS1-H13, a homogenized martensitic microstructure was generated across the interface using a solution treatment. For MS1-P20, a heat treatment sequence including hardening with subsequent aging was found to be effective in this regard. The mechanical properties were greatly enhanced for both hybrid steels in terms of hardness, yield strength, and fracture strength.

Dedication

This thesis is dedicated to my lovely parents, my brother, and sister.

For their endless love, support, and encouragement

Appreciation

I deeply express my sincere to my supervisor Dr. Mohsen Mohammadi, who provided me with this great opportunity to follow my academic career as a Ph.D. student. My Ph.D. journey would not have been possible without his supervision. He always motivates me to solve my research problems and supports me in my personal life with his great source of energy.

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Nomenclature

AM: Additive manufacturing

BJT: Binder jetting

MEX: Material extrusion

MJT: Material jetting

SHL: Sheet lamination

VPP: Vat photopolymerization

PBF: Powder bed fusion

DED: Direct energy deposition

LPBF: Laser powder bed fusion

CAD: Computer-aided design

EBM: Electron beam melting

DED: Directed energy deposition

WAAM: Wire Arc Additive Manufacturing

SLM: Selective laser melting

DMLS: Direct metal laser sintering

SLS: Selective laser sintering

SLRS: Selective laser reactive sintering

LMD: Laser metal deposition

DMD: Direct metal deposition

LENS: Laser engineered net shaping

EBAM: Electron beam additive manufacturing

SEM: Scanning Electron Microscope

XRD: X-ray Diffraction

EBSD: Electron Backscattered Diffraction

TEM: Transmission Electron Microscope

EPMA: Electron probe microanalysis

EDS: Energy dispersive X-ray spectroscopy

IPF: Inverse Pole Figure

EDS: Energy-dispersive X-ray spectroscopy

KAM: Kernel average misorientation

EDM: Electrical discharge machining

GB: Grain boundary

FIB: Focus ion beam milling

HAGBs: High angle grain boundaries

LAGBs: Low angle grain boundaries

BD: Building Direction

TZ: Transition zone

PMZ: Partially melted zone

G: temperature gradients (K/m)

R: solidification rate (m/s)

HAZ: Heat affected zone

TMAZ: Thermo-mechanically affected zone

DAZ: Diffusion affected zone

VLABs: Very low angle boundaries

HAM: Hybrid additive manufacturing

IAMA: Internal average misorientation angle

PAG: Parent austenite grains

UTS: Ultimate tensile strength

DIC: Digital image correlation

MAM: Metal additive manufacturing

XEDS: X-ray energy dispersive spectroscopy

LM: Low magnification

AB: As-built

HT: hardening treated

HAT: Hardening and aging treated

GOS: Grain orientation spread

3PB: Three-point bending

4PB: Four-point bending

1 Introduction

The modern era of technology undoubtedly has increased the demand for new engineering applications, which can bring about new problems and issues. Therefore, engineers have come up with advanced materials and processing technologies to respond to these challenges effectively. Accordingly, advanced materials involve incredibly complex structures and properties. These complexities usually cannot be met by conventional manufacturing, and new techniques should be invented. Thanks to creative engineers and scientists, a revolutionizing technique was invented, which transcended the limitations of conventional manufacturing and introduced outstanding capabilities. It is well known as additive manufacturing (AM) and can produce extremely versatile geometry and material.

Beyond its unique features, additive manufacturing (AM) demonstrates compatibility with conventional manufacturing. This enables engineers to produce parts with a hybrid strategy. An obvious example is bimetallic systems, in which a component is made using both AM and conventional methods. Such components constitute advantages of both methods, including the large-scale production of conventional manufacturing and the high geometrical complexity of additive manufacturing. In addition, AM has allowed more freedom in combining materials of various kinds and producing versatile dissimilar components with a defect-free interface.

As an illustration, maraging steels can be additively deposited on many alloys to produce bimetals. Maraging steel is well adapted to AM techniques, and many types of research has been conducted on this material. It exhibits high density, fine microstructure,

and superior mechanical properties in AM. Therefore, it can be a possible candidate for AM bimetals, and exceptional functions might emerge from bimetals in which maraging steel is used.

1.1 Literature Review

1.1.1 Additive Manufacturing (AM)

According to ISO/ASTM 52900, the term “additive manufacturing” refers to the manufacturing processes in which a three-dimensional geometry is created by joining materials [1]. This is a general term and involves many applications and materials. The invention of additive manufacturing (AM) dates back to 1987 when thin layers of a UV-sensitive polymer were solidified using a laser for 3D stereolithography [2]. In the 1990s, AM technology was developed for metals based on sintering techniques. AM processes were categorized into seven main types [3]:

- Binder jetting (BJT)
- Material extrusion (MEX)
- Material jetting (MJT)
- Sheet lamination (SHL)
- Vat photopolymerization (VPP)
- Powder bed fusion (PBF)
- Direct energy deposition (DED)

The latter two techniques mainly form the metal additive manufacturing (MAM) processes [4]. A computer-aided design (CAD) is used as the 3D model in both PBF and DED; however, they follow different strategies to build a 3D object from the CAD model.

In PBF, the feedstock material is always in the form of powder, and the heat source can be a laser or an electron beam, depending on the type of material to be printed. The feedstock material in DED, however, is more versatile since either a powder or wire feedstock can be used. In terms of heat sources, laser, electron beam, gas metal arc, and plasma arc can be used to fuse the feedstock material in DED [5].

The PBF strategy involves thin layer deposition of feedstock powder within the range of 30-150 μm in thickness, and then the laser or electron beam impingement selectively melts certain areas of the powder layer. By moving the laser beam, the melted powder particles solidify and form a single layer of the whole 3D CAD model [6]. This procedure repeatedly continues until the final part is made layer-by-layer, as shown in Figure 1-1. Three main factors distinguish various PBF formats, including the heat source, feedstock material, and melting intensity. Table 1-1 shows the main categories of PBF based on these factors.

Table 1-1. Powder Bed Fusion (PBF) processes [6].

Process	Heat source	Feedstock material	Melting intensity
Selective laser melting (SLM)	Laser	Metal powder	Full
Direct metal laser sintering (DMLS)	Laser	Metal powder	Partial
Selective laser sintering (SLS)	Laser	Polymer powder	Partial
Selective laser reactive sintering (SLRS)	Laser	Si, ZrO ₂ , Cr, Cr ₂ O ₃	Partial
Electron beam melting (EBM)	Electron beam	Metal powder	Full

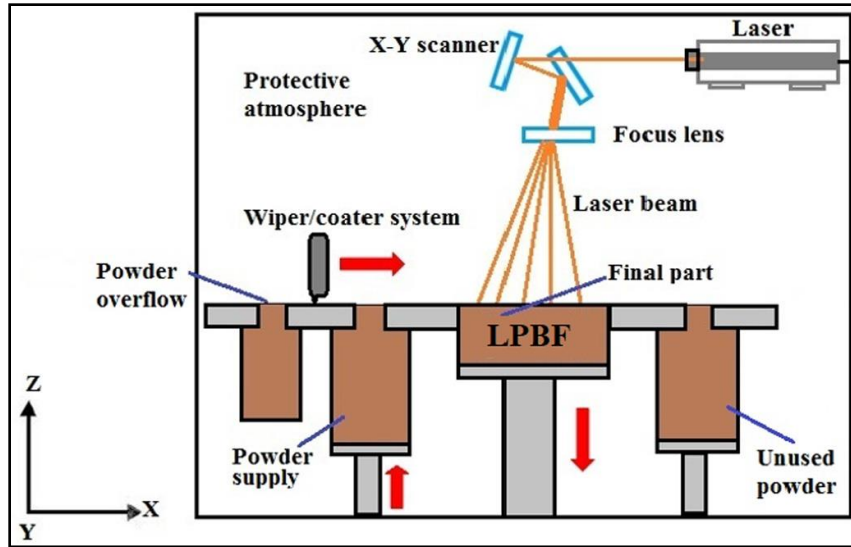


Figure 1-1: Illustration of powder bed fusion (PBF) technique [7].

Unlike the powder-bed basis of PBF, the DED technique utilizes a feeding strategy in which a powder or wire metal is fed from a nozzle, then a heat source melts the feedstock and causes the formation of a melt pool on a substrate (see Figure 1-2). Subsequent solidification occurs when the heat source moves away, and a solid bead is deposited as an increment of the final part. The repetition of this sequence finally forms the intended part. Herein, various heat sources and feedstock materials generate different kinds of DED processes. Table 1-2 shows the different DED techniques classified by these two factors [8].

Table 1-2. Direct energy deposition (DED) processes.

Process	Heat source	Feedstock material
Laser metal deposition (LMD)	Laser	Powder
Direct metal deposition (DMD)	Laser	Powder
Laser engineered net shaping (LENS)	Laser	Powder
Electron beam additive manufacturing (EBAM)	Electron beam	Powder or Wire
Wire arc additive manufacturing (WAAM)	Arc or Plasma	Wire

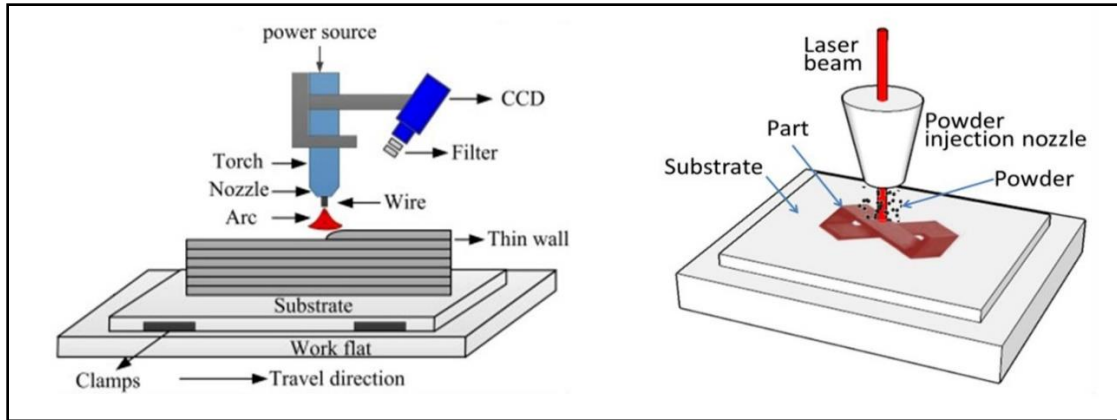


Figure 1-2: Schematic illustration of direct energy deposition (DED) technique [5].

1.1.2 AM Bimetals

A bimetal can be defined as the combination of two materials that are different in terms of chemical composition, microstructure, and properties. In other words, a bimetal is a dissimilar weldment, which provides the beneficial properties of both materials. As a prerequisite, a bimetal shall involve a metallurgical bond, so the mechanical fastening or chemical bonding cannot be considered as bimetals. Accordingly, bimetallic structures were typically produced using welding or brazing techniques for many years. A typical example is the aluminum-stainless steel bimetal, which offers a good corrosion resistance, high thermal conductivity, and excellent mechanical properties in cryogenic environments [9]. Many applications were reported for this bimetal, including couplings in pipelines, connectors in liquified gas storage vessels, and large chemical containers [9,10].

Due to the limitations of conventional manufacturing techniques, additive manufacturing has recently been considered an effective alternative for the fabrication of bimetals. Interestingly, most metal additive manufacturing techniques such as PBF and DED are suitable for this purpose. AM bimetals can be either manufactured fully or partially

via AM techniques. The latter is a hybrid approach, where one material is conventionally made, but the other material is additively manufactured. In this study, we used this approach to produce the tool steel bimetal. Although additive manufacturing of bimetal is relatively new, it has shown incredible potential for industrial applications. Figure 1-3 shows two applications of AM bimetal used in aerospace engineering. Figure 1-3(a) is a turbine blade that was fabricated by depositing TiAl on a Ti6Al4V turbine disk using the DED technique [11]. Figure 3(b) also shows NASA's recent approach to fabricating a bimetallic channel wall nozzle using the DED technique [12].

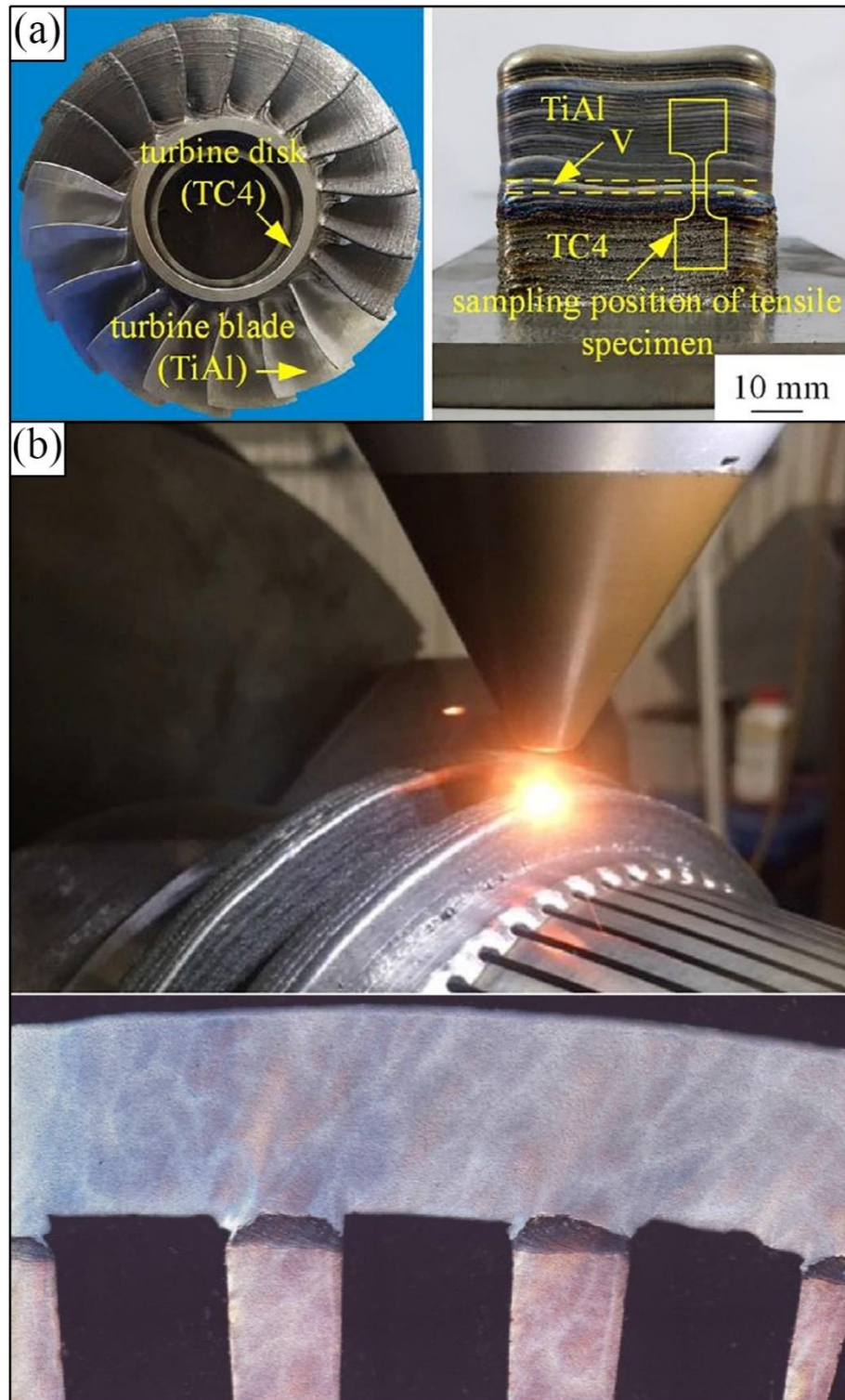


Figure 1-3: AM bimetal components in aerospace industry a) TiAl-Ti6Al4V turbine blade [11] b) NASA's channel wall nozzle [12].

1.1.3 Processing Techniques for AM Bimetals

Bimetallic structures are commonly fabricated using two main AM techniques, including the PBF and DED methods. Herein, a conventionally-made alloy is used as the build plate inside the printing machine, and powder or wire feedstock materials are deposited on top of that, forming a bimetallic structure.

1.1.3.1 Direct Energy Deposition (DED) Technique

Most DED processes are suitable for producing bimetals. WAAM, LENS, DMD, and LMD are frequently used in this regard [13–16]. Each of these processes contains certain capabilities and limitations. For instance, large-scale components up to 1 m can be manufactured using the WAAM process [13]. This process has the potential for large industrial bimetals. On the other hand, the powder-based DED processes contain smaller chambers, and small size bimetals can be made. The merit of these processes is the ability to achieve finer surface roughness and more geometrical complexity. In addition, the powder-based DED machines can be equipped with several feeders, and each feeder can deliver the powder feedstock independently at different rates. Bimetallic components can be easily manufactured using multiple feeder systems [17].

Figure 1-4 illustrates the DED procedure for manufacturing bimetals. In DED, the feedstock can be either a powder or wire material. The powder feedstock is usually carried using an inert gas from the powder container to the focal point of the laser, where the powder particles are melted and then form a melt pool on the substrate [16]. The wire feedstock is delivered via a torch to the molten area, so no gas is needed for the delivery purpose. Once melting along with subsequent solidification is achieved, a computer numerical control system (CNC) moves the build plate in the X and Y direction in each

pass, and once the whole layer is completed, it moves the build plate downward in the Z direction [13,16]. The feedstock delivery, melt pool formation, solidification, and stage movement are repeated sequentially until the final bimetal is manufactured.

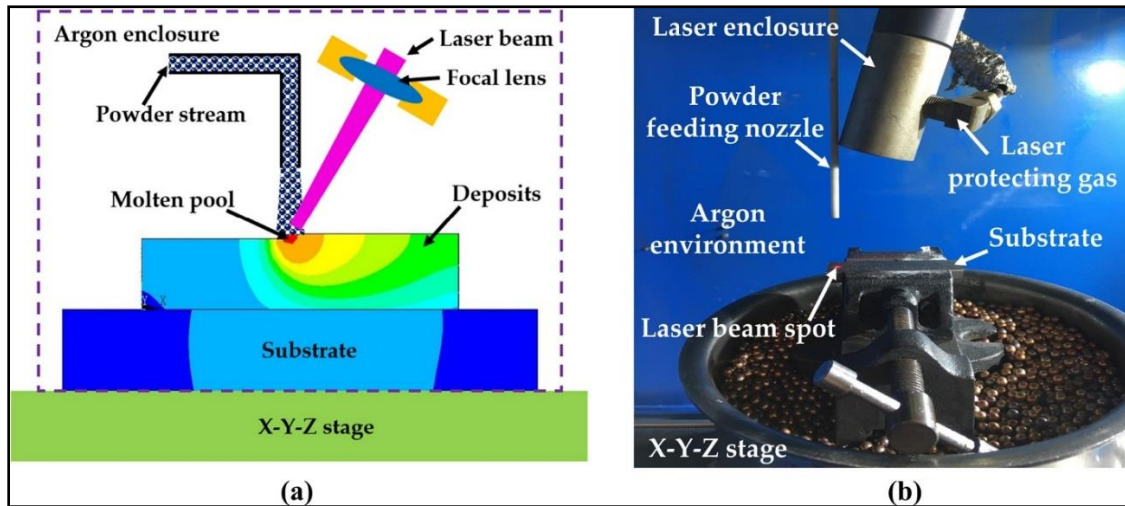


Figure 1-4: a) Schematic illustration of DED technique b) equipment for bimetal fabrication [16]

1.1.3.2 Powder Bed Fusion (PBF) Technique

In PBF, a conventional substrate is preplaced inside the printer as the build plate, and a feedstock powder is deposited layer-by-layer on the build plate. The most common PBF processes for producing bimetals are SLM, DMLS, and EBM. Like the DED method, a melt pool is formed because of laser impingement with the powder feedstock and substrate, and the consolidation of melt pools develops a single layer of the final part. This procedure iterates hundreds or thousands of times to finally generate the bimetal parts. Figure 1-5 demonstrates how a bimetal is manufactured using the PBF technique [18]. As indicated in this figure, several important parameters are involved in manufacturing a bimetal, including the power source, scanning speed, layer thickness, hatch spacing, and inert gas. The process parameters should be perfectly controlled to avoid manufacturing defects and ensure the soundness of the bimetal. These parameters generate several

phenomena affecting the quality of bimetals. Since we used the PBF technique in our work, the physical phenomena occurring during this process will be discussed in the next section.

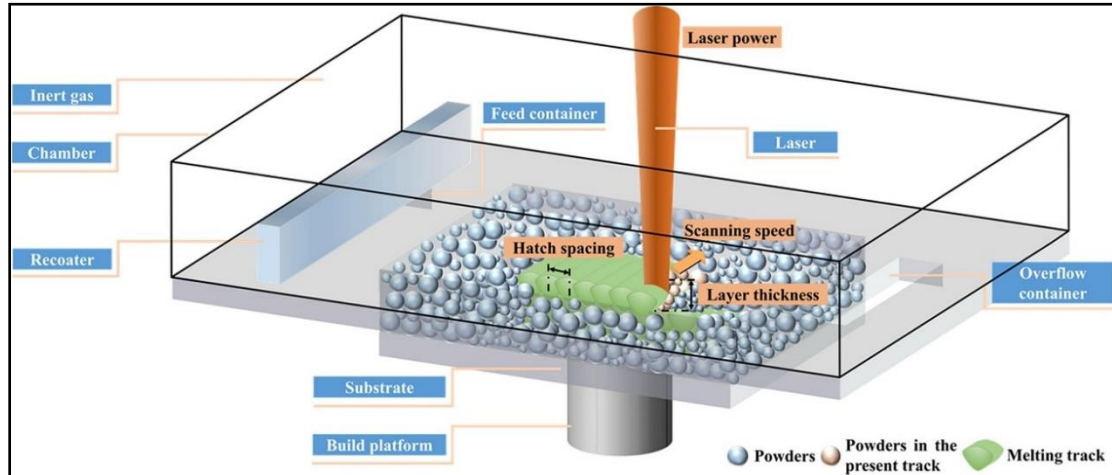


Figure 1-5: Schematic of powder bed fusion (PBF) technique for bimetal fabrication [18].

1.1.4 Physical Phenomena in AM Bimetal

The success of manufacturing bimetals depends highly on the effects caused by the process parameters. Figure 1-6 represents a comprehensive schematic of different phenomena during the PBF process [19]. The first phenomenon is heating, which is generated by a laser or electron beam source. Simply, the heat energy is either absorbed by the powder, causing melt pool formation, or dissipated, causing heat loss. The power source transfers the heat energy to the powder layer by irradiation, and the powder particles absorb the heat energy and reflect a fraction of heat energy to the underneath particles [19]. The heat reflection causes heat penetration to other powder particles. However, heat loss is inevitable during the process, mainly due to radiation, convection, and evaporation of light alloying elements. The convection-induced heat loss is insignificant in EBM because of the vacuum chamber, while convection exists between the powder particles and the inert gas in SLM or DMLS. The heat conductivity can also add to the heat loss, which relies on

the thermal conductivity and sintering grade of the feedstock powder. Preheating the powder layer can mitigate the heat loss related to thermal conduction [19].

Upon the heat absorption, a molten area called a "melt pool" is formed. The behavior of the melt pool significantly influences the solidified layer, microstructure, and hence the mechanical properties of the final part. In this regard, several forces affect the melt pool, including surface tension, buoyancy, capillarity, Marangoni, evaporation pressure, and gravity [19]. The gravitational force has a negligible effect on the melt pool and can be neglected. The flow of the melt pool is mostly affected by Marangoni and buoyancy forces, where the latter seems insignificant [5]. Hence the Marangoni convection determines the flow of the melt pool, and it is directly proportional to the surface tension [20]. As mentioned earlier, some alloying elements might evaporate and form a gas inside the melt pool, which can produce pressures to the melt pool. This pressure can form a keyhole and a vapor capillary [19]. Once the power source is passed, the resultant cooling promotes rapid solidification of molten areas; however, some residual stresses and distortion are developed [21,22]. Since the PBF process is iterative, each layer experiences cyclic heating and cooling; thus, the deposited layers are being heat treated. This effect can partially remove the residual stresses and decrease distortion in the final part.

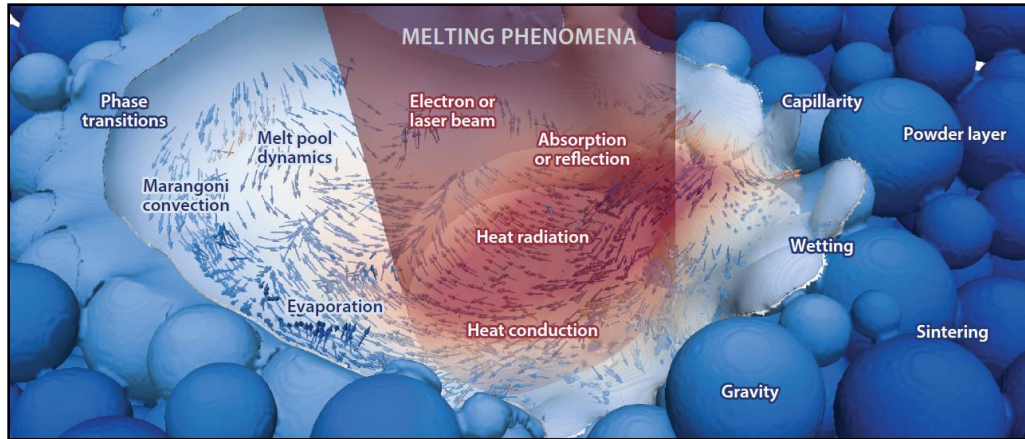


Figure 1-6: Schematic of physical phenomena in additive manufacturing of bimetal [19].

1.1.5 AM Bimetal Classification

As mentioned earlier, two main techniques (i.e., DED, PBF) proved applicable to manufacturing bimetals. The other contributing factor is the alloying systems being used to produce an AM bimetal. The alloying systems mean a combination of two materials to be bonded using additive manufacturing. Simply, we designated them as material A and material B in this section. The diversity of alloying systems is normally involved with remarkable dissimilarity and might result in incompatibility and discontinuity in many cases. Depending on the mismatch degree between the two materials, the following approaches were developed:

1.1.5.1 Directly Bonded Bimetal

Although many engineering alloys are dissimilar in terms of composition and microstructure, they can be joined and combined to form a bimetallic component. These materials feature metallurgical compatibility, which means a rich solid solution is formed because of bonding two different materials. Due to simplicity and ease of manufacturing, this approach has been extensively used in the additive manufacturing of bimetals.

Aluminum, titanium, copper, nickel, and steels are the most applicable alloys for AM direct bonding. Table 1-3 summarizes the bimetals fabricated directly via additive manufacturing.

The direct bonding approach seems a reliable technique for many alloy systems since high mechanical properties were achieved in many cases. For instance, Hadadzadeh [23], Bhaduri [24], Wang [25], and Tan [26] conducted the uniaxial tensile loading test and proved that the interfacial region was strong so that fracture did not occur at the interface. Figure 1-7 shows their results in which the fracture location is far from the interface between materials A and B. This is a significant achievement indicating additive manufacturing of bimetals is successful in terms of mechanical strength and can be practically considered as an alternative to conventional dissimilar welding techniques.

Table 1-3. Directly bonded AM bimetals

Alloying System		AM Technique	Reference
Material A	Material B		
Al cast alloy	AlSi10Mg	SLM	Hadadzadeh et al. [23]
AA6082-T6	AlSi10Mg	SLM	Bhaduri et al. [24]
Al-12Si	Al-3.5Cu 1.5Mg-1Si	SLM	Wang et al. [25]
Ti6Al4V	TiAl	EBM	Zhai et al. [27]
CP Ti	Ni	DED	Afrouzian et al. [28]
Inconel 625	SS 316L	DED	Chen et al. [29]
Nickel aluminum bronze	SS 316L	WAAM	Chalasani et al. [30]
Copper	Maraging steel	SLM	Tan et al. [26]
Inconel 718	SS 316L	EBM	Hinojos et al. [31]
H13	Maraging steel	DMLS	Cyr et al. [32]

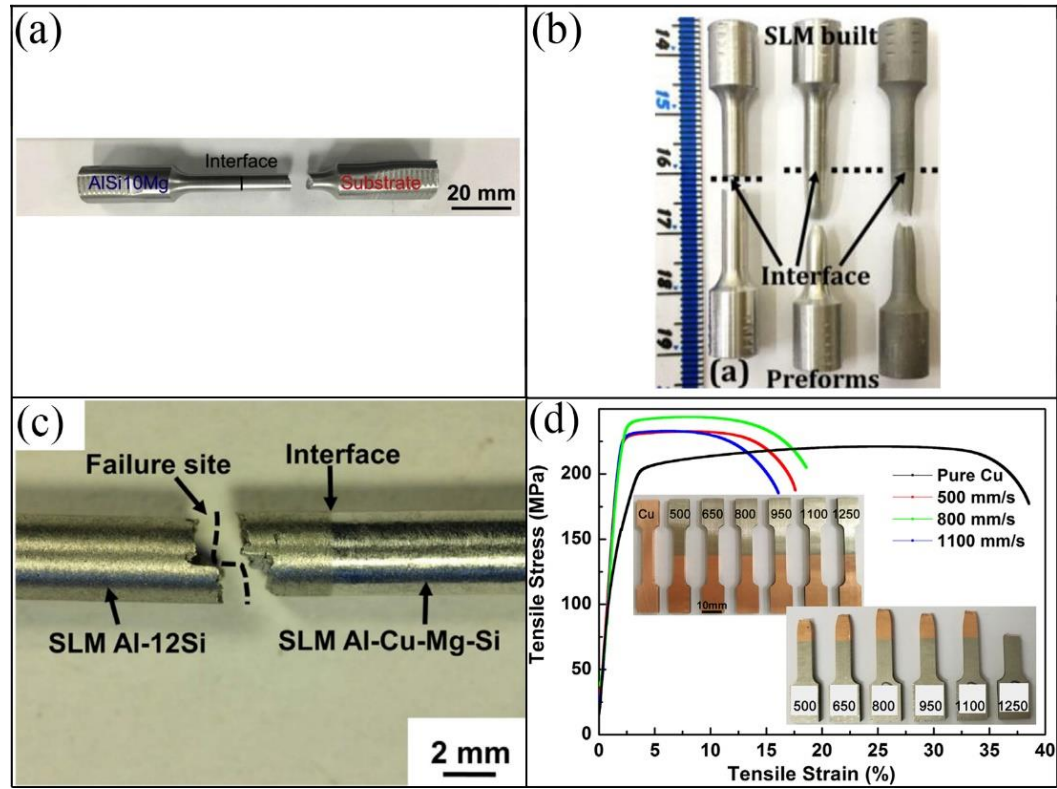


Figure 1-7: uniaxial loading of bimetals designated in table 3: a) Al cast alloy-AlSi10Mg [23] b) AA6082-AlSi10Mg [24] c) Al12Si- Al3.5Cu1.5Mg1Si [25] d) Copper-Maraging steel [26]

In terms of microstructure, the directly bonded bimetals exhibited good cohesion at the interface; however, they feature a considerable variation in grain morphology from material A to material B. This morphological gradient normally appears as a large-to-fine grain size transition at the interface, which can develop microstructural heterogeneity within the fabricated bimetals. Figure 1-8 represents the IPF maps between the two sides in the directly bonded AM bimetals. The interface generally consists of epitaxial growth of grains on the coarse grains of the substrate. The deposited material (i.e., top side) normally contains ultrafine grains deviated towards the building direction; however, coarser grains were solidified when the WAAM process is used (see Figure 1-8(b)).

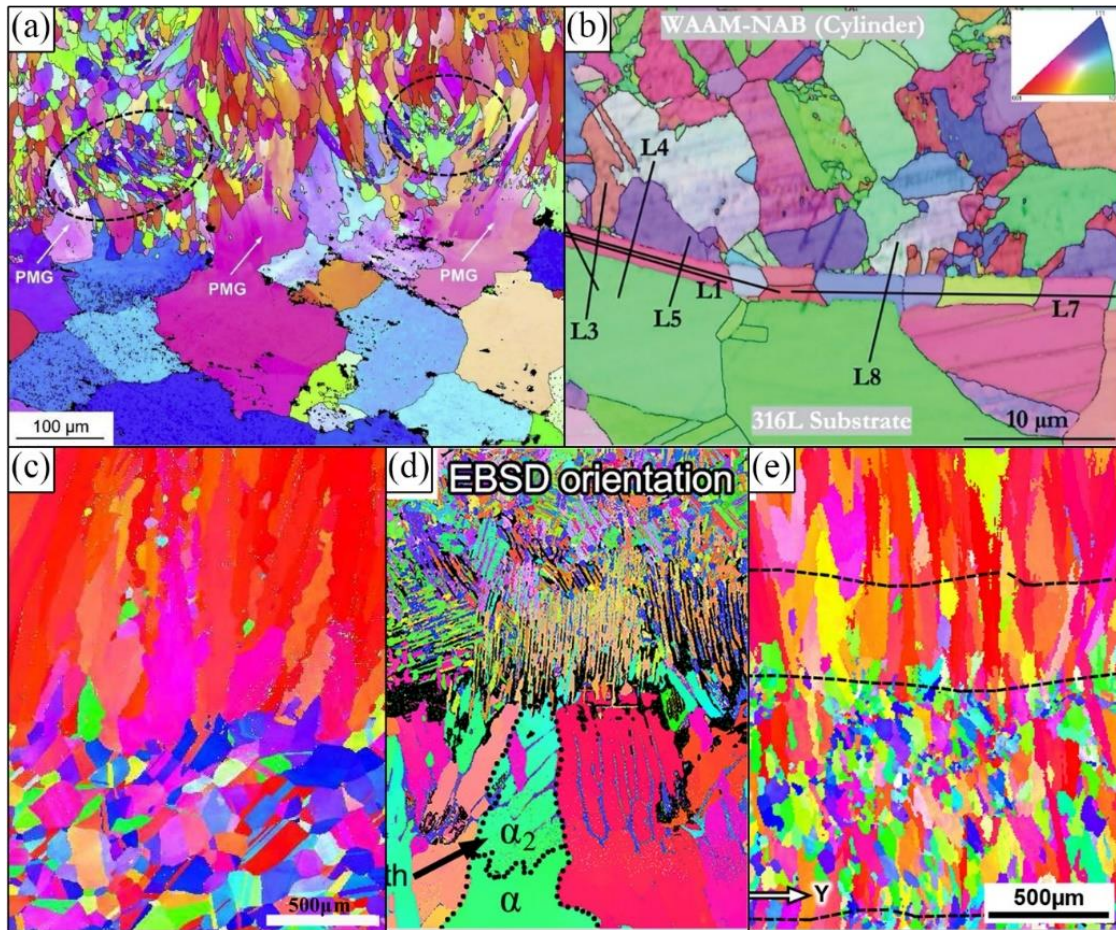


Figure 1-8: IPF mapping across the interface: a) Al cast alloy-AlSi10Mg [23] b) NiAl bronze-SS316L [30] c) Inconel 718-SS316L [31] d) Ti6Al4V-TiAl [27] e) Inconel 625-SS316L [29]

1.1.5.2 Interlayer-assisted bimetal

The chemical composition difference between two alloying systems might result in an undesirable reaction between the two materials forming brittle compounds such as intermetallics at the interface. In addition, the thermal properties can sometimes be considerably different between the two materials, so direct bonding causes cracking and delamination of the interface [33–35]. Thus, such alloying systems cannot be bonded via the direct bonding approach. In this case, an intermediate material is used to prevent the formation of detrimental phases at the interface. This interlayer resembles the filler material in the welding and brazing techniques and should be compatible with both

materials intended to be bonded. Herein, the level of dissimilarity is higher between the two materials as compared to the direct bonding approach. Table 1-4 refers to the typical AM bimetals fabricated using an interlayer. The degree of dissimilarity determines whether one or multiple interlayers should be utilized in this approach. For instance, Zr MG-SS 304 bimetal was manufactured using multiple layers of V, Ti, and Zr (see Figure 1-9) [33].

Table 1-4. AM bimetals fabricated using interlayer

Alloying System				AM Technique	Reference
Number	Material A	Interlayer	Material B		
1	Zr Metallic glass (MG)	V/Ti/Zr	SS 304	Foil-based AM	Li et al. [33]
2	Ti6Al4V	Nb	SS 410	LENS	Onuike et al. [34]
3	Inconel 718	Compositional Layer	GRCop-84	LENS	Onuike et al. [35]
4	Inconel 718	Copper	SS 316L	DED	Zhang et al. [36]
5	Inconel 718	Compositional Layer	Ti6Al4V	LENS	Onuike et al. [37]
6	Ti6AL4V	V/Cr/Fe	SS 316L	LMD	Li et al. [38]
7	HSLA steel	Compositional Layer	Copper-Aluminum	WAAM	Rodrigues et al. [39]

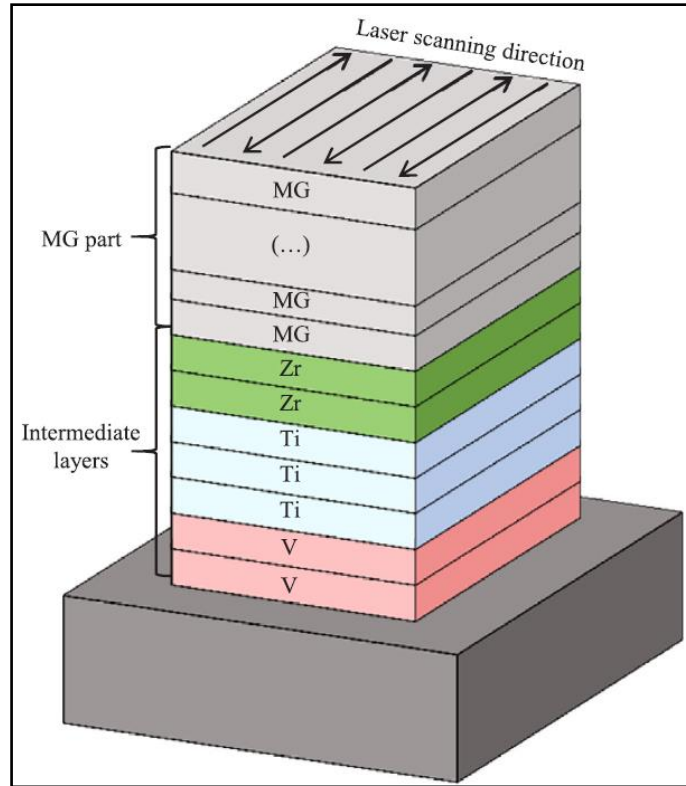


Figure 1-9: Illustration of interlayers in Zr MG-SS 304 bimetal [33].

The interlayer plays a key role in creating a cohesive bond between the two dissimilar materials. It can create a barrier against the diffusion phenomenon, so the alloying elements of two materials can no longer penetrate and react with each other to form brittle intermetallic compounds. Indeed, the interlayer acts as a matrix, where dilution occurs, and the alloying elements dissolve instead of segregating as secondary phases. The other function of the interlayer is that the gradient of chemical composition occurs from material A to material B leading to a smooth transition of thermal properties as well as mechanical properties across the interface; thus, the fabricated bimetal contains lower residual stresses [37,38]. The gradient of thermal and mechanical is shown in Figure 1-10 for the steel-CuAl bimetal [40].

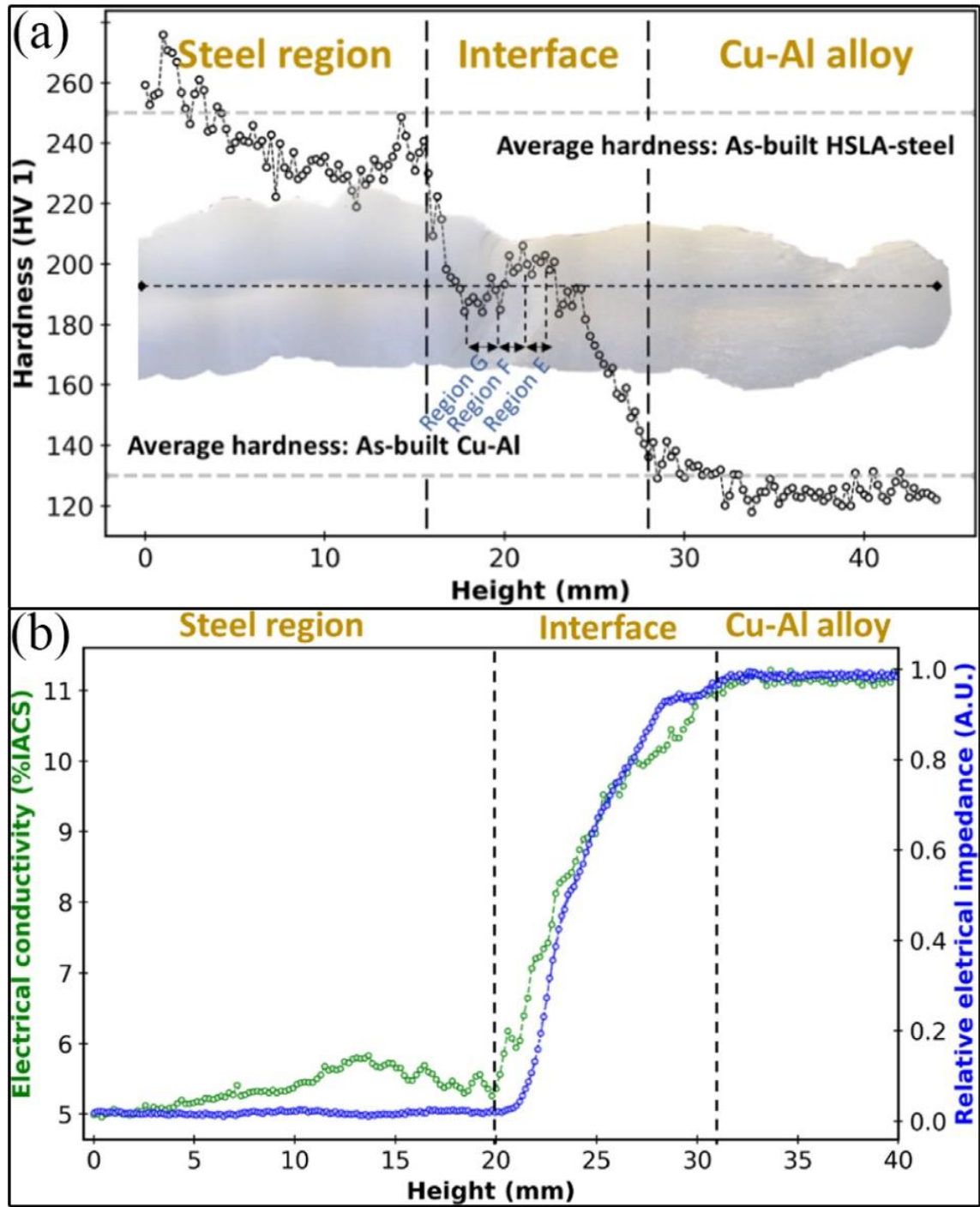


Figure 1-10: a) microhardness variation b) electrical conductivity across the steel-CuAl interface [40].

1.1.6 Bimetals with Maraging Steel Bases

Maraging steels are advanced ferrous alloys with superior mechanical properties. Maraging steels can simultaneously exhibit high strength, ductility, and toughness in working conditions [41–43]. They also have proper weldability and can be joined to both ferrous and nonferrous alloys. The fabrication of maraging steels requires fast cooling conditions to ensure martensite transformation. This factor has made these materials highly printable since rapid cooling is the main feature in additive manufacturing [44]. These features motivated many researchers to use maraging steels in the fabrication of AM bimetals.

Table 1-5 shows the recent works on the maraging steels used in AM bimetals. Maraging steels exhibited good compatibility with tool steels, stainless steels, and many other grades of steel. It is also compatible with other alloys, including copper alloys [26,45]. The common AM process used to fabricate such bimetals is the selective laser melting (SLM) technique. Figure 1-11 summarizes the general microstructural and mechanical aspects of AM bimetals with maraging steels. The maraging steels bimetals normally feature a sharp interface between the substrate and maraging steel and an interfacial region with gradient composition towards the maraging steel side, as shown in Figure 1-11 (a). At the interface, the epitaxial growth of maraging steel usually occurs towards the building direction, and very fine grains are developed on the substrate (see Figure 1-11 (b)). In terms of mechanical properties, higher hardness and tensile strengths can be achieved for the maraging steels due to the martensitic transformation, and the bimetals never failed at the interface (see Figure 1-11 (c)-(d)). These results can confirm that successful bimetals can be produced using additively manufactured maraging steel.

Table 1-5. AM bimetal fabricated using maraging steel

Alloying System			AM Technique	Reference
Year	Substrate	Maraging Steel		
2018	H13	MS1	SLM	Cyr et al. [32]
	T2 Copper	MS1	SLM	Tan et al. [26]
2019	H13	MS1	SLM	Azizi et al. [46]
	CuSn	18Ni300	SLM	Zhang et al. [45]
2020	CMnAlNb Steel	18Ni300	SLM	Kucerova et al. [47]
2021	CrMn	MS1	SLM	Bai et al. [48]
	420 Stainless Steel	Corrax	SLM	Samei et al. [49]
2022	N709 Steel	M789	SLM	Tian et al. [50]
	410 Stainless Steel	MS1	SLM	Zhang et al. [51]

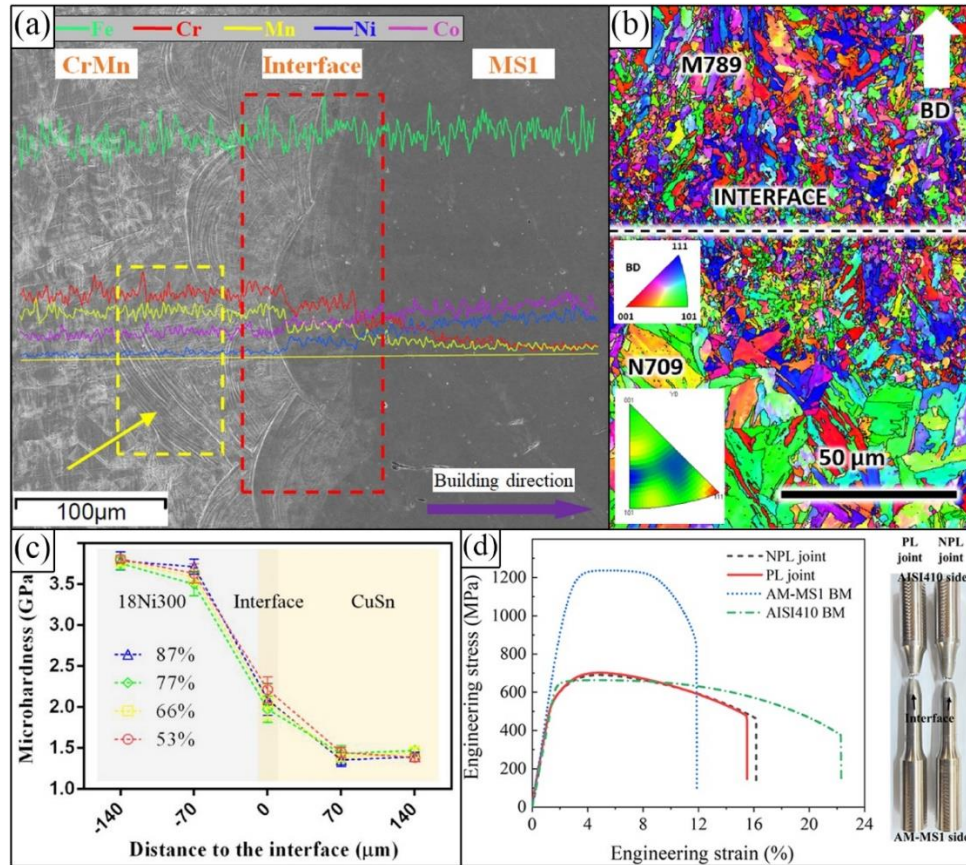


Figure 1-11: a) Compositional gradient of CrMn-MS1 [48] b) grain morphology of N709 Steel-M789 [50] c) hardness variation of CuSn-18Ni300 [45] d) tensile properties of SS410-MS1 [51].

1.2 Gap of Knowledge

Additive manufacturing is still a new method with new opportunities and challenges compared to conventional and traditional manufacturing. The literature review also proved that additive manufacturing of bimetals has just been introduced in the last five years, and only a few relevant studies can be found in this field. In addition, the diversity of alloying systems applicable to AM bimetals can be enormous, and each alloying system requires independent and comprehensive analysis. Therefore, the characterization of AM bimetals is a key component to better understand the advantages of additive manufacturing. Since AM bimetals usually involve complex morphological features, advanced characterization techniques including EBSD, FIB, and TEM should be carried out and developed for these materials.

Furthermore, the as-build bimetals often suffer from major deficiencies regarding microstructural and mechanical properties. The interfacial area incorporates substantial differences from the substrate to the additive side. The substrate contains a soft phase with coarse grains, while the additive side is solidified as a hard phase with very fine grains. This inhomogeneity can result in steep hardness variations across the interface and subsequently weaker mechanical behavior, restricting the applications of AM bimetals in working conditions. As a result, new approaches should be studied to remove the anisotropic properties of AM bimetals in the as-built condition and enhance the mechanical properties. In this regard, post-processing techniques such as heat treatment would be a good candidate. However, most researchers have paid attention to heat treating the additively manufactured bulk materials, and the investigations on the heat treatment of AM bimetals have been overlooked. For this reason, we also aimed to investigate the

modification approaches of AM bimetals and figure out the optimum treatments in each case.

1.3 Study Outline

In this study, we aimed to study tool steel bimetals using the powder bed fusion (PBF) technique. Several targets were considered to better understand such bimetallic structures, including fabrication of successful tool steel bimetals, comprehensive characterization of the interface, modification processes, and evaluation of tool steel bimetals in complex loading conditions. This thesis was prepared in six chapters, where Chapter One is an introduction, and the remaining chapters incorporate our attempt at additive manufacturing of tool steel bimetals. This study involves two sets of bimetallic structures, including MS1-H13 and MS1-P20, to comprehensively study both hot work tool as well as cold work tool steel in AM bimetals.

We first investigated the fabrication of a defect-free AM bimetal using H13 hot work tool steel as the substrate, conducting initial characterization on the MS1-H13 bimetal in the as-built condition. This primary work was fully presented in Chapter Two and published as “Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique” in the journal of Additive Manufacturing. Our next attempt was to promote a more uniform microstructure across the MS1-H13 interface and increase the mechanical properties. Thus, we studied the effect of heat treatment on the MS1-H13 bimetal and managed to remove the interfacial contrast between MS1 and H13 and enhance the mechanical strength of this bimetallic structure. This covers Chapters Three and Four, where Chapter Three was published as “Interface engineering of additively manufactured maraging steel-H13 bimetallic structures” in the journal of Materials Characterization.

Chapter Three, entitled "Microstructure control of hybrid additively manufactured 18Ni300-H13 bimetals by sequential heat treatment" has been submitted to the journal of Materials Engineering and Performance.

We investigated the fabrication of the MS1-P20 bimetallic system. The P20 mold steel was used as the substrate, and the MS1-P20 bimetal was fabricated via the PBF technique. Chapter Five discusses the characterization of MS1-P20 bimetals in the as-built condition, which has been recently submitted as "Hybrid Manufacturing of MS1-P20 Tool Dies by Powder Bed Fusion" in the journal of Additive Manufacturing. Our last attempt in Chapter Six examines the heat treatment-based modification of MS1-P20 bimetal and evaluation of the mechanical behavior under bending conditions with more complex loading. This work entitled "On the Bending of MS1-P20 Hybrid Steels Additively Manufactured Using Laser Powder Bed Fusion" was our last submission in the journal of Materialia. Finally, our investigations were summarized in Chapter Seven, and the future works were presented in Chapter Eight.

1.4 Hypothesis

This study shows that bimetallic components can be additively manufactured for practical use in many industries. The tool steel bimetal fabricated using the maraging steel can be used as an approach to produce dies and tools with exceptional functionality. These bimetals involve perfect bonding with very high strength at the interface. The bimetal approach presented in this study can also enable engineers to produce parts with more complex geometries and accurate dimensions. For instance, an internal cooling channel can be perfectly created on the AM side of bimetallic dies to assist cooling in the service

condition. This study introduces heat treatment procedures to modify interfacial microstructure in tool steel bimetals. This can be a good reference for engineers to favorably manipulate the resultant performance of final components. Moreover, the characterization techniques and mechanical testing methods of this study can be considered as helpful and reliable guidelines for any research in mechanical engineering.

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2 Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique

2.1 Introduction

Additive manufacturing (AM) has brought about a new era in the manufacturing world. Since AM processes are fast, precise, and economically efficient often for custom-made parts in comparison with mass produced components, they have been considered in various industries [1] making them ideal for rapid fabrication in aerospace, automotive, energy, and biomedical applications [2]–[5]. In contrast to subtractive manufacturing methods (e.g. machining), metal additive manufacturing processes form three dimensional parts via layer-by-layer deposition of a melted feedstock, either in the form of powder or wire [6], [7]. AM features the direct production of complex and near-net shaped parts from the design without extra tooling, and minimal material waste. In addition, it can reduce the lead time and overall cost and increase flexibility in the range of alloys and materials that can be produced [1], [8]–[11]. Among the current additive manufacturing methods, the laser powder bed fusion (LPBF) family has received tremendous attention due to the resultant ultrafine microstructures and the subsequent superior mechanical properties [12]–[15].

Maraging steels belong to a high strength and ductile family of steels, which possess a relatively ductile martensitic matrix with a very low carbon content, and nanoscale intermetallic precipitates strengthening the material [16], [17]. Conventional maraging steels contain a high nickel content (around 18%), which ensures martensite formation during the decomposition of austenite. Other common alloying elements in these steels include cobalt and molybdenum. Maraging steels have been widely utilized in

industries such as aerospace, automotive, and tooling, where high strength, good damage tolerance, and more importantly high-temperature performance are necessary [18], [19]. Due to the importance of maraging steels as high strength structural materials on the one hand, and the advantages of AM techniques, on the other hand, it is imperative to adopt different AM processes to produce maraging steel parts.

Tan et al. [20] fabricated a fully dense grade 300 maraging steel with low surface roughness using LPBF. The optimization of different process parameters was studied by many researchers. For instance, Mutua et al. [21] investigated the effect of scan speed, laser power, overlap rate, and energy density on the microstructure and mechanical properties of LPBF-18Ni maraging steels with superb hardness and strength as compared to conventionally built ones. The as-built samples obtained hardness values of 330-403 HV, considerably higher than the conventionally fabricated maraging steels with 280 HV hardness [21]. They also found out that a solution treatment can manipulate the grain orientation and martensitic grain growth. In addition to the process parameter optimization, Bai et al. [22] investigated the effect of solution and aging treatments on LPBF-18Ni-300 maraging steel. Martensite to austenite transformation along with the disappearance of cellular structure resulted in a hardness reduction after the solutionizing heat treatment. However, the solutionizing heat treatment followed by an aging heat treatment had an inverse effect and increased the hardness. In this study, LPBF-18Ni-300 maraging steels with an ultimate tensile strength of over 2000 MPa were achieved using only aging heat treatment [22].

Several grades of steels such as H13 tool steels (high strength at high temperatures, with application in aluminum die-casting), and grade 420 stainless steels (high strength and

corrosion resistance, with application in plastic and rubber injection molding) are currently used in the tool and die industry as wrought alloys [14]. Additive manufacturing of tool steels has recently attracted the attention of researchers, where Fe85Cr4Mo8V2C1, 5CrNi4Mo, X65MoCrWV3-2, H11, and H13 were fabricated using LPBF techniques [23]–[30]. This is due to the extensive applications and associated challenges of tool steels in different industries. The fabrication of these steels using additive manufacturing is still challenging due to susceptibility to cracking and low wettability of these alloys [30]. Even the compositionally graded tool steel-maraging steel produced using laser metal deposition was not ideally favorable since various heat treatments should be applied [31]. The problem of very different heat treatment procedures for alloyed steels versus high carbon content tool steels is still an issue for depositing maraging steel powder on top of conventionally fabricated tool steels via powder bed fusion techniques.

In many applications, multi-material parts consisting of two different alloys (also called bimetals) have been so far developed by conventional dissimilar joining techniques [32]. These bimetals can satisfy superior mechanical, thermal, and corrosion resistance required for aerospace and tooling industries. Recently, it was shown that additive manufacturing can be combined with other conventional methods such as casting and metal forming to fabricate dissimilar parts [33]–[35]. For instance, electron beam melting of 316 stainless steel and Inconel 718 carried out by Hinojos et al. [34] was among the primary studies in this field. In addition, LPBF of Zr65.7-Cu15.6-Ni11.7-Al3.7-Ti3.3 (wt%) metallic glass on a 304 stainless steel substrate was successfully conducted by applying transition layers, which inhibited the formation of brittle intermetallic compounds [35]. More recently, maraging steels have been deposited using LPBF on both ferrous and non-

ferrous alloys such as AISI 420, H13, and copper [33], [36], [37]. Additively manufactured MS1-H13 bimetals can have the benefits of both conventional and additive manufacturing methods, where the 3D printed part can possess curvilinear cooling channels for better fatigue life with complex designs and the conventional part can be a block of tool steel, where there is no need for specific design characteristics [33].

Additive manufacturing has the potential to repair parts, as laser powder metal deposition techniques have been used for this purpose for many years in tool and die and aerospace industries. Powder bed fusion techniques can also be used in this regard to decrease the overall cost of replacing a damaged part. In addition, there is a huge interest in using hybrid procedures for on-platform maintenance (on the board of a moving vessel) in the marine and advanced shipbuilding industries. This study is an initial step to deposit hard stainless steels on top of shipbuilding steels, which contain more carbon content and has more joining issues. Despite the available studies in this field, the microstructural aspects of maraging steel-tool steel bimetals should be investigated in more detail. Therefore, the current work aims at investigating the microstructural features together with the microstructure-mechanical properties correlation of the deposited maraging steel on a conventional H13 tool steel fabricated by the LPBF process. MS1 is a high Ni content maraging steel powder developed by EOS GmbH using the gas atomization technique, which belongs to the 18% Ni maraging 300 class [33].

In this study, maraging steel MS1 powder was laser deposited on top of a H13 tool steel block using the LPBF process. Optical and scanning electron microscopy techniques were employed for initial observation of the MS1-H13 interface. Elemental analysis was conducted using electron probe microanalysis (EPMA) and energy dispersive X-ray

spectroscopy techniques (EDS) in the interface region of MS1 and H13. The microstructural study was then complemented by electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) techniques to further study the interface formation mechanism. Microhardness test results primarily formed the initial microstructure-mechanical properties correlation discussion mainly in the interface region. Further conducting uniaxial tensile tests on the LPBF-MS1-H13 samples and the assessment of the mechanical properties along with the fractography of fracture surfaces helped to investigate the bonding of the two alloys.

2.2 Experimental procedure

2.2.1 Materials and LPBF process

Gas atomized maraging steel (MS1) powder provided by EOS GmbH was used for the additive manufacturing process using the LPBF technique with the chemical composition shown in Table 2-1. The powder particles were 15-45 μm in diameter (Figure 2-1). A block of H13 hot work tool steel (chemical composition shown in Table 2-1) with 150mm $\times$ 70mm $\times$ 100mm (height) dimensions was fixed to the build plate of an EOS M290 additive machine (located at Additive Metal Manufacturing (AMM) in Concord, Ontario), where the bed temperature was held at 40 $^{\circ}\text{C}$ for the whole additive manufacturing process. The EOS M290 machine was a non-modular unit equipped with a 400W laser, which had a deposition platform with 250mm $\times$ 250mm $\times$ 325mm dimensions, a Yb-fiber laser type, and 100 μm spot size.

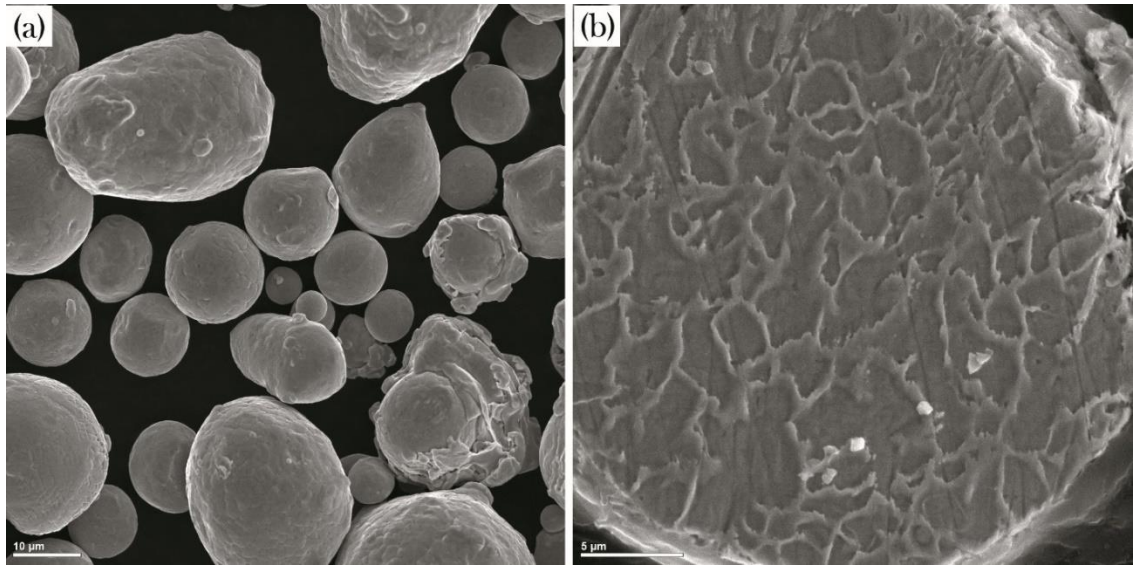


Figure 2-1: SEM powder particles, a) morphology, and b) microstructure

Table 2-1: Chemical composition (wt%) of the materials used in the current study

MS1 maraging steel							
C	Ni	Co	Mo	Mn,Si	Ti	Al	Cr,Cu
P,S	Fe						
≤0.03	17-19	8.5-9.5	4.5-5.2	≤0.1	0.6-0.8	0.05-0.15	≤0.5
≤0.01	Bal.						
H13 hot work tool steel							
C	Cr	Mo	Si	V	Fe		
0.32-0.45	4.75-5.5	1.1-1.75	0.5-1.25	0.8-1.20			
Bal.							

The H13 block acted as the substrate for depositing MS1 powder through the whole LPBF process. The H13 block was provided by Uddeholm Canada in Mississauga, Ontario. Round bars of 12 mm diameter with 100 mm height of MS1 were then deposited vertically on top of the H13 block as shown in Figure 2-2. Argon gas with a maximum 0.1% oxygen content was blown throughout the building process to protect the material from oxidation. The oxygen content was kept under 0.1% throughout the whole procedure. The H13 block was polished using normal mechanical grinding procedures for better interface formation

and superior bonding between the two steels. The process parameters used to deposit the MS1 powder throughout the process are listed in Table 2-2. Bars were then cross-sectioned along the building direction, as shown in Figure 2-2, using the electrical discharge machining (EDM) technique for material characterization purposes.

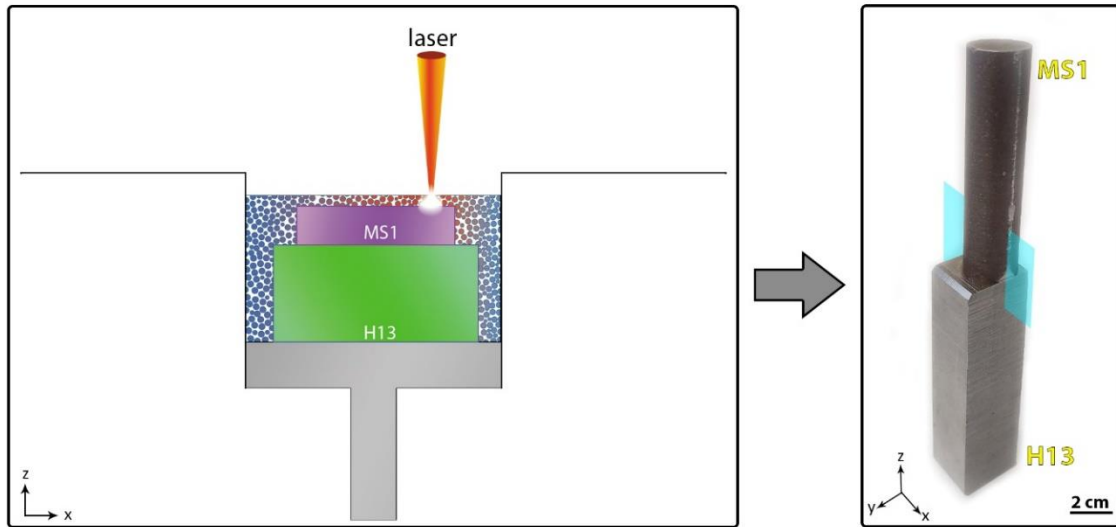


Figure 2-2: Schematic diagram of additively manufactured MS1-H13 bimetals using LPBF

Table 2-2: Process parameters

Laser power	Scan speed	Hatch distance	Layer thickness	Scanning strategy
285 W	960 mm/s	0.11 mm	40 μ m	Stripe-like

2.2.2 Mechanical testing

Three of the LPBF-MS1-H13 samples were sectioned vertically such that the interface positioned in the middle, then mounted using a Beuhler (SimpliMet 4000) hot mold and polished with SiC papers of 320-4000 grades. Vickers microhardness was measured using a Leica microhardness machine Wetzlar Model 8278 across the interface

with a 50 g load and the dwell time of 15 seconds according to ASTM standard E384-17 [38]. All the microhardness trials were repeated 5 times to ensure repeatability, their average values were then reported.

The rest of the samples were machined according to ASTM E8M standard for uniaxial tensile tests with 6 mm gauge diameter [39]. An Instron universal testing frame (model 1332) equipped with a hydraulic loading mechanism and a 25 mm Instron contact extensometer was used to conduct the uniaxial tensile tests at quasi-static strain rate of $9 \times 10^{-3} \text{ s}^{-1}$. The tests were performed three times until failure, where the extensometer ensured accurate measurement of the displacement. Engineering stress-strain curves were recorded, and a typical uniaxial tensile curve was then reported.

2.2.3 Microstructure studies

After polishing the LPBF-MS1-H13 steels, they were etched in Nital 1% ($1 \text{ cm}^3 \text{ HNO}_3 + 99 \text{ cm}^3 \text{ Ethyl alcohol}$ 1) for 210 seconds. Optical micrographs were taken using a Zeta-20 optical microscope at different magnifications. Scanning electron microscopy analysis was carried out using a Jeol 6400 SEM equipped with a Genesis energy disperse X-ray spectrometer (EDS).

Phase analysis of the two sides of the LPBF-MS1-H13 samples was performed using X-ray diffraction (XRD) technique with a Bruker D8 DISCOVER with DAVINCI DESIGN diffractometer. The instrument was equipped with Co-K α radiation ($\lambda = 0.178886 \text{ nm}$) and 0.02° (1 second) step size, over a 2θ range of 45° - 105° at 35 kV and 45 mA. Bruker-AXS software was used to collect the data, where Topas software version 4.2 was used to perform the Rietveld analysis.

Electron probe microanalysis (EPMA) to identify the elemental composition of the interface was performed using a Jeol JXA 733 microprobe. The instrument was equipped with four 2-crystal wavelength dispersive spectrometers including LDE1, TAP, PET, LIF crystals, where a Geller microanalytically automation control was used to conduct the probing.

Electron backscatter diffraction (EBSD) in a field emission gun scanning electron microscope (FEG-SEM) FEI Nova NanoSEM-650 equipped with a Hikari EBSD system was carried out to observe grain morphology of the substrate-H13 and LPBF-MS1. The EBSD samples were prepared using standard mechanical grinding and polishing up to 0.05 μm colloidal silica. The samples were analyzed at two magnifications of $\times 300$ and $\times 2000$, with step sizes of 300 nm and 80 nm, respectively. The results were presented in the form of inverse pole figure (IPF) and the corresponding grain boundary (GB) maps using high angle grain boundaries. Since additive manufacturing involves a building direction (z), inverse pole figure maps were presented along the z direction in form of an IPF-Z map. Thus, the crystallographic orientation of the grains was shown along the building direction.

Identification of grains in the printed MS1 material was conducted considering the grain shape aspect ratio, ϕ . This parameter represents the ratio between the grain minor axis (L_2) and major axis (L_1), where $\phi = L_2/L_1$. L_1 and L_2 were evaluated by fitting an ellipse to each individual grain, and the columnar and equiaxed grains were identified with $\phi \leq 0.33$ and $\phi > 0.33$, respectively [9].

Using an FEI Tecnai Osiris TEM equipped with a 200 keV X-FEG gun, the microstructure of the substrate-H13 and LPBF-MS1 samples were further studied. The Super-EDS X-ray detection system was used to acquire elemental analysis data mainly in

the interface formed between the two steels. TEM samples were prepared using the focused ion beam (FIB) lift-out automatic procedures from the interface region, using an FEI Helios NanoLab 650 dual-beam FIB.

2.3 Results

2.3.1 Microstructure of H13 and LPBF-MS1

Figure 2-1 (a) shows an SEM micrograph of the MS1 powder. The morphology of the typical MS1 powder particles used in the sintering process with near-spherical shapes and relatively smooth surfaces is clearly observed in the image. The finer particles are more spherical, while the coarser ones tend to form oval shapes. The fine spherical powder particles can enhance the packing density and flow of the powder as well as uniform distribution in each layer, which are beneficial for the mechanical properties of the final part [14], [40], [41]. Figure 2-1 (b) shows the microstructure of the cross-section of one powder particle. The cellular structure of MS1 powder with almost equiaxed cells solidified during the gas atomization process can be clearly seen in this figure.

SEM micrographs of the substrate-H13 are shown in Figures 2-3 (a). It shows a low magnification image of the hot work tool steel H13 in annealed condition prior to deposition of the MS1 powder. As shown, this material consists of equiaxed grains with almost equiaxed geometry and uniform distribution of precipitates in the matrix. In terms of grain morphology and texture, the substrate-H13 material consists of uniformly distributed equiaxed grains with almost a random texture (Figure 2-3 (b)). The evolution of such a structure and texture in the substrate-H13 is mainly due to the annealing treatment of the material prior to the LPBF process.

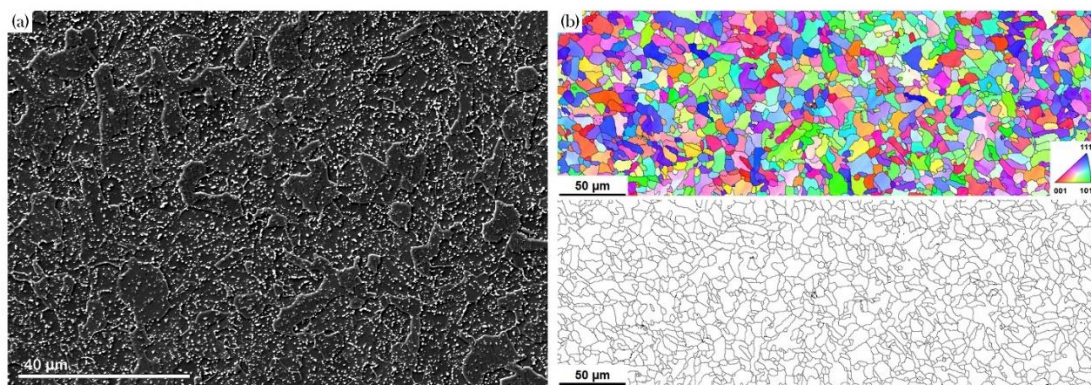


Figure 2-3: (a) SEM image of H13 (b) EBSD IPF-Z map along with the corresponding HAGB map of H13

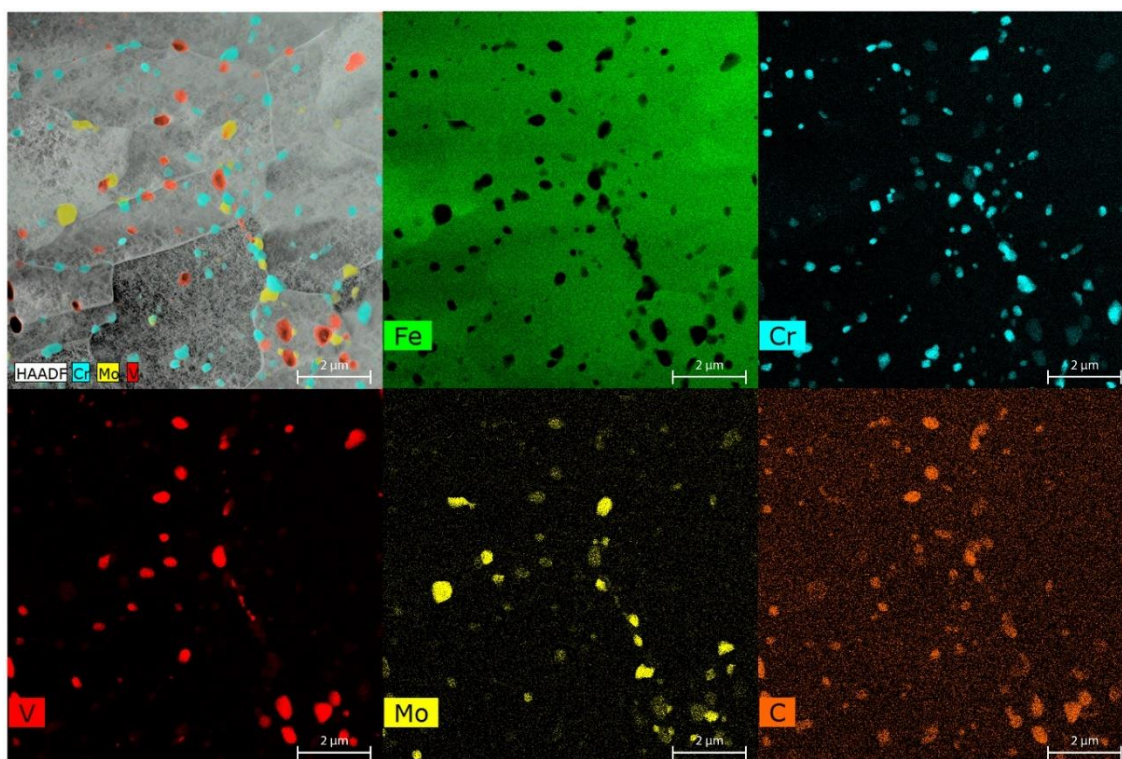


Figure 2-4: STEM-HAADF image of substrate-H13 along with the corresponding EDS elemental maps

To further study the composition of these precipitates, TEM elemental analysis was carried out at high magnification. Figure 2-4 illustrates the distribution of the main alloying elements highlighting the dominant elements of precipitates. The microstructure of H13 is highly saturated by relatively granular precipitates distributed in both transgranular and

intergranular spaces. The highlighted spots in each map present the precipitates enriched in the corresponding element. The V, Cr and Mo carbides reported to be V-rich MC, Cr-rich M_7C_3 and $M_{23}C_6$, and Mo-rich M_2C and M_6C carbides [42]–[45].

Figure 2-5 (a) shows the solidification morphology of the LPBF-MS1. The typical characteristics of additively manufactured metals can be observed, where overlapped melt pools with relatively semi-elliptical shapes were formed. The formation of semi-elliptical melt pools is inherent to LPBF techniques, for both ferrous [46] and non-ferrous [47] alloying systems due to the nature of the process, i.e. melting powder layers track after track. In terms of dimensions, melt pools are statistically 105-130 μm in width and 70-90 μm in depth. Melt pools were formed by solidification of both equiaxed and columnar cells of various sizes.

Figure 2-5 (b) presents the solidification morphology of the LPBF-MS1 in more detail, which is a cellular-dendritic morphology also reported earlier by Campanelli et al. [48]. This morphology comprises of both equiaxed and columnar cells. The geometry of the solidification cells (either equiaxed or columnar) can also be dependent on the cutting angle as Kürnsteiner et al. [19] reported earlier, where perpendicular and parallel sections to the build direction appeared equiaxed and elongated in the micrograph of a laser processed maraging steel, respectively. It is noted that, the evolution of solidification cells is related to the segregation of alloying elements at cell boundaries resulting in microsegregation, which was also observed in the LPBF of maraging steels [22]. Equiaxed cells can be seen in both fine and coarse sizes of 0.2-0.6 μm and 1-2 μm , respectively.

Figure 2-5 (c) shows the grain structure of the LPBF-MS1 based on high angle grain boundaries (HAGBs). A bimodal microstructure consisted of equiaxed and columnar

grains is observed in the LPBF-MS1. The columnar grains mainly evolved with a deviation from the building direction (z), probably due to the deviated heat gradient during solidification. Based on the alloy system, the evolution of columnar grains along the building direction can be predominant [49], [50]. Nevertheless, in the case of LPBF-MS1, such a structure did not evolve. Moreover, the crystallographic orientation of grains did not follow the common fiber texture in LPBF metallic materials due to the deviation of the columnar grains from the building direction [51]. In addition, the volume fraction of the columnar grains was less than the equiaxed ones weakening the fiber texture. Martensite laths can also be observed inside the grains as indicated by red arrows in Figure 2-5 (c), which is a common feature of laser processed maraging steels [20].

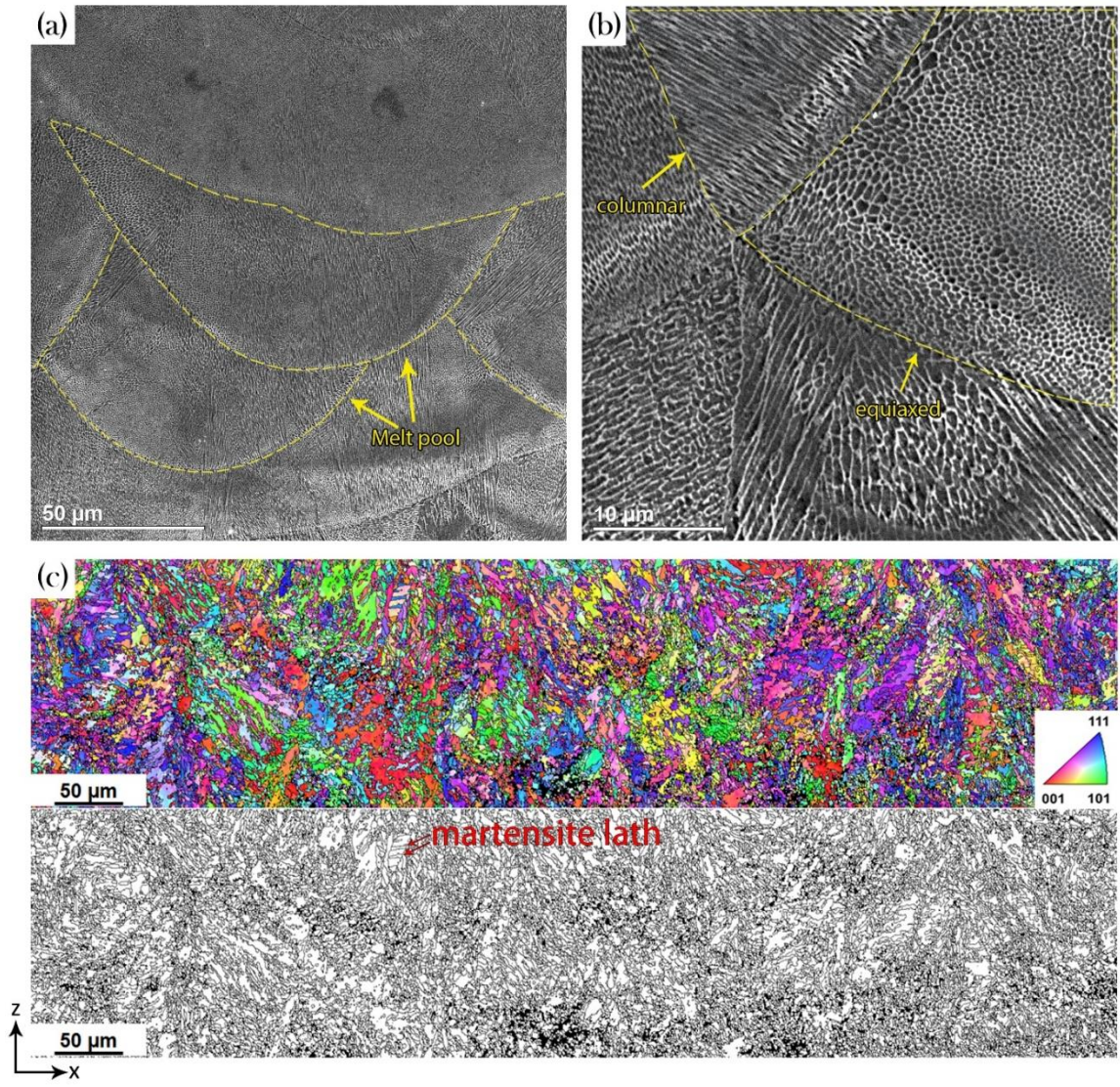


Figure 2-5: (a) and (b) scanning electron micrograph of LPBF-MS1 (c) EBSD IPF-Z maps along with the corresponding GB maps of LPBF-MS1

It is noted that, equiaxed and/or columnar grains should not be confused with equiaxed and/or columnar cells mentioned previously. Cells and grains are two different microstructural features of additively manufactured metals [46], [52]. A grain is defined as areas with the same crystallographic orientation surrounded by high angle grain boundaries (HAGBs). HAGBs are defined as boundaries with the misorientation of over 15° , while the misorientations between $2-15^\circ$ are considered as low angle grain boundaries (LAGBs)

[53]–[55]. The alignment of several solidification cells in the same crystal direction forms a grain. In other words, the LPBF-MS1 possesses a hierarchical microstructure, where solidification cells can be considered as the subset of grains. The hierarchical microstructure was observed in many additively manufactured metals including maraging steel grade 300, AlSi10Mg, Co-base alloys and 316L stainless steel [15], [22], [46], [56], [57].

The characteristics of grain structure in both the substrate-H13 and LPBF-MS1 were quantitatively analyzed using the EBSD results, as shown in Figure 2-6. Figure 2-6 (a) shows the fraction of grains with various ϕ -values in the LPBF-MS1 material. While approximately 33% of the grains possessed a columnar morphology, around 67% of them evolved with an equiaxed morphology. The dominance of the equiaxed grains in the LPBF-MS1 could be due to both variations of G/R ratio and the presence of dispersed oxide particles (mainly titanium and aluminum oxides [18]), which the latter promoted heterogeneous nucleation during solidification and the evolution of equiaxed grains.

The grains in both the substrate-H13 and LPBF-MS1 were analyzed in terms of grain size area and the results are shown in Figure 2-6 (b). While large grains with an average area of $88.2 \mu\text{m}^2$ were observed in the substrate-H13 material, much finer grains with an average area of $29.5 \mu\text{m}^2$ were developed in the LPBF-MS1 material. The evolution of fine grains in the LPBF-MS1 is due to very fast solidification cooling rates inherent to the LPBF process [6]. Due to the presence of columnar grains in the microstructure of the LPBF-MS1 material, the grain size was not evaluated in terms of grain diameter. Alternatively, the grain size in both the substrate-H13 and LPBF-MS1 materials was analyzed using the line intercept method by applying both vertical and horizontal lines, where the results are

presented in Figure 2-6 (c) and (d). Similar to the trend observed in the grain size, much finer grains in terms of intercept length were measured in the LPBF-MS1 as compared to the substrate-H13. While the average horizontal and vertical intercept length in the substrate-H13 was 4.7 μm and 5.1 μm , respectively, these values were 1.31 μm and 1.58 μm in the LPBF-MS1, respectively.

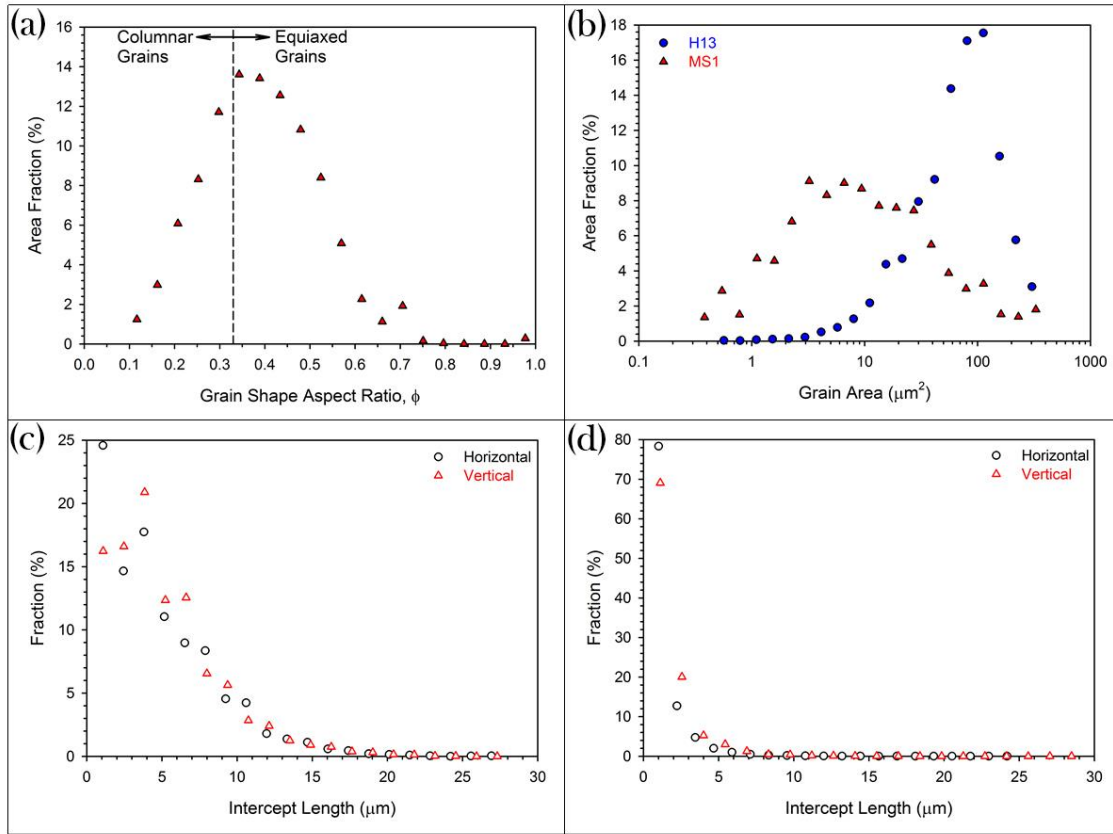


Figure 2-6: (a) Grain shape aspect ratio of LPBF-MS1, (b) grain size area in the substrate-H13 and LPBF-MS1, and line intercept length distribution in (c) the substrate-H13 and (d) LPBF-MS1

The X-ray diffraction patterns of the LPBF-MS1 and substrate-H13 are shown in Figure 2-7. The annealed substrate consists of only α peaks as ferrite. However, the LPBF-MS1 contains mainly α' martensite and a small amount of γ austenite phase. The presence of γ austenite was also confirmed in LPBF-maraging steel 300 in the as-built condition [21]. In fact, the high nickel content of MS1 facilitated the formation of martensite phase

[58]. Maraging steels are hardened by nanosized intermetallic precipitates upon aging heat treatment. Herein, precipitates could not be detected by XRD due to their small volume fraction. Using high energy X-Ray techniques, e.g. synchrotron measurements, the nm-sized precipitates in these alloys can easily be identified [59]. In addition, Jagle et al. [60] showed the absence of precipitates in LPBF-maraging steels via the atom probe tomography technique. Therefore, additively manufactured maraging steels are solid solutions with martensite and a small fraction of retained austenite phases without the presence of intermetallic precipitates.

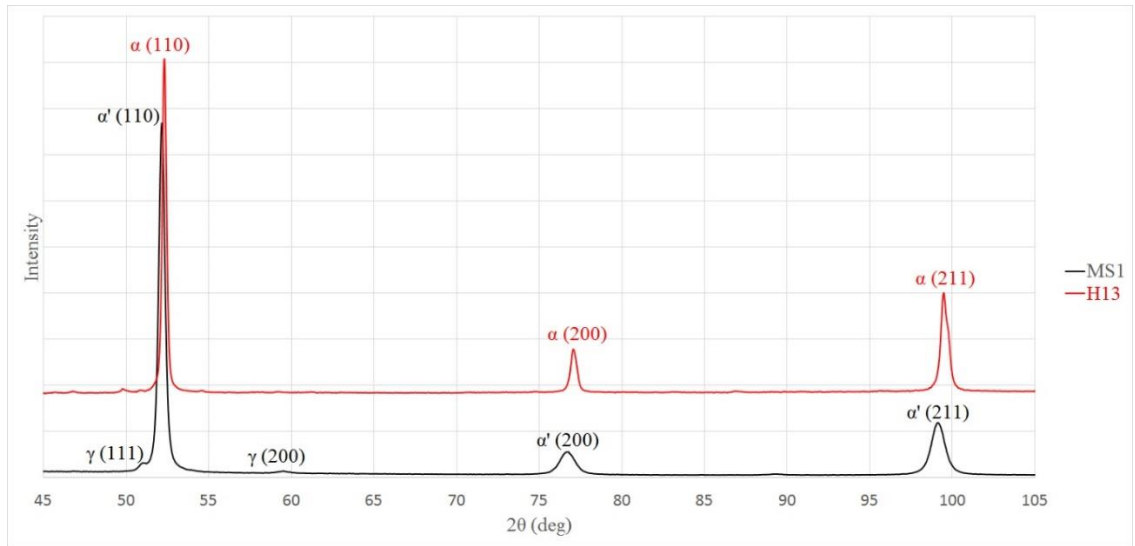


Figure 2-7: XRD patterns of the as-built MS1 and conventional H13

2.3.2 Characteristics of the interface in MS1-H13 bimetals

Figures 2-8 (a) and (b) show the optical microstructure of the MS1-H13 bimetals at different magnifications. As seen, MS1 is perfectly deposited on H13 without any discontinuity or cracking at the interface indicating a sound joint between the two sides. The fish-scale structure of melt pools is also evident indicating layer-by-layer deposition of MS1 on H13. The same structure was also observed in LPBF-maraging steel in previous

studies, where the height and the width of melt pools were related to powder layer thickness and laser diameter, respectively [20], [21]. The interface between H13 and MS1 is wavy in shape similar to other additively manufactured bimetals such as Zr-based metallic glass/304SS and MS1/420SS [35], [36]. The melt pools adjacent to H13 seem to be etched in different colors, which need further investigation. Backscattered electron micrograph of the MS1-H13 steel is also shown in Figures 2-8 (c) and (d). As observed, three elemental distinction regions were detected comprising of the substrate-H13, LPBF-MS1 and a transition zone between the two materials. At a higher magnification shown in Figure 2-8 (d), there are areas of irregular distribution of elements (called circulate flow), where they seem to be stirred and spread along the melt pool boundaries.

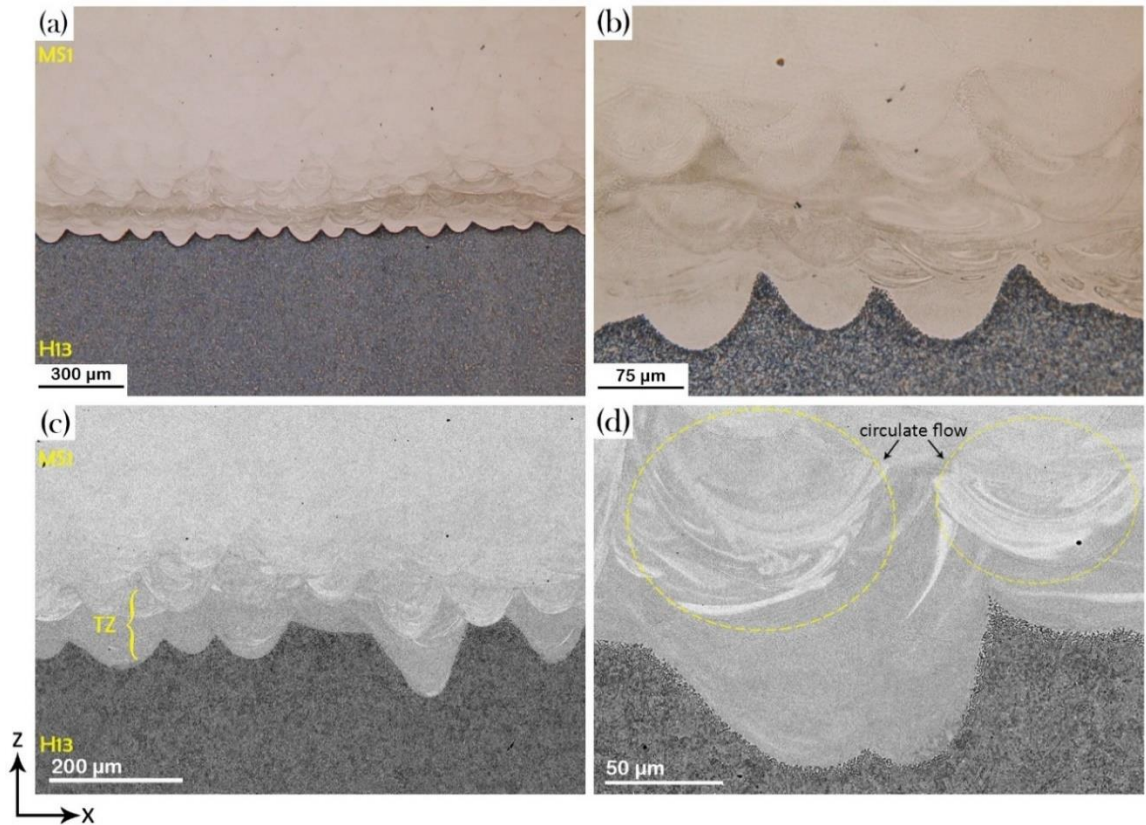


Figure 2-8: Interfacial micrographs of the MS1-H13 sample at (a) and (b) optical images (c) and (d) BSE images

The EBSD analysis of the interface revealed a visibly flawless joint between the LPBF-MS1 and substrate-H13, as shown by unique color grain map in Figure 2-9. The LPBF-MS1 exhibits a very fine microstructure in the first layer solidified on top of the substrate-H13 due to rapid heat transfer from the liquid material to the solid substrate. By depositing the successive layers, a slightly coarser microstructure evolved in the LPBF-MS1 due to high temperature of the previously deposited layers. In terms of average grain area, the first deposited layer contains ultrafine grains with an average area of $5.17 \mu\text{m}^2$, while this value for the LPBF-MS1 (away from the interface) is $38.34 \mu\text{m}^2$. In addition, no grain coarsening is observed in the substrate-H13 adjacent to the interface.

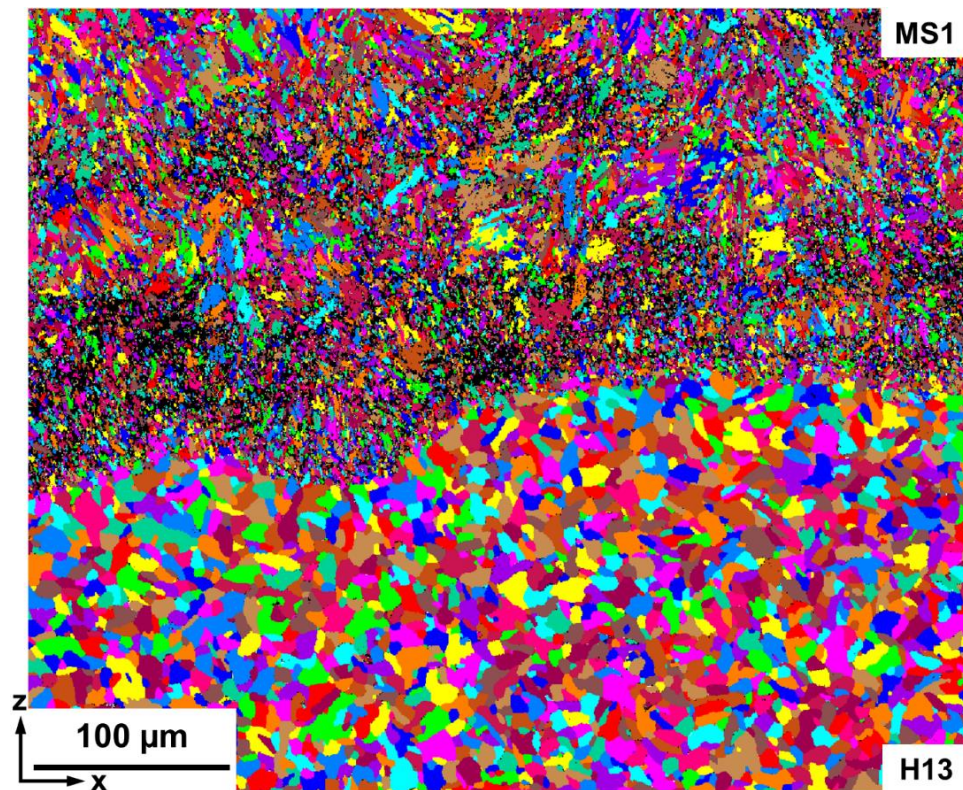


Figure 2-9: EBSD unique color grain map of the MS1-H13 sample

Further observation of the interface area between the substrate-H13 and LPBF-MS1 is depicted in Figure 2-10 (a), indicating a distinction between H13 and the printed side above it. Figure 2-10 (b) clearly shows the interface between the substrate-H13 and the first deposited layer. It seems that the precipitates in the substrate-H13 were coarsened due to partial melting and subsequent mixing with molten powder; hence forming a partially melted zone. In fusion welding, the partially melted zone (PMZ) was defined as the area where liquation can occur [61]. In other words, liquid-to-solid transition occurs in PMZ [62]; thus, the coarsened precipitates are representative of PMZ as shown in Figure 2-10 (b). The partially melted zone is approximately 2 μ m thick and highlighted the interface between H13 and MS1. As compared to the LPBF-MS1 microstructure, the printed area adjacent to the interface shown in Figure 2-10 (c) and (d) consists of similar morphology with melt pools accommodating coarse and fine equiaxed cells as well as columnar cells of submicron sizes.

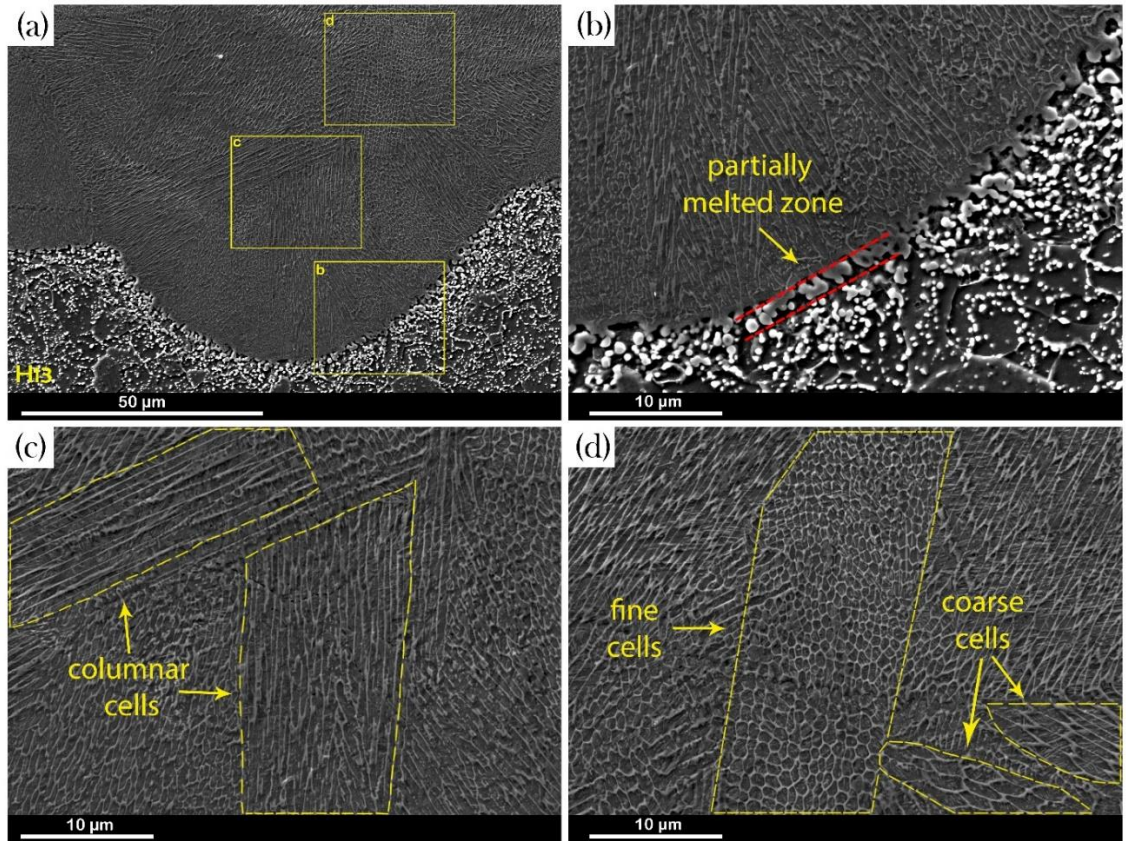


Figure 2-10: scanning electron micrographs of the MS1-H13 interface

Grain morphology of the interface in the MS1-H13 steel was analyzed using the EBSD technique at high magnification ($\times 2000$) and the results are shown in Figure 2-11. The EBSD results are presented in the form of IPF-Z and GB maps as shown in Figure 2-11 (a). In the GB map, the grain boundaries were separated to low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs), based on their misorientation (see Figure 2-11 (b)). Referring to the IPF-Z map of the interface, some of the equiaxed grains of the substrate-H13 were partially melted during their interaction with the laser beam at the interface (see Figure 2-11 (c)). Above the interface where the H13 grains were fully melted and mixed with the molten MS1 powder, ultrafine mostly-equiaxed grains were developed after solidification. These grains mostly developed a texture different from the

underlying equiaxed grains. Solidification of a liquid metal over a solid substrate generally follows the epitaxial growth [61], which results in the evolution of a columnar structure [56]. However, in the case of solidification of the MS1 liquid metal over the substrate-H13, epitaxial growth and columnar structure did not dominate the solidification process.

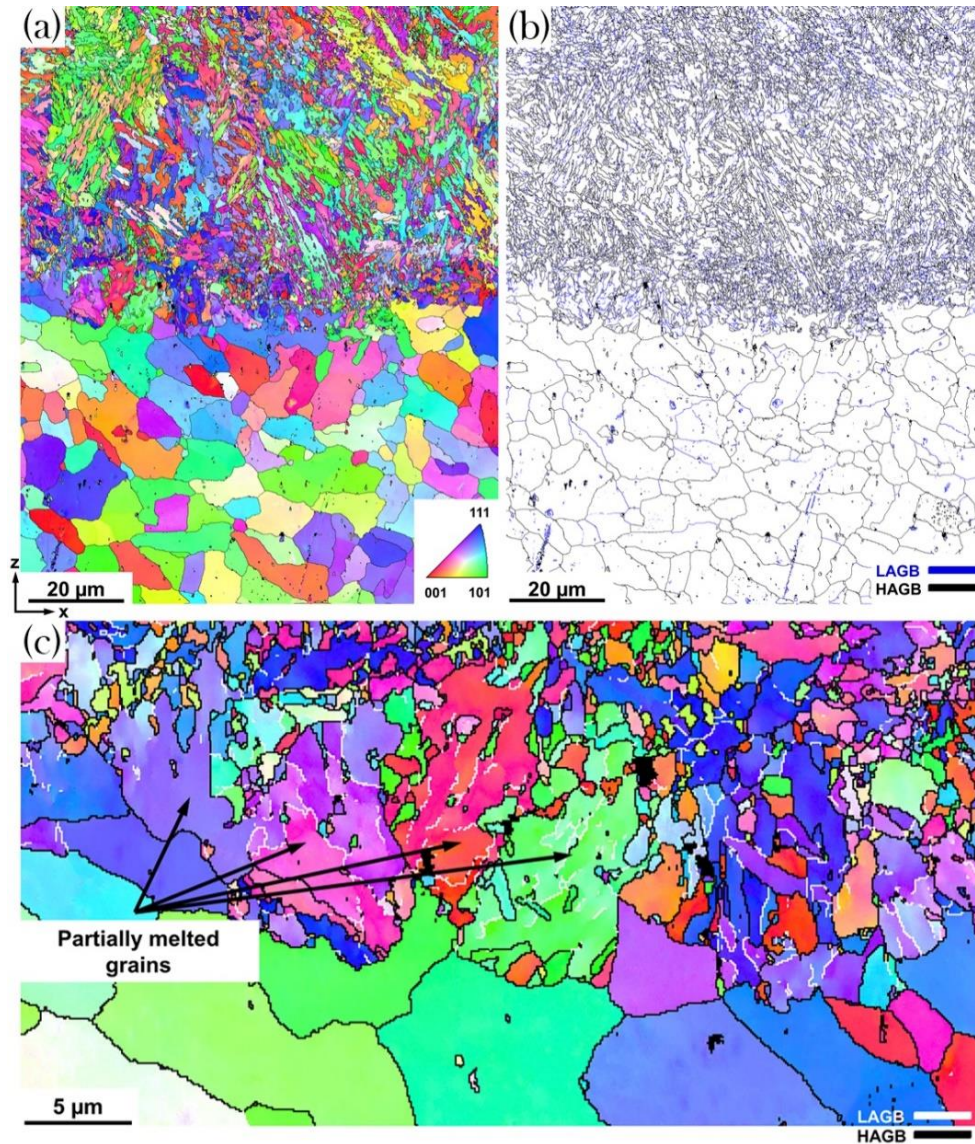


Figure 2-11: EBSD images of interface area in the MS1-H13 sample, (a) IPF-Z map, (b) GB map revealing both low angle and high angle boundaries, and (c) details of partially melted grains in the substrate-H13

Referring to the GB map of the interface (Figure 2-11 (b)), both LAGBs and HAGBs exist in the LPBF-MS1 structure adjacent to the interface. However, the majority of the grain boundaries in the LPBF-MS1 are HAGBs (~91%). Evolution of LAGBs and HAGBs follows the solidification behavior of the material. As seen in Figure 2-11 (c), the partially melted equiaxed grains in the substrate-H13, re-solidified by developing LAGBs inside the grains. It appears that these grains followed the original texture of the grain. However, due to partial melting and re-solidification, minor tilting occurred inside the grains, which resulted in the evolution of LAGBs. On the other hand, fine equiaxed grains solidified mostly by evolving HAGBs rather than LAGBs. Since the evolution of the interface was mainly controlled by full melting of the grains in the substrate-H13 and mixing of the MS1 melted powder and H13 liquid metal, the majority of the grain boundaries are HAGBs.

SEM-EDS elemental analysis carried out quantitatively across the MS1-H13 steel. Figure 2-12 (a) shows the location of all nine measurements, while Figure 2-12 (b) and Figure 2-12 (c) show the normalized concentration of Ni, Co, Mo, and Ti as the main alloying elements of MS1 and V, Cr, and Si as the main alloying elements of H13, respectively. It should be noted that, all the values were normalized to better present the trends. Equation (1) shows the normalization procedure used in this manuscript for each element as follows:

$$(N_C) = \frac{A_i(\text{wt}\%)}{A_{\max}(\text{wt}\%)} \quad (1)$$

where N_C is the normalized concentration, A_i is the concentration of the element at point i (i : 1-9) and $A_{\max}$ is the maximum concentration of the element. The first three points in

Figure 2-12 (a) represent H13 chemical composition in which there is no Ni or Ti content and other existing elements are in the range of nominal chemical composition of H13. Point 4 presents elemental concentrations in the first deposited layer adjacent to the interface. Interestingly, all elements from both sides coexist at this point; indicating that MS1 and H13 were chemically mixed, forming a region with considerable amount of both H13 and MS1 alloying elements. By moving to the next points (i.e., 5, 6 and 7) although the composition is still a combination of both metals, two opposing trends can be observed, the increase of MS1 alloying elements and the drop of H13 alloying elements. As an illustration, the percentage of Ni and Co almost doubled from point 4 to point 6, while V and Cr decreased by 70% and 90%, respectively. It can be stated that the chemical composition varies smoothly from the substrate-H13 to the LPBF-MS1 by forming an area of transition, which is here called “transition zone” (TZ) as highlighted in Figure 2-12 (a). The transition zone is actually from the interface up to point 8 forming an area of around 150 μm thickness with gradient concentrations. In other words, the chemical composition of the transition zone is neither MS1 nor H13, instead, a mixture of both alloys was formed.

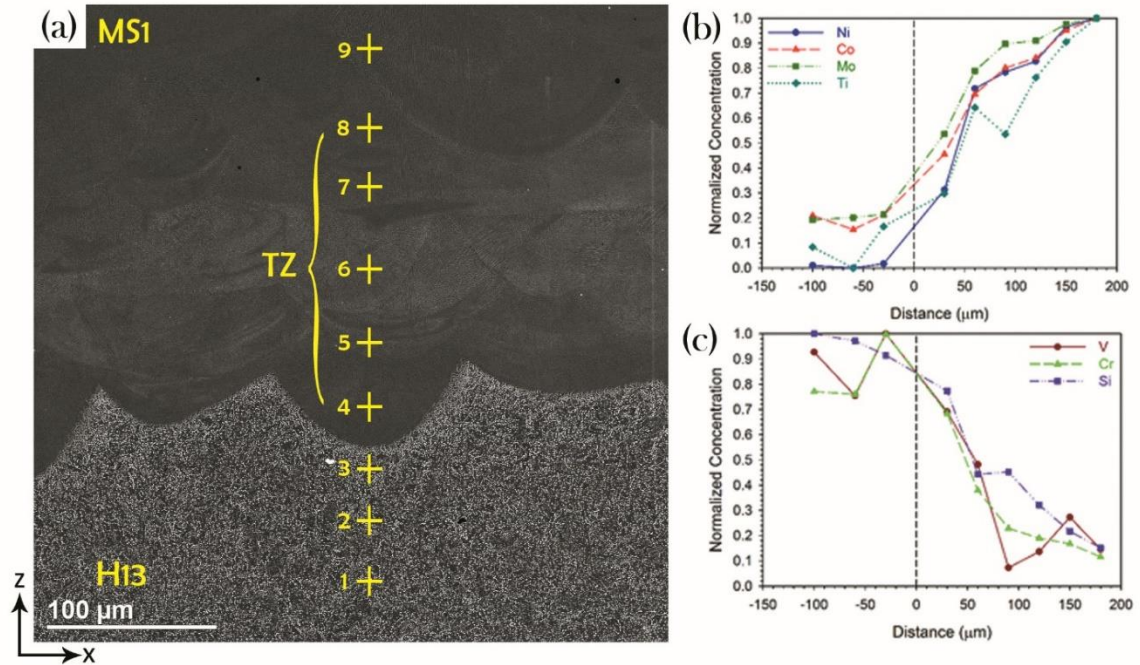


Figure 2-12: a) Interfacial SEM microstructure of the MS1-H13 sample with highlighted EDS analysis spots b) MS1 alloying elements distribution across the MS1-H13 interface c) H13 alloying elements distribution across the MS1-H13 interface

Figures 2-13 and 2-14 show electron probe microanalysis maps of the MS1-H13 steel. Since EPMA was equipped with four detectors, the analysis was conducted in two runs to detect all six elements. Ni, Co, and Ti were selected as the main alloying elements of MS1 as well as Cr, V, and Si for H13 tool steel. Due to the existence of Mo in both metals, it was disregarded from the elemental analysis. As shown, the elemental maps of Ni and Co as the MS1 alloying elements show higher concentrations further away from the interface, while Cr and V are more concentrated in the layers adjacent to the interface. In the first layer, where initial melt pools solidified on H13, all these four elements coexist; however, Cr and V are more noticeable than Ni and Co. Cr and V seem to decrease by moving from the first layer to the top layers, while Ni and Co become the major alloying elements. A similar trend was also observed for Ti and Si as shown in Figure 2-14, where transportation of both elements in opposite directions confirmed elemental dilution.

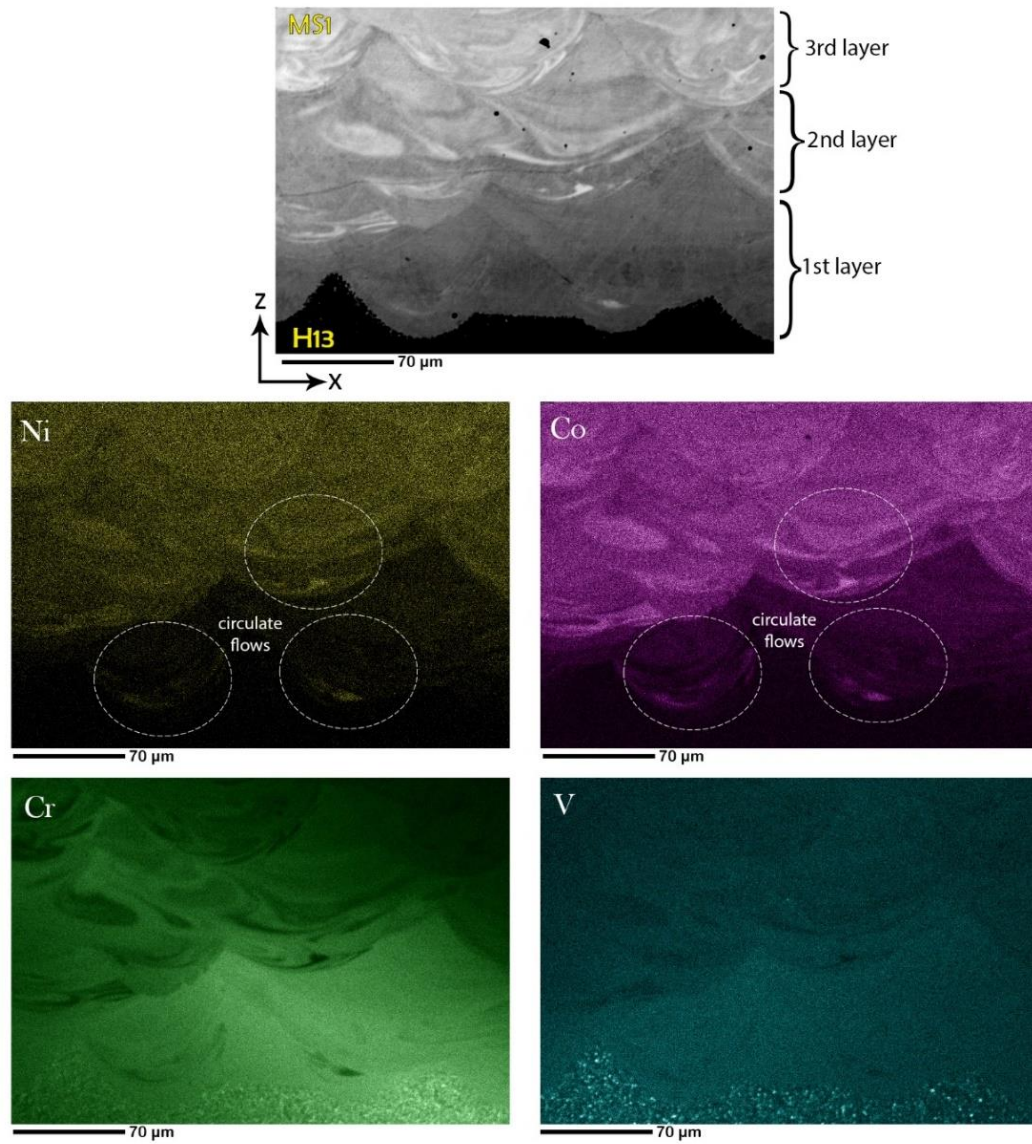


Figure 2-13: EPMA elemental mapping of the MS1-H13 interface

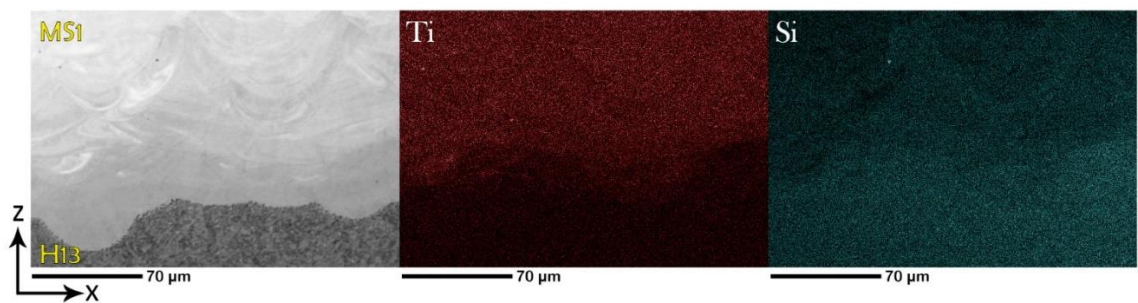


Figure 2-14: Ti and Si distribution taken by EPMA across the MS1-H13 interface

In addition, there are areas of irregular distribution of elements called circulate flows, where they seem to be stirred and spread along the melt pool boundaries as shown in Figure 2-13. Obviously, circulate flows are dominated by Ni and Co and simultaneously depleted from Cr and V. These areas (circulate flows) were formed in the bottom center of the melt pools and interestingly concentrated in the MS1 alloying elements (i.e., Ni and Co); thus, the effect of fluid flow particularly Marangoni convection is evident in the formation of the circulate flows. It is well understood that fluid flow of the melt pool is governed by forces such as thermocapillary convection (Marangoni effect) and Buoyancy force in additive manufacturing [1]. However, the effect of Buoyancy force is negligible and hence Marangoni convection governs the flow of the molten metal [1]. These areas were also related to Marangoni convection in additive manufacturing of maraging steel on copper and mentioned as circulate flows [37].

Due to the fine solidification morphology of additively manufactured maraging steels, details of the interface characteristics were analyzed using TEM in the scanning mode (STEM). Figure 2-15 shows the STEM-high angle annular dark field (HAADF) image of the MS1-H13 steel and the corresponding EDS elemental maps of the alloying elements. Regarding the substrate-H13, the precipitates can be Cr-, V- and Mo-rich carbides as were also discussed in Figure 2-4. The precipitates of H13 seem to be surrounded by a cloud of Cr, V, and Mo highlighted in faint colors. These elements diffused out from the core of the precipitates to the substrate most probably as a result of thermal cycles of the process. Moving upward, the partially melted zone (PMZ), i.e. few microns, appears with the agglomerated precipitates rich in Mo, Cr and V. On top of PMZ where the transition zone is located, the alloying elements distributed almost uniformly and no

precipitate was formed. However, Cr, Mo and V segregated slightly at the interdendritic regions of the cell regions indicating microsegregation of the main alloying elements of the substrate-H13 due to rapid solidification. This also proves the dissolution of the substrate-H13 alloying elements as presented previously in the EPMA results. The circulate flow region can also be observed from Ni mapping, which is also rich in Co.

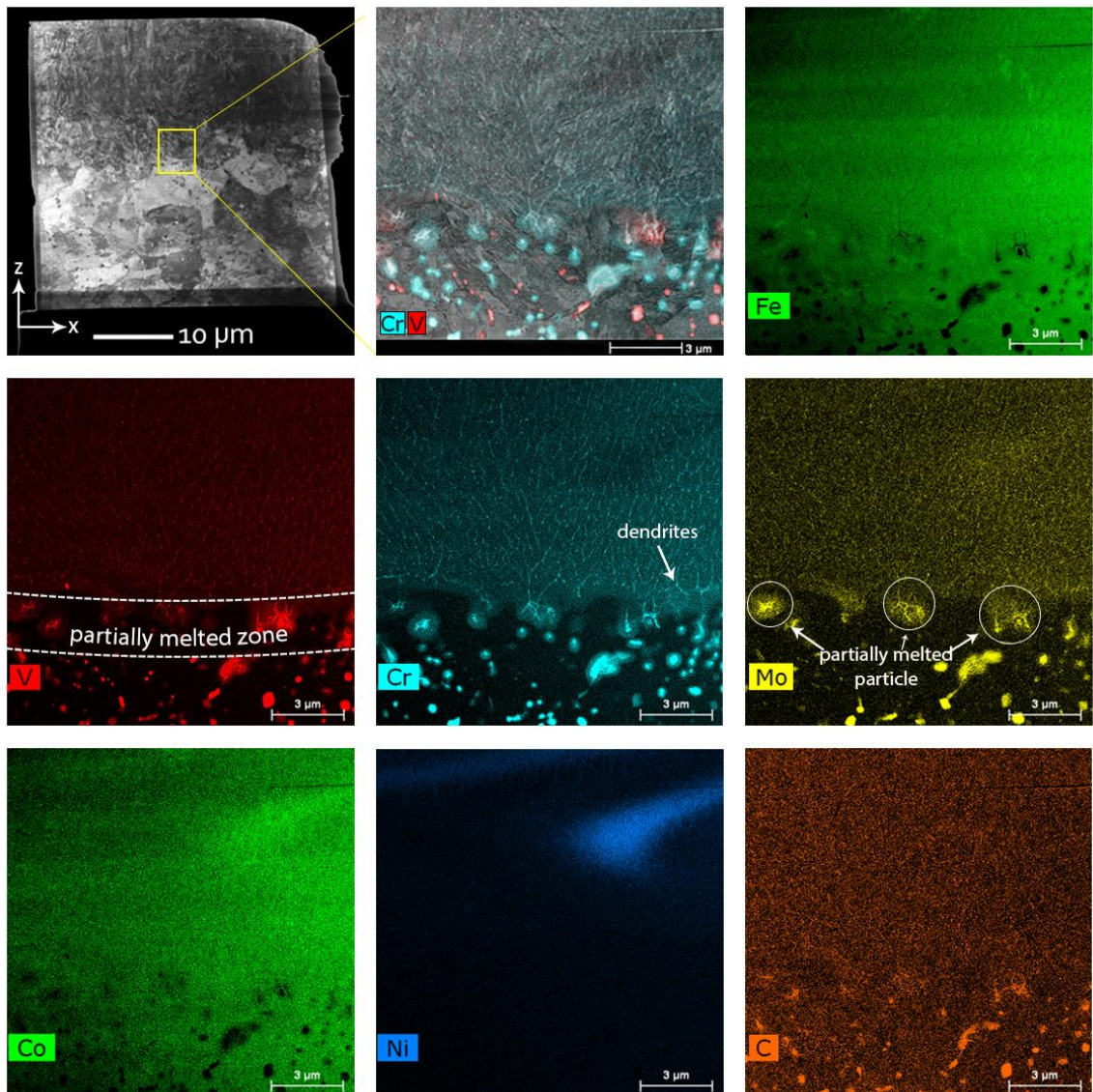


Figure 2-15: STEM-HAADF image of the MS1-H13 interface

2.3.3 Mechanical properties of MS1-H13 bimetals

The hardness variation across the steel as a quantitative representation of the mechanical properties is illustrated in Figure 2-16. Low hardness of H13 with respect to the printed side is apparent with an average value of 232 HV. The hardness of annealed H13 was reported to be 229 HB, which is equivalent to 235 HV [63]. There is no hardness gradient in the substrate, particularly adjacent to the interface. From the interface, the hardness values mount abruptly peaking at 644 HV at the nearest point of 30 μm to the interface and then drop steadily until reaching MS1 hardness of 423 HV. Apparently, the first deposited layer is the hardest region with a mean value twice as MS1 and three times higher than H13.

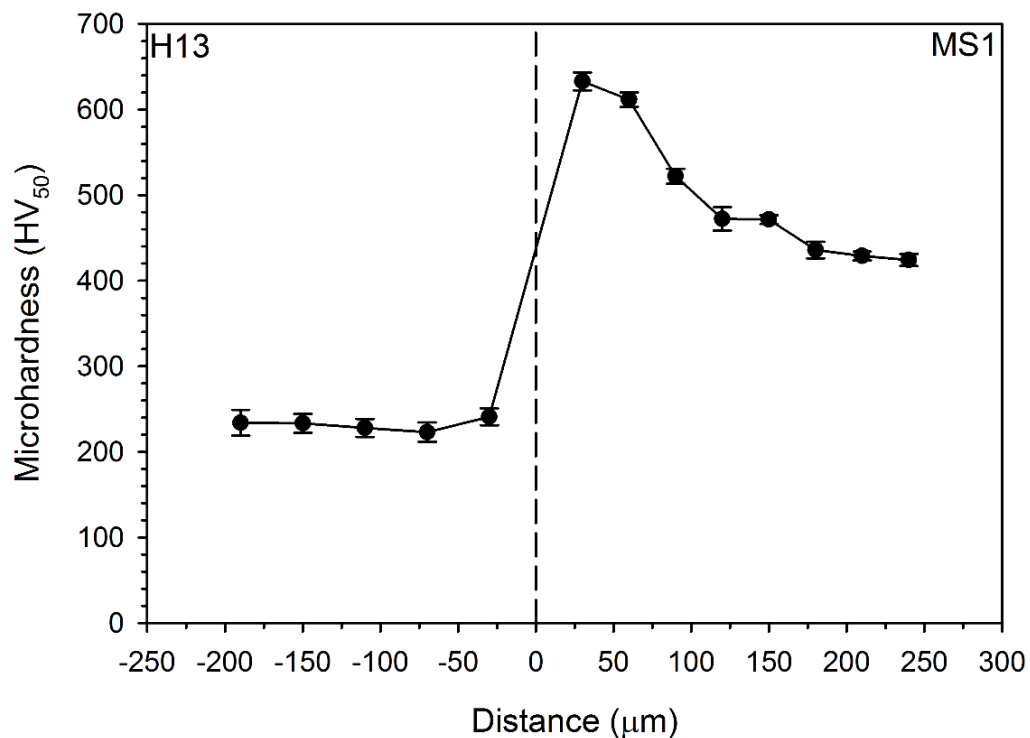


Figure 2-16: Microhardness profile across the MS1-H13 sample

The engineering stress-strain curve for the MS1-H13 steel is presented in Figure 2-17 (a) as well as that of as-built MS1 for comparison. The as-built MS1 exhibited ultimate tensile strength (UTS) of 1126 MPa, whereas the corresponding value for the MS1-H13 steel is 664 MPa. However, the MS1-H13 steel fractured in higher ductility in comparison with the as-built one.

Scanning electron microscopy of the fracture surface of the MS1-H13 steel is shown in Figures 2-17 (b) and (c), where fracture occurred in the substrate-H13. As shown, this is a ductile fracture with typical cup and cone features in macroscopic scale. At higher magnifications, the fibrous zone of the center contains fine dimples indicating the fracture mode of ductile rupture (Figure 2-17 (c)). It is well known that, the dimple rupture is due to a mechanism called microvoid coalescence, where microvoids nucleate at regions high in concentrated strain [64]. These regions were determined as secondary phases, grain boundaries and dislocation pile-ups [64]. In case of annealed H13, the dislocation pile up during tension increases stress concentrations, which causes the separation of the precipitates and matrix and as a consequence the formation of dimples [65].

Fine precipitates of the substrate-H13 could act as microvoids since the sizes of precipitates are comparable to that of dimples. The central fibrous region is surrounded by radial marks representing rapid propagation of cracks by shear stresses (Figure 2-17 (b)).

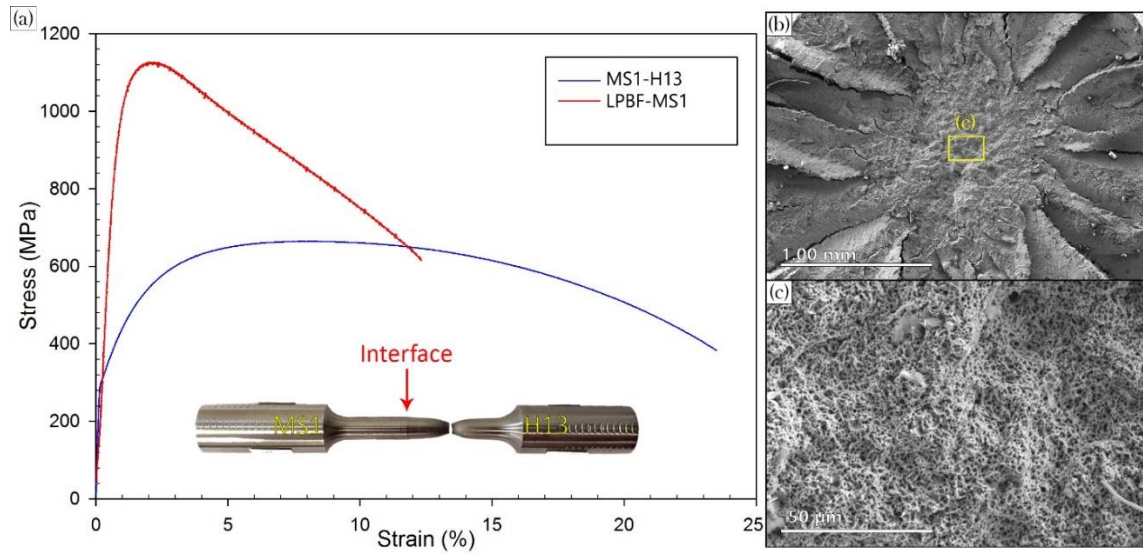


Figure 2-17: (a) Engineering stress-strain curve of the MS1-H13 steel and LPBF-MS1 (b) and (c) SEM image of the fracture surface at H13

2.4 Discussion

The reliability of the MS1-H13 steel fabricated through the LPBF process was shown in Figure 2-8. In terms of crystallographic structure, both steels seem relatively similar to each other since martensite with BCT structure is close to BCC structure of ferrite. Thus, a compatible bonding can be expected between MS1 and H13. One of the serious issues towards bimetallic structures is delamination, which causes the complete failure of the sample. The common solution is to gradually change chemical composition between the two materials via interlayers [66]. However, no delamination was observed in the current work and the sound joint between the two materials indicates the success of additive manufacturing to fabricate bimetals. Microstructural characterization of the MS1-H13 steel reveals four main areas as substrate-H13, partially melted zone (PMZ), transition zone (TZ) and LPBF-MS1. In particular, the characteristic areas of the MS1-H13 steel are PMZ and TZ developed via the interaction of the laser with the MS1 powder layer and

substrate-H13. The evolution of the interface and the microstructure-mechanical properties correlation are further discussed in the following two subsections.

2.4.1 Formation mechanism of the interface in MS1-H13 bimetals

The laser first fuses the powder and a fraction of the substrate to form a melt pool. In this case, the chemical composition of the melt pool is no longer that of the maraging steel; instead, it is a combination of laser processed maraging steel particles and the substrate-H13 forms the melt pools due to the dilution effect. Dilution is the result of the partial melting of the substrate-H13 and the mixing of its alloying elements with the melted MS1 powder. Dilution of alloying elements has also been observed in IN718/GRCOP-84 bimetallic structures fabricated through LPBF [67]. In this work, quantitative and qualitative elemental analyses confirmed this phenomenon in the melt pools adjacent to the interface (Figures 2-12 to 2-15). Indeed, dilution formed a solid solution rich in Cr, Mo and V (H13 alloying elements), which was previously introduced as the transition zone. Therefore, the solid solution strengthening mechanism can be expected in TZ leading to higher strength of the interface. The effect of dilution becomes negligible when further layers are deposited and finally by approaching point 9 in Figure 2-12 (a), the chemical composition is fully equivalent to the maraging steel MS1.

Marangoni convection (shown as circulate flow in Figure 2-8 (d)) causes the stirring of the melt pools and segregation of the MS1 alloying elements such as Ni and Co to the bottom of the melt pools. It was reported that, the temperature difference between the center and the edge of the melt pools generates surface tension and consequently Marangoni convection [68]. It can also assist dilution of alloying elements leading to the distribution of the substrate-H13 alloying elements throughout the melt pools. Thereafter, the PMZ

forms via liquation of the substrate precipitates followed by agglomeration resulting in coarser precipitates adjacent to the melt pools. The agglomerated precipitates act as nucleation sites for solidification. The circulate flow along with dilution can be considered as the unique characteristics of the printed material close to the interface area in comparison with the LPBF-MS1 far from the interface.

Heterogeneous nucleation commences on top of the agglomerated precipitates. Since the LPBF process is inherently high in cooling rates, rapid solidification causes the formation of a fine dendritic-cellular structure. The solidification morphology of the transition zone is identical to the as-built maraging steel (see Figure 2-10). It is well understood that various combinations of temperature gradients (G in K/m) and solidification rates (R in m/s) lead to the formation of different morphologies [61]. The variations of G/R ratio in different locations as a result of local changes in the cooling conditions and thermal boundaries result in the variation of constitutional undercooling during solidification [61]. As a result, the stability of the solidification front changes so that the solidification mode varies between columnar and equiaxed [56]. On the other hand, the fineness of the structure is determined by the product of G and R ($G \times R$ in K/s) [47], i.e. higher cooling rates result in finer structures. This is the reason for the very fine microstructure of the first deposited layer on top of the substrate-H13. In fact, the substrate-H13 acted as a powerful heat sink medium and promoted rapid solidification in the first layer of the LPBF-MS1. The fine cellular structure was reported to have a direct effect on the hardness and strength of the LPBF- maraging steel [18]. Moreover, no secondary phase was formed upon the solidification in the transition zone (TZ) although the substrate alloying elements (i.e., Cr, Mo and V) were dissolved in the melt pools via dilution. This

could be related to the low carbon content of maraging steel and the low tendency to form carbides. Hence, the transition zone (TZ) solidifies as a rich solid solution with fine grains and solidification morphologies.

Figures 2-10 and 2-11 clearly show no precipitation dissolution and/or grain growth underneath PMZ; thus, the substrate-H13 did not go under any microstructural evolution. This could be due to the local melting of the powder on top of the substrate-H13, which did not result in a heat pile-up in the substrate-H13 and grain growth. Therefore, it can be concluded that no heat affected zone was formed and the substrate-H13 was remained unaffected during the process. Heat affected zone (HAZ) was defined as the area of the weldment, where solid state phase transformations occur [62]. Additive manufacturing can also be considered as a joining method since two materials were bonded via this process. In this regard, the unaffected substrate is of great achievement in the additive manufacturing process. HAZ or its derivatives such as thermo-mechanically affected zone (TMAZ) or diffusion affected zone (DAZ) have always been a serious challenge in welding and brazing methods, which deteriorates mechanical properties and the performance of materials [69]–[73].

2.4.2 Correlation of microstructure and mechanical properties in MS1-H13 bimetals

The higher hardness values of MS1 as compared to the substrate-H13 can be attributed to the finer grain structure, cellular solidification morphology, and martensitic matrix of the as-built maraging steel. Likewise, the transition zone revealed the same behavior since this area is dominated by grain and solidification morphologies identical to maraging steels (see Figures 2-5, 2-9 and 2-10). Furthermore, this area is a solid solution

enriched in the substrate alloying elements and hence affected by solid solution strengthening. Consequently, Hall-Petch strengthening, hardening effect of the cellular structure, and solid solution strengthening mechanisms contribute to the hardness of the transition zone. The hardness peak represents the considerable effect of solid solution strengthening in TZ adjacent to the interface. In the regions farther from the interface, the descending trend of hardness of TZ towards MS1 implies the decline of dilution and its consequent effect of solid solution strengthening.

It seems that the fabricated MS1-H13 steel is weaker than the as-built MS1, however, the MS1-H13 steel necked and failed from the substrate-H13. The higher ductility and lower strength of the MS1-H13 steel were affected by the substrate that had been in soft annealed condition and lower hardness as compared to the maraging steel. As a consequence, the interface remained immune from failure under uniaxial tensile loading. The fractography results showed a ductile fracture, which is typical of annealed H13 tool steels [65]. It confirms that the fracture location as the weakest part of the MS1-H13 steel did not undergo any microstructural evolution during the process and no heat affected zone was generated in the MS1-H13 steel. More importantly, fracture was in the substrate and considerably far from the interface, which means the additive manufacturing process was successful.

2.5 Conclusions

In this study, laser-powder bed fusion of maraging steel MS1 on H13 hot work tool steel was carried out resulting in a sound MS1-H13 steel. Using different multiscale characterization techniques including OM, SEM, EPMA, EBSD, FIB-SEM, TEM, EDS, and XRD along with different mechanical and physical tests, the interface and

microstructure of the developed MS1-H13 steel were studied. No cracks, delamination or discontinuities as well as no HAZ in the substrate were formed, indicating the success of the LPBF process for the MS1-H13 system. Thus, the process can be used and further substituted for applications, where huge HAZ zones are detrimental. The following points can be deduced from the current study:

- A wavy shape interface of approximately 2 μm between two dissimilar steels was generated via LPBF forming four main microstructural areas: substrate-H13, partially melted zone, transition zone, and LPBF-MS1.
- The interface consisted of the agglomerated precipitates with the partially melted grains of the substrate-H13 forming the partially melted zone.
- Solidification of ultrafine grains on top of the partially melted zone led to an abrupt change from the coarse grains of the substrate to the ultrafine grains of the transition zone. In addition, this zone was enriched in the substrate alloying elements forming a rich solid solution. Due to the low carbon content of the MS1 powder, no carbide or intermetallic compound was precipitated.
- Hardness values increased suddenly at the interface and decreased steadily until reaching the MS1 hardness. Higher hardness of the transition zone can be due to Hall-Petch strengthening, hardening effect of the cellular structure, and solid solution strengthening mechanisms.
- The substrate-H13 remained intact during the LPBF process as no grain evolution, solution, and coarsening of precipitates were observed adjacent to the interface.

- Performing uniaxial tensile tests, failure of the MS1-H13 steel occurred in the substrate-H13 far enough from the interface.

2.6 Reference

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3 Interface engineering of additively manufactured maraging steel-H13 bimetallic structures

3.1 Introduction

Additive manufacturing (AM) has been considered a game-changer in the fabrication of metallic parts due to its high accuracy and ability to produce near-net-shape, high efficiency in terms of cost and lead time, and flexibility in alloy design [1]–[8]. AM parts are expected to replace many conventionally-fabricated components and assemblies, especially those including cast parts, taking advantage of improved strength and production lead time. Since metal solidification in AM is extremely fast, it leads to very fine microstructures and often higher mechanical properties. Two common AM techniques are direct energy deposition (DED) and powder bed fusion (PBF) [9]. DED is very similar to fusion welding, where a powder or wire feedstock is melted and deposited on a substrate, and then a layer-by-layer accumulation of the deposited feedstock forms a three-dimensional geometry [10], [11]. In PBF, a very thin layer of powder feedstock is first spread on a substrate, then pre-defined zones are selectively melted using a high-powered laser or electron beam. The rapid energy source scanning leads to ultra-fast melting and subsequently cooling and solidification within the scanned area, which then forms a fully-dense solid layer. The sequential repetition of this process finally develops a 3D geometrical shape [12]–[14].

Metal additive manufacturing is capable of producing a wide range of materials, such as high-temperature alloys, metal matrix composites, high entropy alloys, functionally graded materials, and bimetal structures [15]–[21]. The latter has recently been the center

of attention in many publications [22]–[25]. Herein, bimetals incorporate two different materials fabricated by both additive manufacturing and conventional manufacturing. Thus, the whole process can be called hybrid additive manufacturing (HAM) in which the product is a bimetal. In this regard, HAM resembles dissimilar fusion welding, where two different materials are melted and joined to fabricate a multi-material weldment. Generally, an as-cast material is used as the substrate on which the AM feedstock is deposited using DED or PBF techniques [22], [23]. The primary goal is to benefit from the characteristics of two dissimilar materials in order to boost functionality in a component called a bimetal. HAM can also ensure higher strength and endurance of in-service parts due to enhanced mechanical properties related to AM processing. In other respects, AM processing of bimetals can facilitate repair and renovation of tools and dies in metal forming industries as well as offshore maintenance of components in marine and shipbuilding sectors [24].

Several attempts have been made to fabricate bimetals out of different alloy systems such as IN718/GRCop-84, IN718/SS316, Ni6082/SS304, AlSi10Mg/C18400, and Zr-metallic glass/SS304 [25]–[29]. In 2014, one of the first additively manufactured bimetal structures was carried out by Liu et al. [30], where they used selective laser melting (SLM) to print SS316L/C18400 copper alloy for applications in fusion reactors, conformal cooling channels, and high field pulsed magnets. This made use of the good conductivity of copper along with high strength and corrosion resistance of steel. Onuike et al. [31] also studied the laser net energy shaping of Ti6Al4V/SS410 as a promising candidate in the aerospace and petroleum industries. Hadadzadeh et al. [20] developed aluminum two-material component for geometrically complex molds by depositing AlSi10Mg on a block of as-cast Al-Cu-Ni-Fe-Mg alloy using SLM.

Maraging steel is already well known as a material of interest in additive manufacturing due to its excellent printability, and ease of martensite formation via rapid cooling inherent in AM [32]. A good combination of strength and ductility has made this material applicable in aerospace, automotive, and submarine industries [33]. Recently, additively manufactured maraging steel bimetals have fascinated researchers due to maraging steel's excellent weldability to other materials in addition to its predominant mechanical properties. As an illustration, a grade 300 maraging steel and copper bimetal was fabricated using SLM showing higher strength than the substrate and failing away from the interface [34]. The same result was observed in an MS1-H13 bimetal, where a defect-free interface was formed via direct metal laser sintering (DMLS) [24]. Low cycle fatigue strength of maraging steel-H13 bimetal was also compatible with sintered 18Ni300 maraging steel [35], [36].

Although maraging steel bimetal structures show excellent bonding and high strength at the interface, the inhomogeneous microstructure across the interface can degrade the performance of bimetallic components in service conditions. As an illustration, it has been shown that, the MS1-H13 bimetal fails within the soft substrate under uniaxial loading, while the interface and the AM side are higher in strength [24]. In addition, there is an abrupt change of grain morphology, solidification structure, and chemical composition across the interface, which can reduce fatigue life and even corrosion resistance of maraging steel bimetal. In this regard, the modification of microstructure across the interface is essential to ameliorate the properties of maraging steel bimetal structures.

For many years, heat treatment has been applied as a promising method to modify the microstructure of as-fabricated metals. Several studies have investigated heat treatment

of AM maraging steels as well as conventionally made H13 tool steel in order to modify the microstructure and promote mechanical properties [37], [38]. Heat treatments of conventional hot work tool steels were well studied, and hardening treatment was identified as a favorable heat treatment to achieve high strength martensitic structure in hot work tool steels [39]–[41]. For AM maraging steel, Bai et al. [42] did extensive studies on solution and aging of selective laser melted 18Ni-300 maraging steel at different holding times and temperatures showing the modification of microstructure as well as tensile properties upon different heating cycles. Similar study was carried out by Song et al. [43] emphasizing more on sequential heat treatments. In their work various combination of solution treatments followed by aging treatments were applied on 18Ni-300 maraging steel. The enhancement of strength and hardness as well as the formation of martensite phase are typically the commonplace consequences of heat treatments on additively manufactured maraging steels [42]–[45]. However, the literature lacks studies on heat treatment modification of AM bimetal structures, particularly the MS1-H13 system.

This study proposes a heat-treatment process for the MS1-H13 bimetal aiming to remove microstructural inhomogeneity across the interface. The solution and aging treatments of MS1 were applied individually to find out the optimum heat treatment for the MS1-H13 system. Different electron microscopy techniques were carried out to study the microstructural changes as well as the interface removal mechanism upon heat treatment. By performing uniaxial tensile and nanoindentation tests, the enhancement of mechanical properties induced by the appropriate heat treatment was determined for the MS1-H13 bimetal.

3.2 Experimental Procedure

A build plate made of H13 hot work tool steel with 250mm × 250mm × 325mm dimensions, was placed as the substrate in an EOS M290 additive machine. The H13 tool steel was in the fully annealed condition and polished prior to the AM process. Maraging steel powder (MS1) produced by EOS GmbH was selected as the feedstock material with particle diameter of 15 – 45µm. Table 3-1 presents the chemical composition of the substrate and feedstock materials in more detail. The EOS M290 was equipped with a 400W Yb-fiber laser heat source. The direct metal laser sintering (DMLS) process was started at a preheat temperature of 40°C in an argon atmosphere with 99.99% purity. The MS1 powder was deposited and laser welded layer by layer on the H13 build plate in order to print round bars with Ø 12mm × L 100mm dimensions. The final product was several MS1-H13 bimetal consisting of an H13 block and MS1 rods.

As shown in Figure 3-1, the MS1-H13 bimetals were then post-processed via two different heat treatment cycles: (i) Aging treatment, which was carried out at 490 °C for holding time of 6 h with air cooling, and (ii) solution treatment, which was started with preheating at 815 °C followed by rapid heating to 982 °C for one hour and finally air cooling to room temperature. Throughout the study, the samples were designated as set 1 (as-built), set 2 (age-treated), and set 3 (solution-treated).

X-ray diffraction (XRD) technique was carried out using a Bruker D8, equipped with a Davinci-Design diffractometer, where a cobalt sealed tube source with $\lambda_{\text{mean}}=1.79026\text{\AA}$ with a step size of 0.02° and a step time of one second over a 2θ range of 45–105° at 40 kV and 30 mA was used in order to identify the phases of bulk materials (MS1 and H13) in all three sets (six different cases).

Microstructural features were characterized using various microscopic techniques including SEM, EBSD, and TEM. The samples were sectioned, polished and then etched using Nital 3% ($3\text{cm}^3 \text{HNO}_3 + 97\text{cm}^3 \text{Ethyl alcohol}$) and Vilella ($1\text{g picric acid}, 4\text{ml HCl}, 95\text{ml Ethyl alcohol}$) solutions for SEM observations. Using a Jeol 6400 scanning electron microscope equipped with a Genesis energy disperse X-ray spectrometer (EDS), the microstructural studies, as well as elemental analysis, were carried out across the interface. The samples were then polished down to $0.05\mu\text{m}$ colloidal silica for EBSD analysis. A Hikari EBSD detector was used to collect EBSD data, which were analyzed and presented as IPF-Z, kernel average misorientation (KAM), and deformed grain maps by employing HKL Channel 5 software. For EBSD maps, the step sizes of 150 and 60nm were selected for low and high magnifications, respectively. Electron transparent samples across the interface were lifted out via focus ion beam milling (FIB) using a FEI Helios NanoLab 650 dual-beam system. The FIB samples were then analyzed by a FEI Tecnai Osiris TEM equipped with a 200 KeV X-FEG gun. Elemental maps were acquired using a Bruker quad-core X-ray energy dispersive spectroscope (XEDS) in the scanning mode of TEM (STEM mode).

The Vickers microhardness of bimetals was measured across the interface using an iMicroTM Nanoindenter made by Nanomechanics Inc. A diamond Berkovich tip with a 50mN load and strain rate of 0.2s^{-1} were set for the microhardness test. Linear measurements were first performed across the interface with the same spacing used for the EDS elemental analyses. Then microhardness measurements were carried out over an area of $210 \times 210\mu\text{m}^2$ covering 8×8 points across the interface in order to obtain microhardness mapping.

Three samples out of each set were machined appropriately for the uniaxial tensile test regarding the ASTM E8M standard. The tests were carried out at a strain rate of $9 \times 10^{-3} \text{s}^{-1}$ using a hydraulic Instron universal testing machine. The results were then plotted as engineering stress-strain curves to better interpret the mechanical behavior of each set.

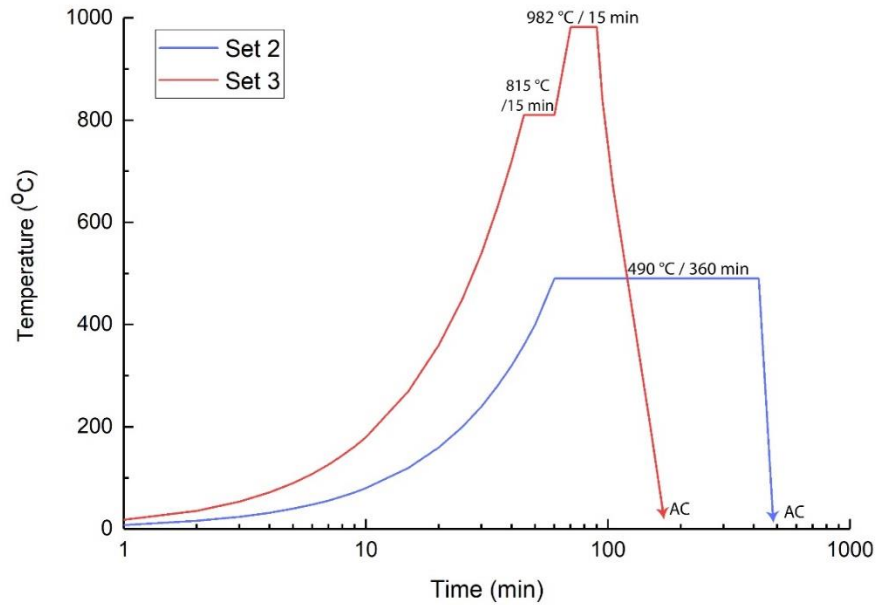


Figure 3-1: Time-temperature profiles of the heat treatment cycles for the MS1-H13 bimetal.

Table 3-1: Chemical composition (wt%) of the substrate-H13 and LPBF-MS1.

MS1 maraging steel									
C	Ni	Co	Mo	Mn, Si	Ti	Al	Cr, Cu	P, S	Fe
≤0.03	17-19	8.5-9.5	4.5-5.2	≤0.1	0.6-0.8	0.05-0.15	≤0.5	≤0.01	Bal.
H13 hot work tool steel									
C	Cr		Mo		Si		V		Fe
0.32-0.45	4.75-5.5		1.1-1.75		0.5-1.25		0.8-1.20		Bal.

3.3 Results

3.3.1 Microstructural modification through heat treatment

Figures 3-2 (a) and (b) exhibit the XRD patterns for different sets of the LPBF-MS1 and substrate-H13, respectively. The aging temperature of 490 °C was found to be optimal for AM 18Ni-300 maraging steel as reported by S. Yin et al. [46]. For set 1, the as-built MS1 was previously reported to be dominated by α' martensite phase [44], while the heat treatments of both sets 2 and 3 developed γ peaks, as shown in Figure 3-2 (a). It was well understood that austenite reversion occurs upon aging the LPBF-maraging steel grade 300, where the volume fraction of austenite raised to six times the value of the as-built condition during aging at 490°C for 5 hours [46]. In set 3, the solution temperature of 982 °C is high enough to trigger austenitization in the LPBF-MS1 since austenite finish temperature is 750 °C, and martensite transformation initiates at 164 °C and finishes at 61°C upon cooling [38]. In maraging steels, the formation of martensite was found to be highly practicable even upon slow cooling from the solution temperature [38], [47].

In Figure 3-2 (b), three peaks representing the BCC ferritic matrix of the substrate-H13 were detected for set 1. Similarly, this pattern was also identified in set 2, where the MS1-H13 bimetal structure was heat treated through the aging treatment. Unlike sets 1 and 2, the substrate-H13 in set 3 involves the martensite phase as a result of rapid cooling from high temperatures. Based on the equilibrium phase diagram, no ferrite exists at 982°C, and the substrate-H13 is fully austenitic, which then transforms to martensite upon quenching [37]. The heat treatment temperature of set 3 is close to the hardening temperature range of H13 hot work tool steel, where the annealed tool steel is fully austenitized and then

transforms to the hard martensitic phase during cooling [40]. In H13, the air cooling was recommended for the hardening treatment particularly for thin cross sections [39]–[41].

Figures 3-3 (a) and (b) clearly indicate identical microstructural features across the interface. For sets 1 and 2, equiaxed coarse grains of the substrate-H13 with a high density of precipitates can be seen. Both sets also share the same morphology in the LPBF-MS1, where the fish scale melt pools were solidified, and circulate flows induced by the Marangoni force are evident as well [24]. In addition, Figures 3-3 (a) and (b) show cellular structure of melt pools consisting of columnar and equiaxed cells.

Figure 3-3 (c) exhibits a totally different microstructure across the interface. In the substrate, lath martensite was formed inside the grains, and the density of precipitates decreased considerably with respect to Figures 3-3 (a) and (b). The LPBF-MS1 no longer shows the melt pools and columnar/equiaxed cells, which were the typical hierarchical solidification morphology of the deposited layer [48]. Instead, lath martensite was developed as a result of solution treatment. This structure was also developed via solution treatment for LPBF-maraging steel 300 [49]. Interestingly, the distinction between the substrate and the LPBF-MS1 can hardly be distinguished. This can be due to the martensitic transformation of both the substrate and LPBF-MS1.

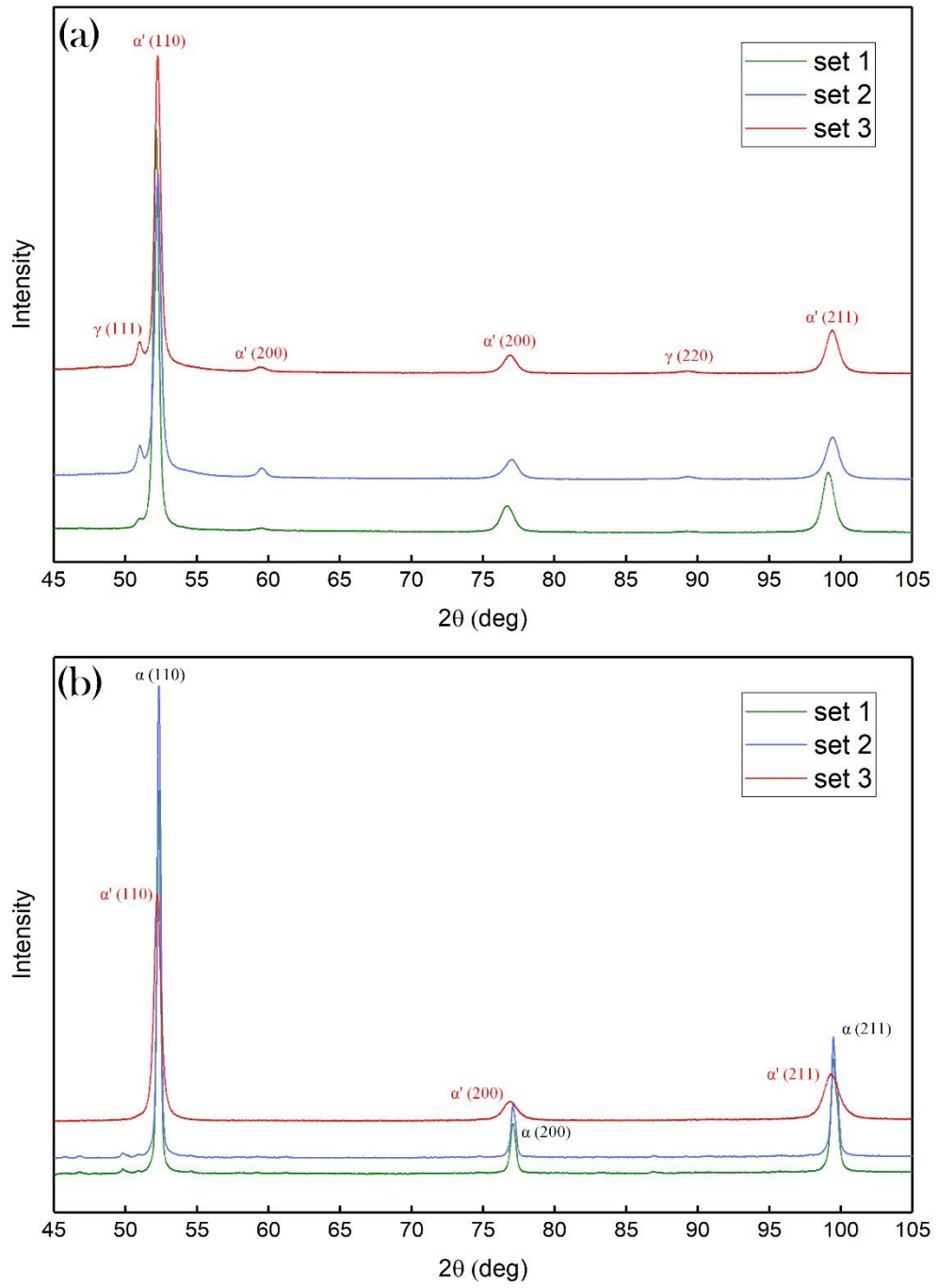


Figure 3-2: X-ray diffraction patterns for (a) LPBF-MS1 and (b) substrate-H13.

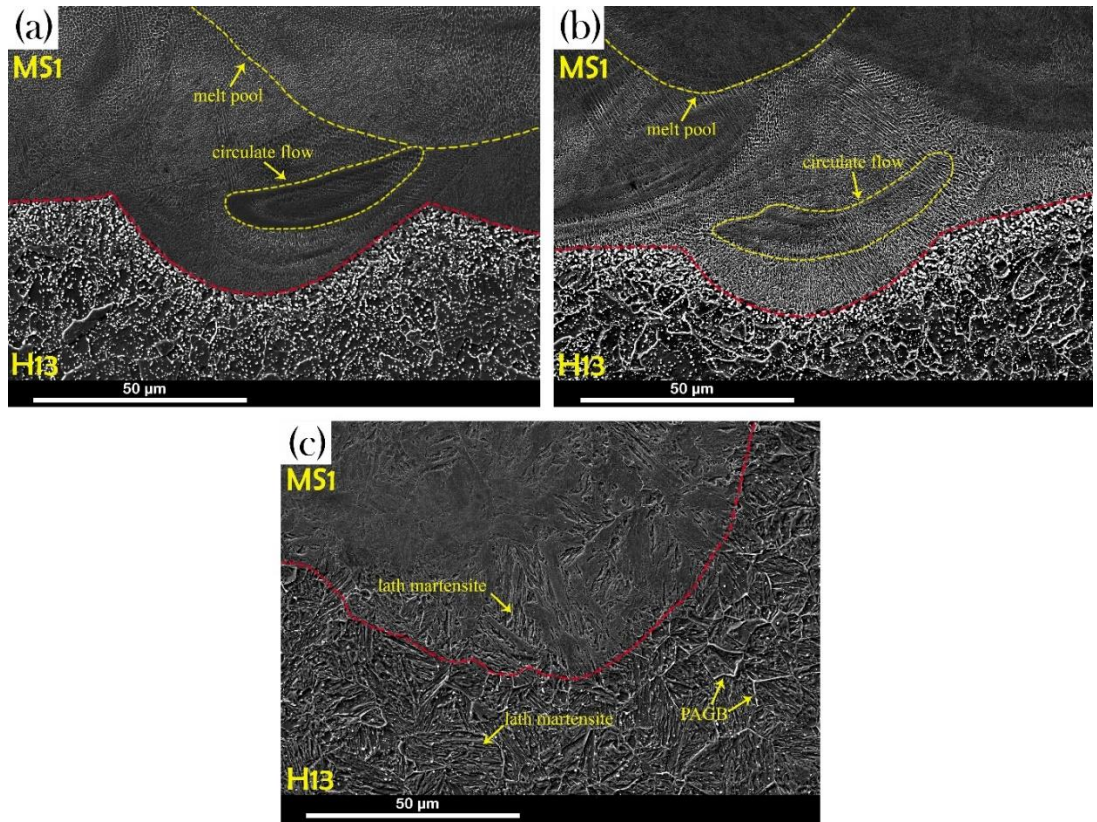


Figure 3-3: Scanning electron micrograph across the interface for (a) set 1, (b) set 2, and (c) set 3.

Figure 3-4 shows IPF-Z, Kernel average misorientation (KAM), and deformed grain mappings across the interface. The latter one represents the internal average misorientation angle (IAMA) within a grain. The deformed regions are defined as grains with more than 1° of IAMA while those grains with IAMA below 1° are identified as recrystallized grains. For the substructured grains, the average angle does not exceed 1° but the subgrain-to-subgrain misorientation angle is above 1° [50]. In set 1, the substrate-H13 contains coarse and equiaxed grains, which are typical of annealed hot work tool steels (see Figure 3-4 (a)). The grain structure on top of the substrate is representative of the LPBF-MS1, where a fine morphology was formed almost along the building direction. Figure 3-4 (b) illustrates the KAM distinction across the as-built interface, which shows intergranular misorientations up to 5° representing lattice distortion related to the density

of dislocations [51]–[53]. As can be seen a high density of local misorientations was detected in the LPBF-MS1. The substrate-H13 shows considerably low misorientation angles, which can be due to annealing treatment prior to the HAM process. The deformed grain mapping shown in Figure 3-4 (c) presents more deformed grains in the LPBF-MS1 showing higher storage energy [54], which is also consistent with the KAM mapping. Likewise, set 2 exhibits identical grain morphology, misorientation angles distribution, and deformed grains to the as-built condition (see Figures 3-4 (d)–(f)). The substrate-H13 is decorated with coarse and equiaxed grains, and a fine grain structure was formed in the LPBF-MS1, where misorientation angles, as well as deformed grains, were accumulated.

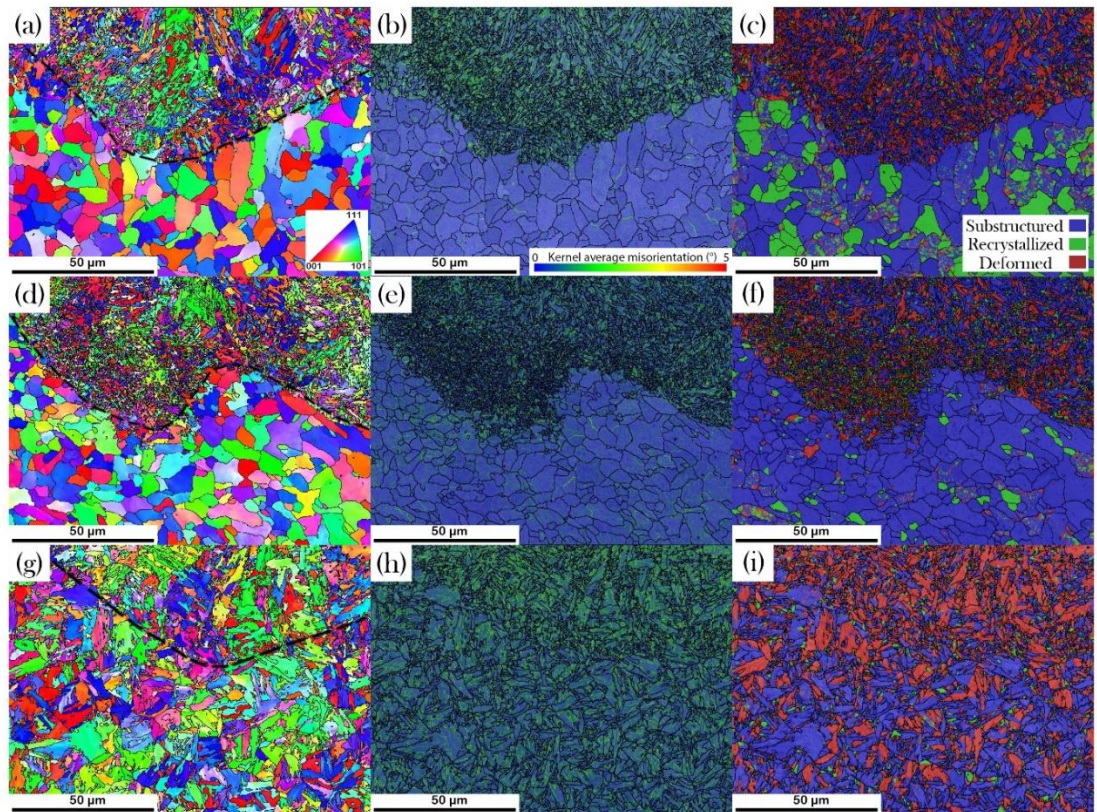


Figure 3-4: Electron back scattered diffraction (EBSD) analyses across the interface for set 1: (a) IPF-Z, (b) KAM, (c) deformed grain; set 2: (d) IPF-Z, (e) KAM, (f) deformed grain; and set 3: (g) IPF-Z, (h) KAM, (i) deformed grain.

As shown in Figure 3-4 (g), the grain morphology of set 3 is obviously different from sets 1 and 2. The H13 grains were filled with transgranular martensite packets leading to a “rougher” morphology than set 1 and 2, as also shown in Figure 3-3. In the LPBF-MS1, the as-built microstructure with fine grains solidified towards the building direction is not present anymore. Instead, there are coarse irregular grains with martensite laths, as well as fine grains, were formed, leading to a relatively identical microstructure to that of the substrate-H13. In other words, the boundary between the substrate and LPBF-MS1 can hardly be distinguishable, and hence the microstructure across the interface is being homogenous. This homogeneity can also be seen in Figure 3-4 (h) with a more uniform distribution of local misorientations. In Figure 3-4 (i), it seems that the LPBF-MS1 is more concentrated in the deformed grains; hence the only difference between the substrate and the LPBF-MS1 is the density of deformed grains. For set 3, the deformed grain mapping shown in Figure 3-4 (i) is of great interest since it highlights the contrast between the substrate-H13 and LPBF-MS1. In addition, the deformed grains were also developed in the substrate as a result of quenching and subsequent martensite transformation resulting in grains with a high density of dislocations.

Figure 3-5 shows the grain size and shape in terms of grain area and aspect ratio based on the EBSD results. In the substrate-H13, the grain size is almost the same for sets 1 and 2 resulting in average grain areas of $19.87\mu\text{m}^2$ and $18.45\mu\text{m}^2$, respectively. However, the grain size is considerably smaller for set 3 with the average grain area of $9.54\mu\text{m}^2$ which is almost half the values of set 1 and 2. In the LPBF-MS1, the average grain sizes are 2.53 and $2.13\mu\text{m}^2$ for sets 1 and 2, respectively. The grain size value of set 3 is considerably higher with an average value of $6.25\mu\text{m}^2$.

The grain shape analysis is illustrated as aspect ratio curves in Figures 3-5 (c)-(d). The aspect ratio (φ) has been conventionally used as a criteria to determine the type of grain shape, where grains with $\varphi > 0.33$ are equiaxed and those with $\varphi \leq 0.33$ are considered as columnar [24]. The dominance of equiaxed grains was observed for the LPBF-MS1 grains. In the as-built condition, 71.14% of grains were developed as equiaxed grains, and the rest solidified as columnar ones. The aging treatment, however, promoted the equiaxed morphology and increased the equiaxed grains up to 82.33%, while the columnar ones reached the minimum value of 17.67% in set 2. The solution treatment did not have a significant influence on the grain shape of LPBF-MS1 since the equiaxed and columnar grains were recorded as 75.92% and 24.08%, respectively. Overall, the substrate is dominated by equiaxed grains; however, the extent of the equiaxed grains varies from set 1 and 2, to set 3. The substrate-H13 contains 90.17% equiaxed and 9.83% columnar grains in the as-built condition. The aging treatment did not considerably affect the grain shape of the substrate since 93.01% equiaxed and 6.99% columnar grains were measured for set 2. Upon solution treatment, the portion of columnar grains reached 24.88% and subsequently, the equiaxed grains decreased to 75.12%. The increase of columnar morphology can be related to the ferrite-martensite transformation of the substrate in which equiaxed ferrite grains transform to martensite laths.

Figure 3-6 presents a high magnification EBSD of set 3 across the interface. The homogeneity of grain structure can be observed clearly in Figure 3-6 (a) with martensite laths on either side of the interface. In the LPBF-MS1, the grains still inclined towards the building direction. The low angle (LA) and high angle (HA) grain boundaries were distributed almost uniformly across the interface (see Figure 3-6 (b)). The KAM map also

reveals identical results with homogenous distribution of misorientation angles across the interface (see Figure 3-6 (c)). LA-HA grain boundary and KAM mappings look very much alike, attributing low angle grain boundaries to the high angle misorientations.

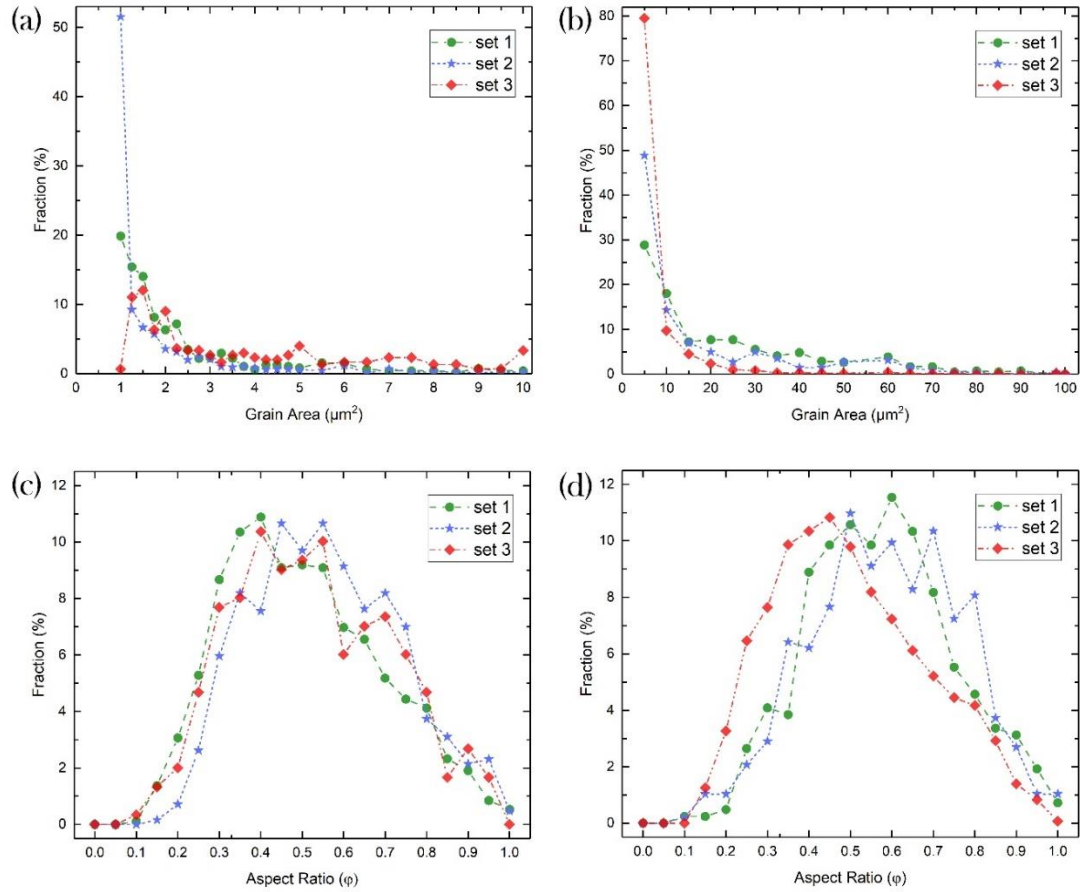


Figure 3-5: Grain area distribution for (a) LPBF-MS1, (b) substrate-H13, and grain shape aspect ratio for (c) LPBF-MS1, (d) substrate-H13.

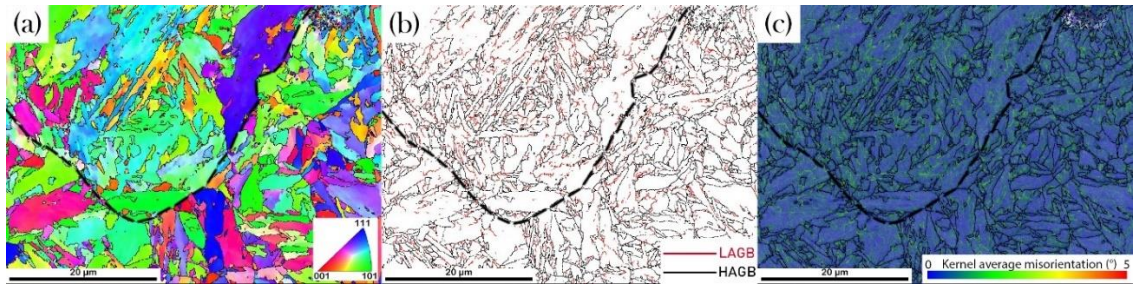


Figure 3-6: High magnification EBSD analyses across the interface in set 3 (a) IPF-Z, (b) LA-HA grain boundaries, and (c) KAM.

Results from elemental distribution using EDS across the interface is plotted in Figures 3-7 (a)-(d). Ni, Co and Cr, Si were selected as the LPBF-MS1 and substrate-H13 alloying elements, respectively. For all sets, the substrate-H13 involves a negligible amount of Ni and Co (Figures 3-7 (a)-(b)), whereas the substrate alloying elements (Cr, Si) reached the highest values for each set (Figures 3-7 (c)-(d)). It seems that, the heat treatment cycles did not affect the distribution of the alloying elements in the substrate; however, set 3 contains more Cr than sets 1 and 2 since the solution treatment caused the dissolution of H13 precipitates and hence the enrichment of H13 matrix in Cr. Since the solution treatment was carried out at high temperatures, the lower concentration of Cr in the LPBF-MS1 can be as a result of outward diffusion from the interface to the bulk MS1 material.

The alloying elements vary substantially in the LPBF-MS1. Two opposing trends have been previously reported for the MS1-H13 bimetal [24]. Likewise, sets 2 and 3 experienced the increase of the LPBF-MS1 alloying elements and the decline of the substrate-H13 alloying elements. In Figures 3-7 (a)-(b), the compositional gradients of set 2 are almost overlapped with that of set 1, except within the first deposited layer with 80 μ m thickness where Ni and Co decrease considerably. This can be due to short-range diffusion of Ni and Co during the aging treatment leading to depletion of the first deposited melt pools from the LPBF-MS1 alloying elements. Overlapping of sets 1 and 2 can also be observed in Cr and Si variations from the interface (see Figures 3-7 (c)-(d)). For set 3, the LPBF-MS1 is more enriched in Ni and Co, while less concentrated in Cr and Si than sets 1 and 2. The higher content of the MS1 alloying elements accompanied by the lower

amount of the substrate-H13 alloying elements obviously indicates atomic diffusion at high temperature during solution treatment.

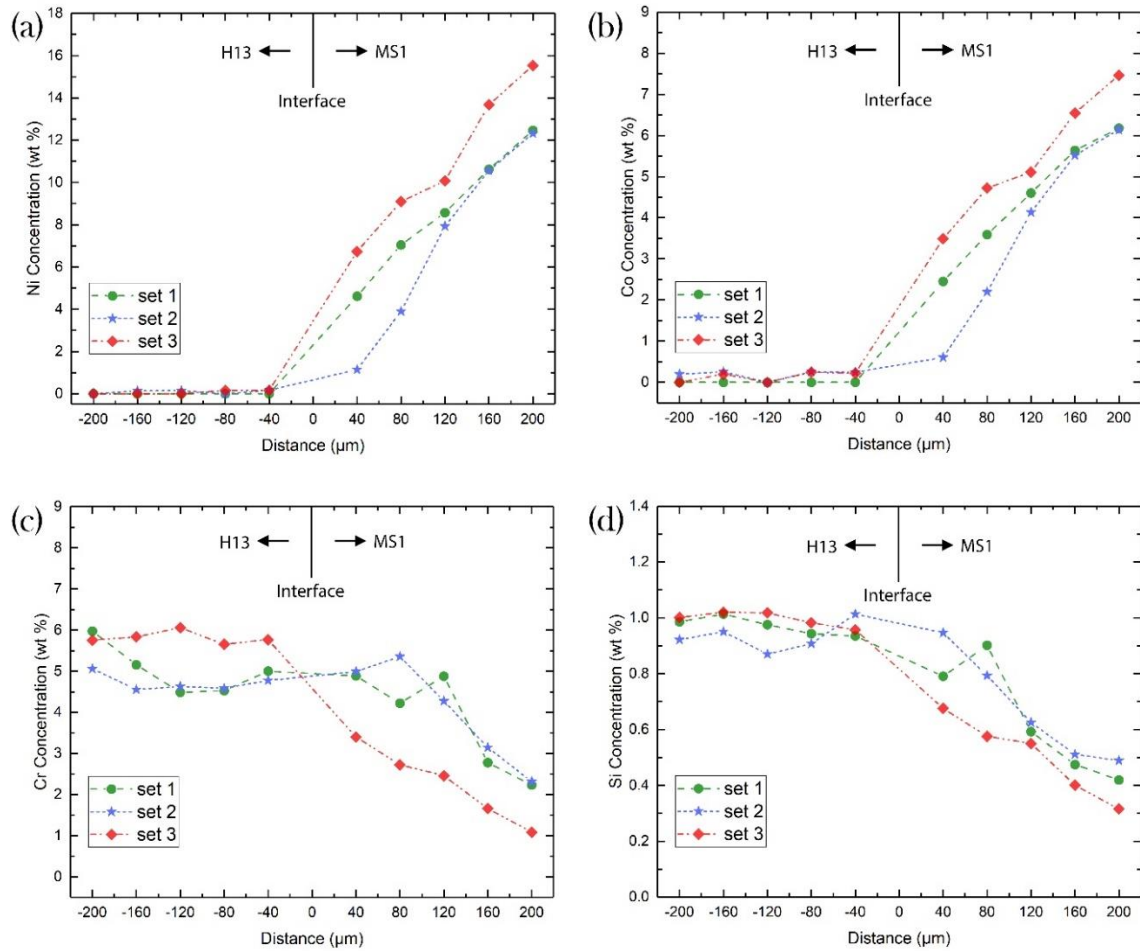


Figure 3-7: Alloying elements distribution across the interface (a) Ni, (b) Co, (c) Cr, and (d) Si.

As mentioned earlier, the MS1-H13 bimetal structure contains a very narrow interface along with fine microstructure on the printed side of the as-built sample or either side of the heat-treated one. Thus, it necessitates deeper analysis via TEM. In this regard, the FIB technique was used to extract a thin layer from set 3, as shown in Figure 3-8 (a). In Figure 3-8 (b), the bright field micrograph of set 3 obviously shows the martensite lath in H13 and relatively finer microstructure of the LPBF-MS1. The EDS analysis was then

conducted to depict qualitative elemental maps from the substrate-H13 as well as the interface. Figure 3-9 presents dissolution of Cr-rich carbides after solution treatment and consequently the decrease of globular precipitates. This confirms the higher Cr content detected in the substrate-H13 of set 3 as shown in Figure 3-7 (c). Figure 3-10 evidently shows alloying elements distribution after the heat treatment on set 3. On the substrate side, coarse globular carbides were formed, which are rich in Cr, Mo, and V. These precipitates were previously mentioned for the as-built H13 [24]; however, no precipitates exist in the LPBF-MS1. There is a relatively uniform distribution of Fe, Ni, and Co elements without elemental segregation in the LPBF-MS1. Thus, the typical cellular solidification structure of the as-built condition [24] was almost removed as a result of the solution treatment.

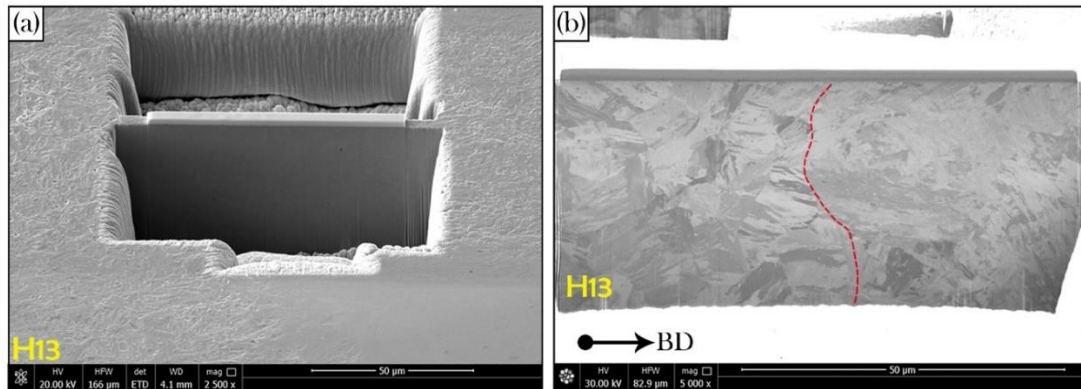


Figure 3-8: (a) FIB lifted out location the MS1-H13 interface in set 3, and (b) secondary ion image of the cross section of the FIB sample before lifting out.

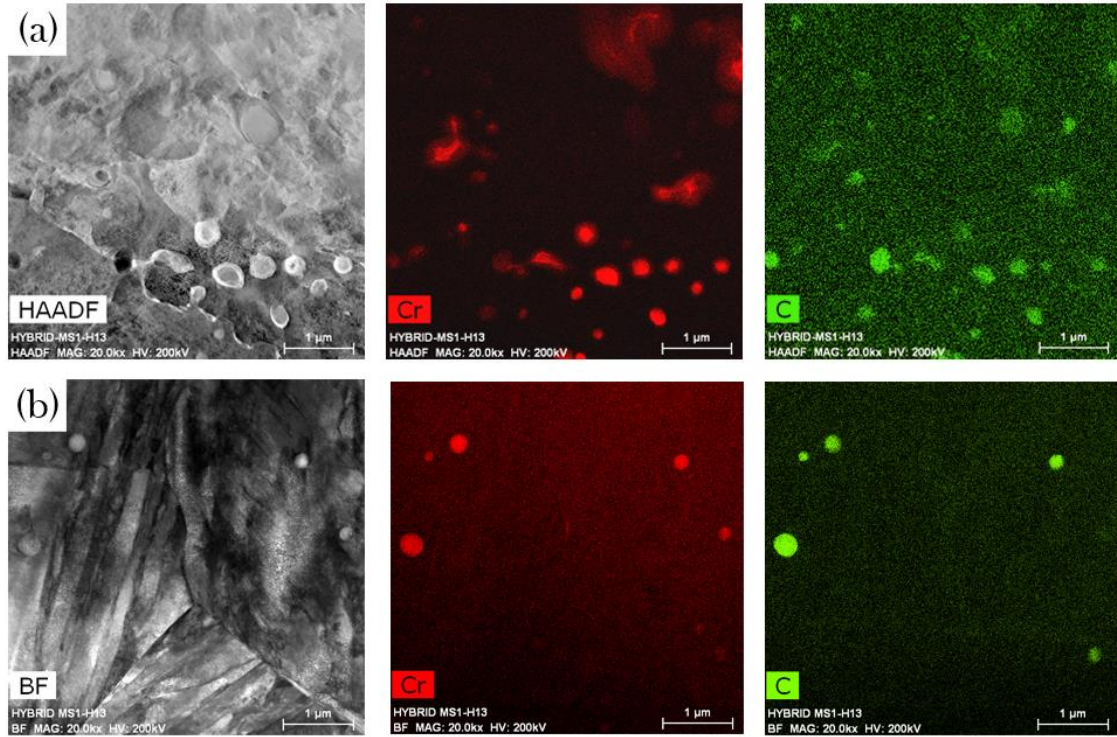


Figure 3-9: XEDS elemental maps of the substrate-H13, showing Cr and C maps (a) set 1, and (b) set 3.

3.3.2 Mechanical enhancement of modified MS1-H13 bimetal structure

The variations of microhardness through the hybrid samples are illustrated in Figure 3-11. Regarding sets 1 and 2, the substrate-H13 is clearly low in hardness with the average values of 230 and 200HV; respectively, these values increase to 332 and 569HV right at the interface, where the impact of the indenter is equally located between the substrate and printed side. The hardness peaks in the LPBF-MS1 adjacent to the interface, where the first powder layer was deposited on the substrate. The hardness peak was measured to be 692 and 857HV for sets 1 and 2, respectively. By moving further from the interface, the hardness values of both sets 1 and 2 decreases smoothly and finally reach the corresponding values of MS1.

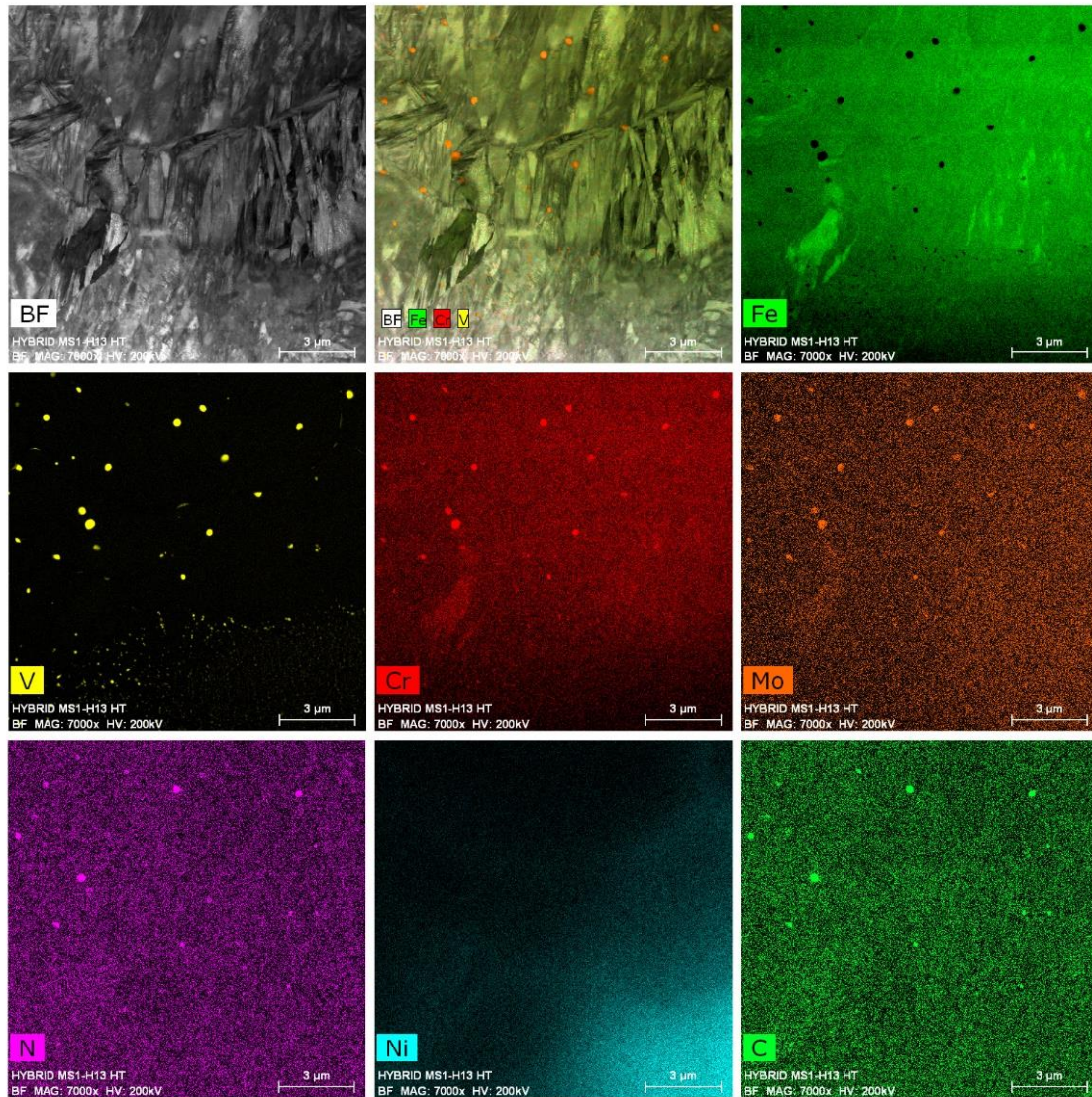


Figure 3-10: Energy dispersive spectroscopy (EDS) elemental maps across the interface in set 3.

As mentioned above, an abrupt variation of hardness from the substrate to MS1 is clearly evident in sets 1 and 2; however, this trend can no longer be seen in set 3. It seems that upon solution treatment, the hardness of the substrate reached the average value of 600HV, three times the corresponding values for sets 1 and 2. By approaching the interface, the hardness profile follows almost the same trend as set 1. Indeed, a uniform hardness profile across the interface was formed in set 3, and the stereotype hardness gradient of set

1 and 2 was removed via solution heat treatment. Despite this uniform hardness gradient, the hardness of MS1 reduced slightly with respect to sets 1 and 2 during the solution treatment. As already stated in Figure 3-5, the LPBF-MS1 in set 3 contains average grain area of $6.25\mu\text{m}^2$, while sets 1 and 2 involve MS1 grains with the average areas of 2.53 and $2.13\mu\text{m}^2$, respectively. Thus, the hardness reduction in the solution-treated MS1 can be attributed to the coarser grains as a result of Hall-Petch effect.

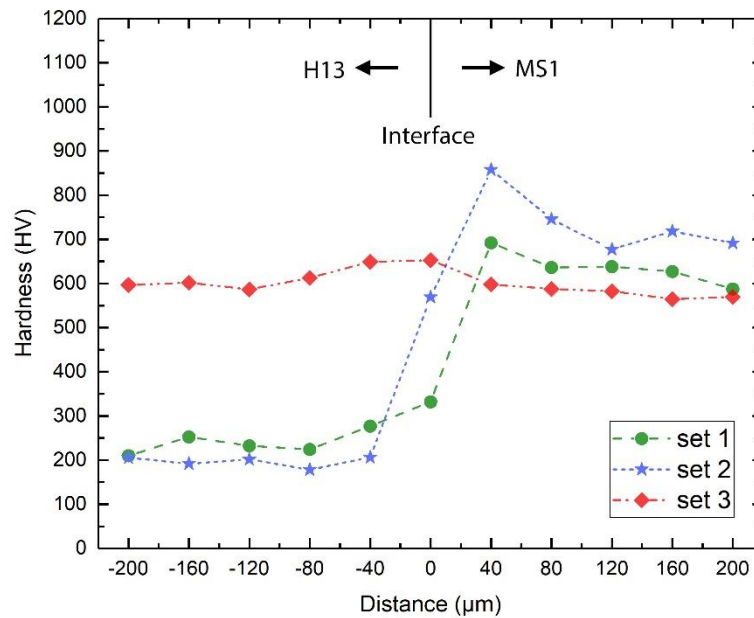


Figure 3-11: Vickers microhardness variations across the interface for sets 1, 2, and 3.

Figure 3-12 shows the nanoindentation mapping as a qualitative illustration of hardness across the interface. For sets 1 and 2, the hardness distinction between both sides is presented in Figures 3-12 (a) and (b), where the blue area of soft substrate turns to the green interface and followed by a hardness increment to the maximum value in red. Herein, the hardness pattern is well consistent with the hardness variation of Figure 3-11 and graphically highlights the position of the interface. The hardness distinction across the interface was also reported for the selective laser melted aluminum bimetal structure [20].

For set 3, the hardness mapping seems totally different from the two other samples, where the interface is no longer distinguishable. Instead, a uniform pattern can be observed, implying homogenous hardness leveled off across the interface. Thus, the process-induced hardness gradient was removed via solution treatment.

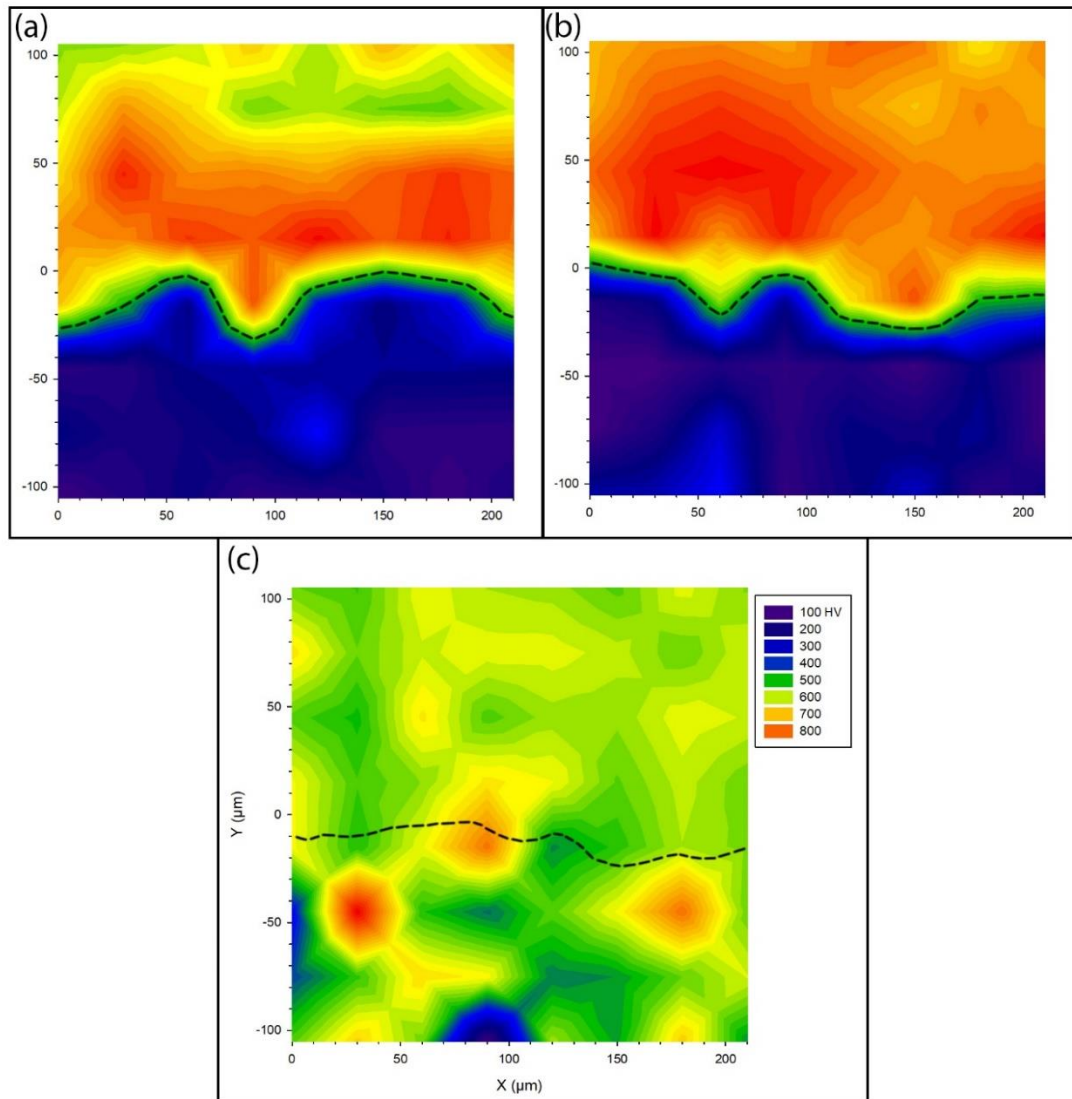


Figure 3-12: Two-dimensional (2D) microhardness gradient across the interface for (a) set 1, (b) set 2, and (c) set 3.

The mechanical behavior of hybrid samples under uniaxial tensile loading is shown in Figure 3-13 (a). In the as-built condition, the hybrid sample exhibited a ductile behavior with relatively low strength, having an ultimate tensile strength of 664MPa and fracture elongation of 23%. The stress-strain curve of set 2 overlaps set 1 showing almost the same yield strength, UTS, elongation, and fracture strength, as indicated in Table 3-2. Both sets 1 and 2 failed in the substrate-H13 away from the interface indicating low strength of the substrate (see Figure 3-13 (b)). Therefore, the aging treatment applied on set 2 seemed not to affect the mechanical properties. However, Set 3 behaved differently from sets 1 and 2. This sample seemed to be strengthened considerably since the yield strength, UTS, and fracture strength are three times as high as the corresponding values of sets 1 and 2. Predictably, ductility dropped at the expense of higher strength, which is consistent with 5.4% elongation measured in set 3. In set 3, fracture occurred at the interface with relatively low necking as shown in Figure 3-13 (b).

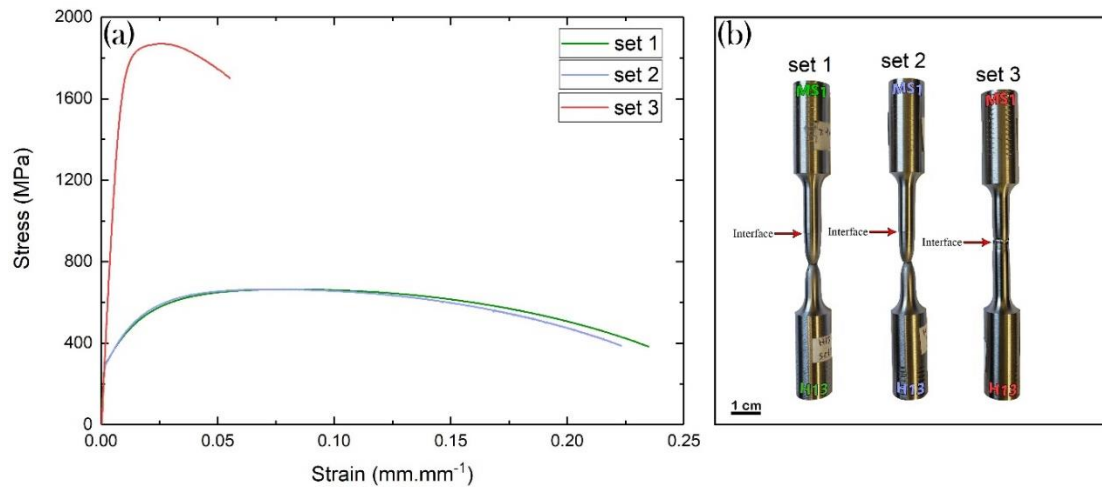


Figure 3-13: a) Engineering stress-strain curves for sets 1, 2, and 3 b) fracture samples after tensile test

Table 3-2: Mechanical properties under uniaxial tensile test

	Yield strength (MPa)	UTS (MPa)	Elongation %	Fracture strength (MPa)
Set 1	566.0	664.2	23.3	386.6
Set 2	566.5	666.1	22.8	385.7
Set 3	1864.0	1865.6	5.4	1700.8

3.4 Discussion

The results represent a modification method for hybrid additively manufactured MS1-H13 bimetals through the solution treatment whilst the aging treatment was not successful in this regard. The modification method here means that the microstructural features were united across the interface; in other words, the interface was removed. Herein, the effective heat treatment of set 3 (optimum procedure) is discussed further in order to shed light on the interface removal mechanism, as detailed below.

In brief, the as-built MS1-H13 sample involves a ferritic matrix with large equiaxed grains supersaturated with globular precipitates on the H13 side (see Figure 3-3 (a)). Since the substrate was in a fully annealed condition, no considerable in-grain misorientation was observed. On the LPBF-MS1 side, very fine martensitic grains were solidified, accommodating the equiaxed and columnar solidification cells along the building direction. Despite the substrate-H13, the in-grain misorientation were high in density, indicating a high density of dislocations stored in the LPBF-MS1 (see Figures 3-4 (a)-(c)). Identical characteristics were also observed for set 2 (see Figures 3-4 (a) and (d)), although microstructural changes were expected as a result of the dissolution of solidification cells during heat treatment of maraging steel [46]. The LPBF-MS1 was enriched in the substrate alloying elements (see Figure 3-7); thus, more thermal energy was required for the diffusion of elements to accomplish dissolution. The aging treatment was also thermally

insufficient to cause grain modification in the substrate-H13; hence the aging treatment was inadequate for modification of MS1-H13 bimetal structures.

The microstructural features associated with set 3 include a fully martensitic substrate with visible prior austenite grain boundaries and transgranular martensite laths diluted in precipitates (see Figure 3-3 (c)). The substrate was higher in strain energy as compared to the as-built condition. The LPBF-MS1 changed extremely as it was composed of coarse martensite laths with no trace of cellular solidification structures. The solution treatment thoroughly homogenized the microstructure since a rough martensitic structure was developed across the interface, which contains a uniform distribution of strain energy (see Figures 3-4 (g)-(i)). The solution treatment for MS1 acts as the hardening treatment for H13 tool steel; thus the hardening phenomenon occurs in the substrate-H13, where the matrix transforms to austenite, and the precipitates were considerably dissolved forming a rich solid solution. The austenitized H13 is then quenched to provoke martensite transformation. Although the nucleation and growth of martensite require thermal activation, the energy barrier was very negligible. In other words, a diffusionless transformation from FCC to BCT structure occurred via rapid cooling [55]. According to EDS analyses, no elemental diffusion occurred in the substrate-H13, and the only possible phase transformation is the formation of martensite laths accompanied by partial dissolution of carbides (see Figure 3-9). This morphology was well studied in the as-quenched H13 tool steel as well as low alloy steel [37], [56].

The solution treatment led to unprecedented changes to the solidification morphology and grain structure of the LPBF-MS1 side. The most prominent evolution can be attributed to the dissolution of the cellular structure shown in Figure 3-10, where cells

dissipated through elemental diffusion. As stated earlier [24], enrichment of alloying elements in the inter-dendritic regions together with the depletion of alloying elements inside the dendrites is a phenomenon called microsegregation, which evolves a cellular solidification structure. However, this structure can be eliminated via diffusion at high temperatures when the atomic movement lower compositional gradient across solidification cells [57]. The dissolution of the cellular structure at 980°C was also reported by Conde et al. [38], who studied the heat treatment of LPBF-maraging steel. Moreover, the prior as-built grains of LPBF-MS1 (see Figure 3-4 (a)) commence growing since the solution temperature is noticeably high enough to cause grain growth, and the coarser grain structure was developed upon air cooling to room temperature (see Figure 3-4 (g)). Grain growth prevalently occurred during solution treating of either conventional or additively manufactured maraging steel [42], [58]. The other consequence of solution treatment is the evolution of lath-like martensite similar to the martensitic morphology of the substrate-H13 mentioned above. Therefore, the microstructure was homogenized across the interface during solution treatment resulting in the dissipation of the interface between both sides (see Figure 3-6). The grain size difference between both sides was calculated for set 1 and set 3 to indicate microstructural homogeneity across the interface, and presented as the percentage of grain area difference. The percentage of grain area difference can be defined as follows:

$$\text{grain area difference (\%)} = \frac{A_{H13} - A_{MS1}}{A_{H13}} \times 100$$

where A_{H13} , and A_{MS1} are the grain area of the substrate-H13 and LPBF-MS1, respectively. The grain area difference decreased from 87.27% for set 1 to 34.49% for set

3 indicating the homogeneity of microstructure quantitatively upon solution treatment. The microstructural evolution directly affected the mechanical behavior of MS1-H13 bimetals. Previously, we reported a remarkable discrepancy in hardness values between the substrate-H13 and LPBF-MS1 in the as-built condition. This event was also observed for the aged MS1-H13 interface since the substrate-H13 and LPBF-MS1 preserved their prior morphologies. In spite of that, the hardness gradient dissipated after solution treatment, and a smooth variation was developed across the interface. This is apparently due to martensite transformation and hence hardness escalation in the substrate-H13 resulting in hardness homogenization for set 3. The uniaxial tensile loading better presented the improvement of mechanical properties, as shown in Figure 3-13 and Table 3-2. Accordingly, the distinct consequence of solution treatment is the increase of strength as well as hardness for the MS1-H13 bimetal.

The solution treatment certainly promoted several strengthening mechanisms due to microstructural changes across the interface. The distinct evolution of martensite definitely plays a leading role in strengthening the MS1-H13 bimetal. On the substrate side, martensite transformation strikingly reduced the grain size due to the hierarchical structure of lath martensite [59], [60]; thus, Hall-Petch effect should be considered as an effective strengthening mechanism. Besides, a great number of dislocations were generated within the matrix (see e.g. Figure 3-4 (h), and 3-6 (c)). This would later encourage more interaction of dislocations and hence more work hardening under tensile loading resulting in higher strength and hardness. This strengthening mechanism is called forest dislocation hardening in martensitic steels [61], [62]. Additionally, few globular precipitates still remain in the substrate after solution treatment (see Figures 9 and 10), which further

interact with dislocations promoting the Orowan strengthening mechanism. On the LPBF-MS1, the lath martensitic structure (Hall-Petch effect), as well as forest hardening, contribute to the hardening of set 3. Despite the substrate-H13, no precipitation was observed, and the Orowan mechanism is no longer valid in the LPBF-MS1 (see Figure 3-10). This can be the reason for the lower hardness of LPBF-MS1, leading to subsequent failure under tensile loading.

The grain size measurement showed a relatively identical grain area of $9.54\mu\text{m}^2$ and $6.25\mu\text{m}^2$ for the substrate and LPBF-MS1, respectively. In addition, the density of dislocations tends to be uniform after solution treatment, as shown in Figures 3-4 (h) and 3-6 (c). For the substrate-H13 and LPBF-MS1, the grain boundary strengthening together with forest dislocation hardening made correspondingly the same contribution to the strength of MS1-H13 bimetal. As a consequence, both the substrate and LPBF-MS1 shared almost an identical microstructure due to the homogenization effect of solution treatment; and finally, the corresponding strengthening mechanisms contributed to the enhancement of mechanical properties in the MS1-H13 bimetal.

3.5 Conclusions

The current study was made to manipulate the microstructural features purposely across the additively manufactured MS1-H13 interface in order to erase the microstructural contrast between the two materials. Using heat treatment techniques, the maraging steel (MS1) solution treatment was determined as an effective method to accomplish this goal. The following outcomes can be concluded:

- In the as-built condition, the coarse morphology of the substrate-H13 together with ultrafine grains of the LPBF-MS1 resulted in an inhomogeneous microstructure across the interface;
- The aging treatment was not successful in provoking microstructural evolution across the interface since the aging temperature was lower than the austenitization temperature for the substrate-H13;
- The solution treatment effectively reconstructed the microstructure across the interface via encouraging martensite transformation on either side of the interface. Interestingly, the MS1 solution treatment stimulated a hardening treatment for the substrate-H13;
- A lath martensitic structure was evolved across the interface, uniting the phase, grain size, and even dislocation density between the substrate-H13 and LPBF-MS1;
- Despite the as-built condition, a well-balanced hardness profile was generated across the interface upon the solution treatment due to the martensite transformation and uniform distribution of dislocation density;
- The solution-treated bimetal structure gained substantial yield strength, UTS, and fracture strength, nearly triple that of the as-built condition. The martensite transformation, along with forest dislocation hardening were determined as the main contributors to the strength and mechanical properties.

3.6 References

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4 Microstructure control of hybrid additively manufactured 18Ni300-H13 bimetals by sequential heat treatments

4.1 Introduction

Additive manufacturing (AM) has been recently considered advanced manufacturing for innovative engineering applications. This well-known 3D-based printing method has become a game-changer in many manufacturing industries [1], [2]. AM techniques ease manufacturing intricate geometries, rapid production of metallic parts, and versatility of feedstock materials [3]–[5]. In addition, multi-material components have been well developed via additive manufacturing of functionally graded material, composite structure, and bimetal [6], where the latter one was frequently fabricated using the direct energy deposition (DED) and laser powder bed fusion (LPBF) methods [7]–[9].

Maraging steels have been the prevalent engineering material in additive manufacturing (AM) due to their compatibility with the AM processes, superior mechanical properties, and extensive applications [10]–[13]. Maraging steels have been recently employed to produce bimetallic structures. Many maraging steel bimetallic structures were successfully deposited on dissimilar materials like copper and tool steel [14], [15]. They showed excellent bonding with the substrate as well as good mechanical strength. The potential applications of maraging steel bimetals can be manufacturing a new generation of tools and dies with interior cooling channels and onshore repair of components in shipbuilding industries [16].

Although maraging steel bimetals have exhibited many remarkable features, they suffer from extreme microstructural and mechanical properties variation across the

bimetallic interfaces. For example, in the as-built maraging steel bimetals, the grain sizes, hardness values, and phase types are incredibly different across the interface [15], [17]. This would cause weak resistance to plastic deformation under uniaxial tensile loading and, subsequently, poor performance in service conditions [18]–[22]. Since additively manufactured maraging steels can also be heat treated to enhance the mechanical strength [23], the heat treatment of maraging steel bimetals can be regarded as a practical solution to conquer this deficiency. However, there is a lack of literature on heat treatment of maraging steel bimetallic systems produced using AM. Therefore, this study investigates the effect of heat treating sequences and proposes the optimum sequence for the 18Ni300-H13 bimetallic structure.

1. Methodology

Virgin maraging steel 18Ni300 powder with particle diameters of 15-45 μm was printed on an annealed H13 tool steel plate using an EOS M290 printing machine. The SLM process was carried out with the process parameters shown in Table 4-1. The as-built 18Ni300-H13 bimetal was heat treated using two heat treatments sequences, as indicated in Figure 4-1 (a). Each heat treatment sequence involves the aging treatment of 18Ni300 and the hardening treatment of H13. The aging treatment was performed at 480 °C for 6 hours followed by air cooling to room temperature. The solution treatment was carried out by preheating at 815 °C for 15 min and then heating at 982 °C for 1 hour with air cooling to ambient temperature. This work defines the two heat treatment sequences as HT-I and HT-II. The HT-I sequence was first started with the aging treatment and then followed by the solution treatment, whereas the HT-II sequence involves the opposite order. The samples were then polished and etched using Modified Fry's reagent and Vilella solution

for primary microstructural study. The optical micrographs were captured using a Zeta-20 optical microscope. The electron backscatter diffraction (EBSD) data was also collected via a Thermo Fisher Scios 2 dual-beam scanning electron microscope equipped with an Oxford Symmetry EBSD detector. A step size of 0.20 μm and a scan area of $200 \times 140 \mu\text{m}^2$ were set for EBSD data acquisition. The EBSD data was then processed through Channel 5 software, and pole figure, inverse pole figure (IPF), and kernel average misorientation (KAM) were extracted. The uniaxial tensile tests were carried out based on the ASTM E8M standard [24] at a strain rate of $9 \times 10^{-3} \text{ s}^{-1}$. The tensile tests were repeated three times, and the mean values and standard deviations were measured. The microhardness measurements were performed using iMicroTM Nanoindenter. A diamond indenter tip with a 50mN load was applied at a strain rate of 0.2 s^{-1} , and the microhardness tests were carried out over 9×9 points of $320 \times 320 \mu\text{m}^2$ area across the 18Ni300-H13 interface. The hardness values were reported in Vickers microhardness (HV). The detailed specification of all instruments is also presented in Table 4-2.

Table 4-1: SLM process parameters

Powder layer thickness	Laser type	Laser spot size	Maximum laser power	Chamber atmosphere
40 μm	Yb-fibre	100 μm	400 W	99.9 % Ar gas

Table 4-2: Specification of equipment and instruments

	Printing Machine	Optical Microscope	SEM Microscope	EBSD Detector	Microhardness Tester
Model	EOS M290	Zeta-20	Scios 2 dual-beam	Symmetry	iMicroTM
Company	EOS	KLA	Thermo Fisher	Oxford Instrument	Nanomechanics
Origin	USA	USA	USA	UK	USA

4.2 Results and Discussion

Figure 4-1 (b) shows the XRD pattern of both bulk materials in the as-built and heat-treated conditions. Both heat treatment sequences caused martensite transformation in the H13 side, and the 18Ni300 preserved its martensitic structure. However, a negligible fraction of austenite was developed in the 18Ni300 side. Figures 4-1 (c)-(e) indicate the formation of martensite laths on both sides of the interface after the HT-I and HT-II sequences. The development of the martensite structure is mainly due to the hardening cycle at the elevated temperature of 982 °C, where both sides are austenitized and followed by martensite transformation upon cooling. The microstructure evolution and phase transformation look similar in both the HT-I and HT-II samples.

Figure 4-2 provides the porosity distribution from the interface to the 18Ni300 side, showing the porosity level along the building direction. The as-built sample had the maximum porosity of 0.1%, and the HT-I and HT-II samples contained 0.065 % and 0.69 %, respectively. It seems that the heat treatment sequences were effective in decreasing the porosity on the 18Ni300 side. In addition, the average density increased from 93.05 % to 95.00 % upon the heat treatment sequences. Thus, the SLM process and the subsequent heat treatments were reliable to produce a highly dense 18Ni300 maraging steel.

The inverse pole figure (BD-IPF) map for the as-built sample on a plane parallel to the build direction is presented in Figure 4-3 (a). The map illustrates the grain morphology across the as-built bimetal interface, where the ultrafine grains of 18Ni300 with $3.39 \mu m^2$ grain area were deposited on the H13 tool steel with $23.33 \mu m^2$ grain area, resulting in an uneven grain morphology across the interface. The variation of grain size from H13

to 18Ni300 indicates a microstructural inhomogeneity. This feature was observed in other as-built AM bimetals [20]–[22]. In Figures 4-3 (b) and (c), both heat treatment sequences led to a similar grain morphology across the interface with almost identical grain sizes for the 18Ni300 and H13 sides (see Table 4-3). In other words, the grain size difference between the 18Ni300 and H13 steels was removed, and the interface was less distinguishable after heat treatment sequences.

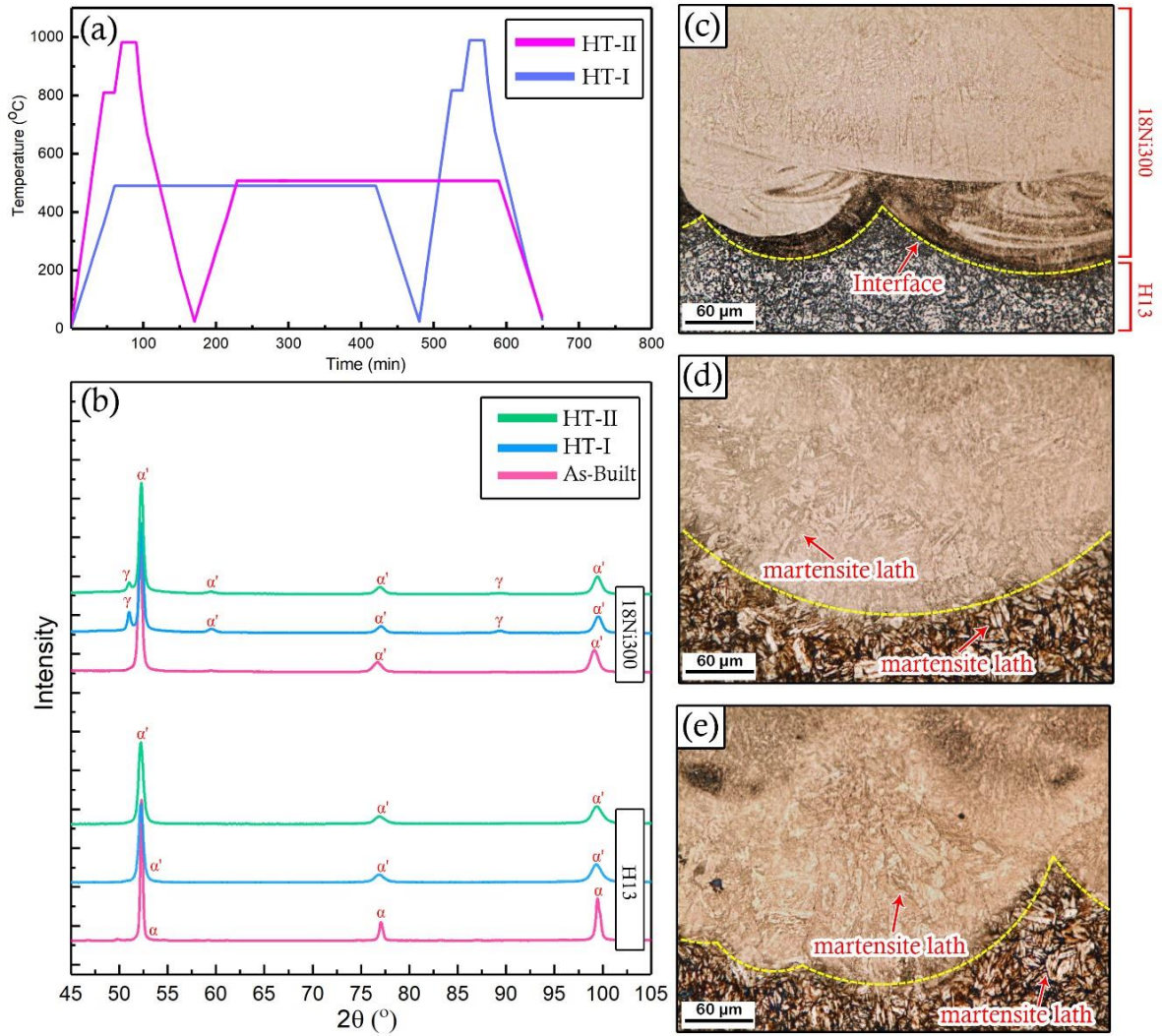


Figure 4-1: a) The profile of heat treatment sequences b) XRD patterns of 18Ni300 and H13; optical micrographs of c) as-built d) HT-I e) HT-II

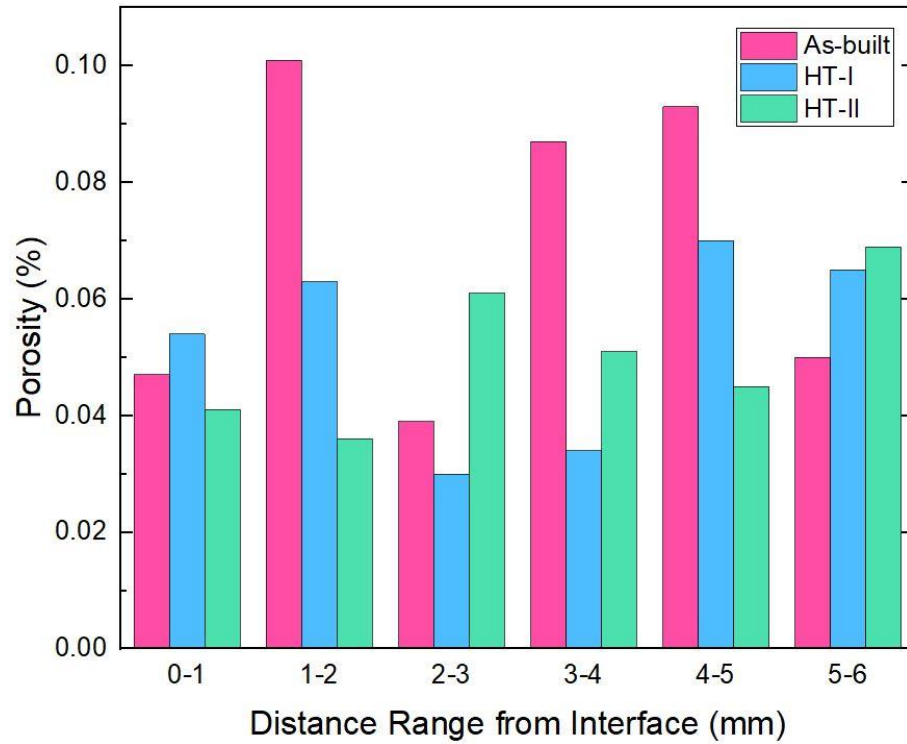


Figure 4-2: Porosity level from the interface to the 18Ni300 side.

The parent austenite grains (PAGs) of heat-treated samples, as well as the as-built 18Ni300 side, were reconstructed in Figures 4-3 (d)-(f). This reconstruction was carried out with the technique developed by Gomes et al. [25], which is based on a random walk clustering method analyzing groups of closely related martensitic variants based on their angular deviation from the governing orientation relationship between austenite and martensite. The PAG morphology of the as-built 18Ni300 side comprises columnar grains due to directional solidification during laser melting. This morphology is different from the PAGs of heat-treated samples, which mainly consist of nearly equiaxed grains (see Figures 4-3 (e) and (f)). This implies that the hardening treatment changed the morphology of the PAGs from columnar to an equiaxed structure. Therefore, the equiaxed grains were formed

in both 18Ni300 and H13 sides at high temperatures, which then transformed to identical martensitic morphology and a homogeneous grain morphology across the interface. HT-II seems to have coarser PAGs with respect to HT-I since the higher hardening temperature can result in larger austenite grains in the HT-II sample. This might affect the mechanical properties and will be discussed later on.

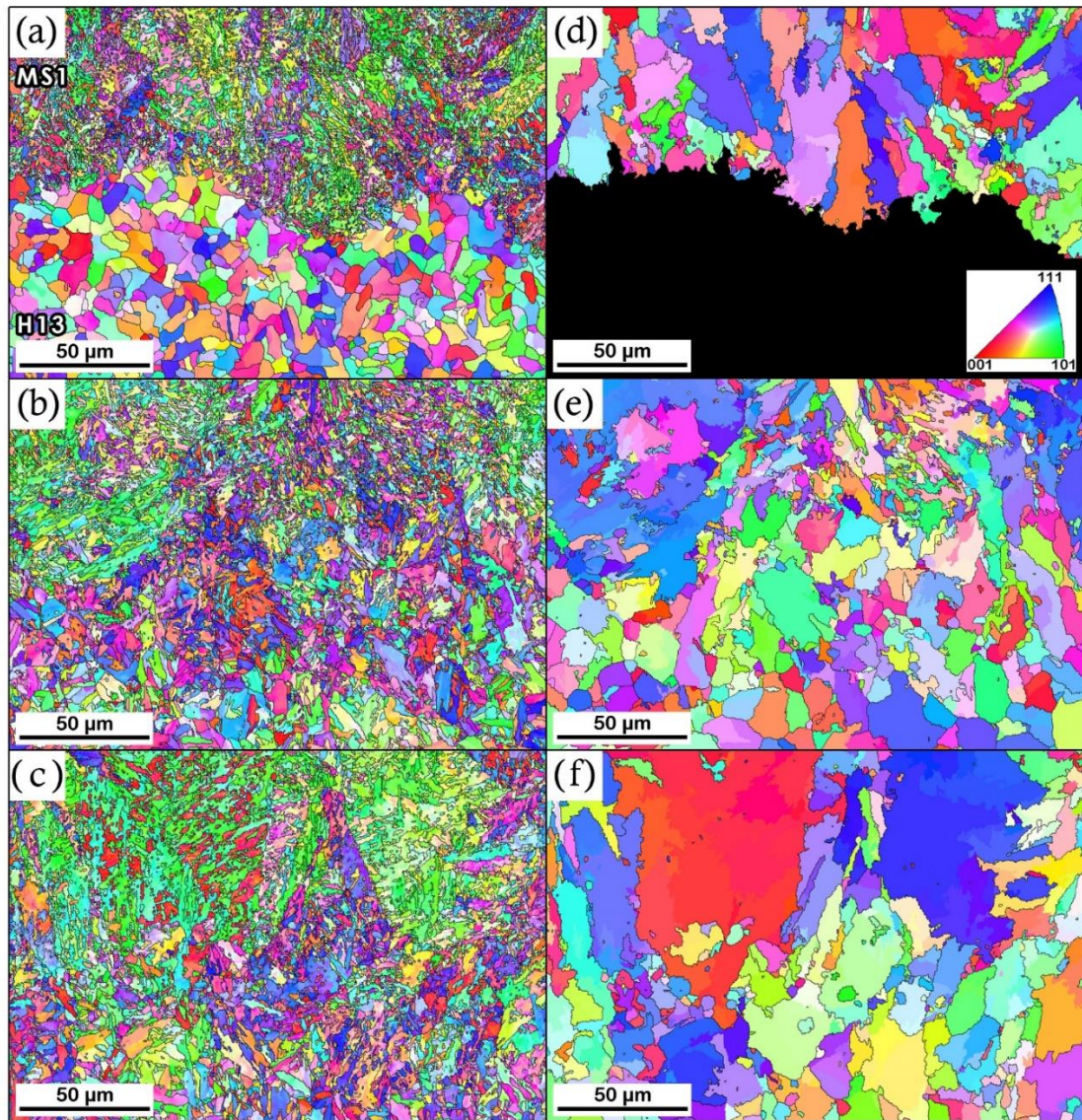


Figure 4-3: Inverse pole figure across the interface: a) as-built b) HT-I c) HT-II;
IPF map of the reconstructed PAGs: d) as-built e) HT-I f) HT-II.

Table 4-3: Grain size area (μm^2) of 18Ni300 maraging steel and H13 tool steel

	As-built	HT-I	HT-II
18Ni300	3.39	5.38	5.63
H13	23.33	5.71	5.83

Texture analysis was carried out to investigate the variations of crystal orientations upon the heat treatment sequences. The orientation distribution function was calculated based on the harmonic series expansion technique with a Gaussian-Half-Width of 5 degrees and truncation of $L = 16$. The sample reference frame for texture calculation was considered an orthogonal system composed of two perpendicular radial directions and the building direction of samples. No sample symmetry was assumed in texture calculations. Figure 4-4 represents the crystallographic pole figures corresponding to both sides of the interface. All three samples feature a relatively weak texture on both sides of the interface. Figure 4-4 (a) shows that two sides of the interface have different crystallographic textures in the as-built condition. The as-built 18Ni300 side represents an axial sample symmetry about the building direction with a dominant $\langle 011 \rangle \parallel BD$ texture. This texture results from the martensitic transformation from the directionally solidified austenite phase with dominant $\langle 111 \rangle \parallel BD$ texture components [25]. The observed axial symmetry resulted from 67° relative rotation of the laser direction between the printed layers. On the other hand, the H13 steel demonstrates orthotropic sample symmetry with dominant but weak $\{111\} \langle 011 \rangle$ and $\{001\} \langle 011 \rangle$ texture components.

In Figures 4-4 (b)-(c), the heat treatment sequences did not significantly alter the discussed crystallographic textures. Similar to the as-built sample, the 18Ni300 side of the heat-treated samples has a dominant $\langle 011 \rangle \parallel BD$ texture with axial symmetry. However, the high texture intensity of 18Ni300 in the HT-II sample (Figure 4-4 (c)) is due to large

martensitic blocks with similar orientations in the EBSD scan of this sample. The slightly different texture observed in the as-built 18Ni300 is justified by the lack of a statistically relevant number of orientations in the EBSD scan. The H13 side of the heat-treated samples also shows a texture identical to the as-built sample. However, the texture intensity decreased due to the martensitic transformation upon the heat treatment sequences.

The hardening treatment and the subsequent cooling resulted in BCC-FCC and FCC-BCT phase transformations. The similarity of crystallographic textures before and after the heat treatment sequences indicates the texture memory effect, where the initial texture is recovered after the martensite transformation due to a particular variant selection mechanism. This phenomenon was observed in the LPBF maraging stainless steel [26].

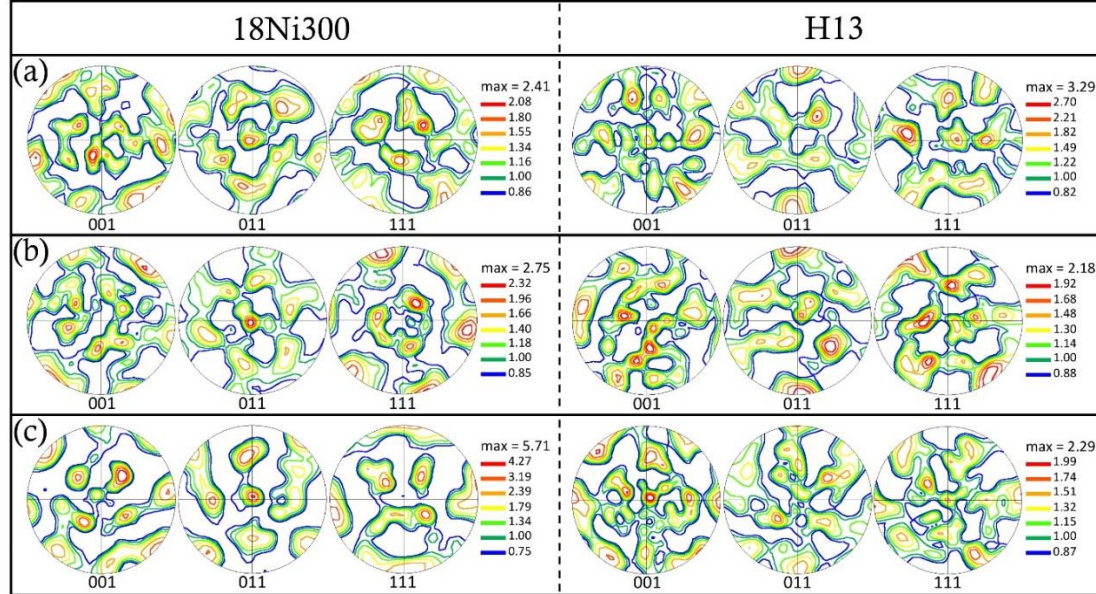


Figure 4-4: Crystallographic texture representation of a) as-built, b) HT-I, and c) HT-II samples. The build direction is located at the center of the pole figures.

Figure 4-5 illustrates the two-dimensional microhardness variation across the interface and the corresponding kernel average misorientation (KAM) in all three samples. In Figure 4-5 (a), significant color contrast is distinguishable between the 18Ni300 and H13 sides, where the substrate appears considerably softer than the 18Ni300 side. This is because the interface hardness lies between the hardness values of both sides in the as-built condition. This figure clearly shows the differences between the mechanical properties of both sides. Interestingly, a relatively uniform profile can be seen in the HT-I and HT-II samples indicating the hardness values were evenly leveled off after the heat treatment sequences (see Figures 4-5 (b)-(c)). Thus, the hardness variations are consistent with the corresponding microstructure in Figures 4-1 (d)-(f). Herein, higher hardness values of the as-built 18Ni300 can be attributed to the Hall-Petch effect as it contains more refined grains with respect to the corresponding heat-treated samples (see Table 4-3).

The stored strain energy of grains in terms of KAM angle is shown in Figures 4-5 (d)-(f), where brighter colors indicate higher strain energy or higher density of dislocations [27]. In the as-built sample, the 18Ni300 side is distinctly higher in strain energy than the H13 side resulting in an uneven distribution of KAM angles across the interface (see Figures 4-5 (d)). This can be due to rapid cooling during the SLM technique and the subsequent formation of crystal defects within the 18Ni300 grains while the substrate is in the annealed condition. However, KAM angles are distributed more homogeneously across the interface in the HT-I and HT-II samples. This can be associated with the martensitic transformation, which occurred simultaneously on both sides of the HT-I and HT-II samples (see Figures 4-5 (e)-(f)). This shows consistency with the hardness variation of heat-treated samples, where a homogeneous hardness gradient was developed upon the

heat treatment sequences (see Figures 4-5 (b)-(c)). Therefore, higher strength can be expected for the HT-I and HT-II samples, which the tensile test will later prove.

The mechanical behavior of heat-treated samples under uniaxial tensile loading is presented in Figure 4-6 and compared with the as-built sample. Figure 4-6 (a) obviously distinguishes the mechanical behavior between the as-built sample and the heat-treated ones. Both heat treatment sequences significantly improved the yield strength, ultimate tensile strength (UTS), and fracture strength at the expense of ductility (see Table 4-4).

The highest yield strength was obtained for the HT-II sample indicating the effect of heat treatment sequence on the mechanical properties. Thus, the HT-II sample seems to be more resistant to plastic deformation and can elevate the loading threshold for the 18Ni300-H13 bimetal. The UTS increased by approximately 60% for the HT-I and HT-II samples, which is remarkably favorable for the strength of bimetal. The as-built fracture strength of 382.54 MPa raised to 1733.82 and 1694.89 MPa for the HT-I and HT-II samples, respectively. In terms of ductility, although the increase of strength declined elongation upon the heat treatment sequences, the HT-II sample is interestingly more ductile than the HT-I sample. It seems that aging after hardening can cause softening; hence, higher elongation can be achieved for the HT-II sample. In addition, the larger PAGs of HT-II can make a positive contribution to the ductility of the 18Ni300-H13 bimetal. The static toughness was also calculated based on the stress-strain curves, where the as-built toughness of 135.85 J/cm³ dropped to 83.47 and 100.90 J/cm³ after HT-I and HT-II sequences, respectively. Since there is a trade-off between strength and ductility, the low ductility of heat-treated samples can reduce toughness. However, higher toughness was

achieved for HT-II compared to HT-I, which means the former heat treatment sequence is more favorable in terms of absorbing fracture energy.

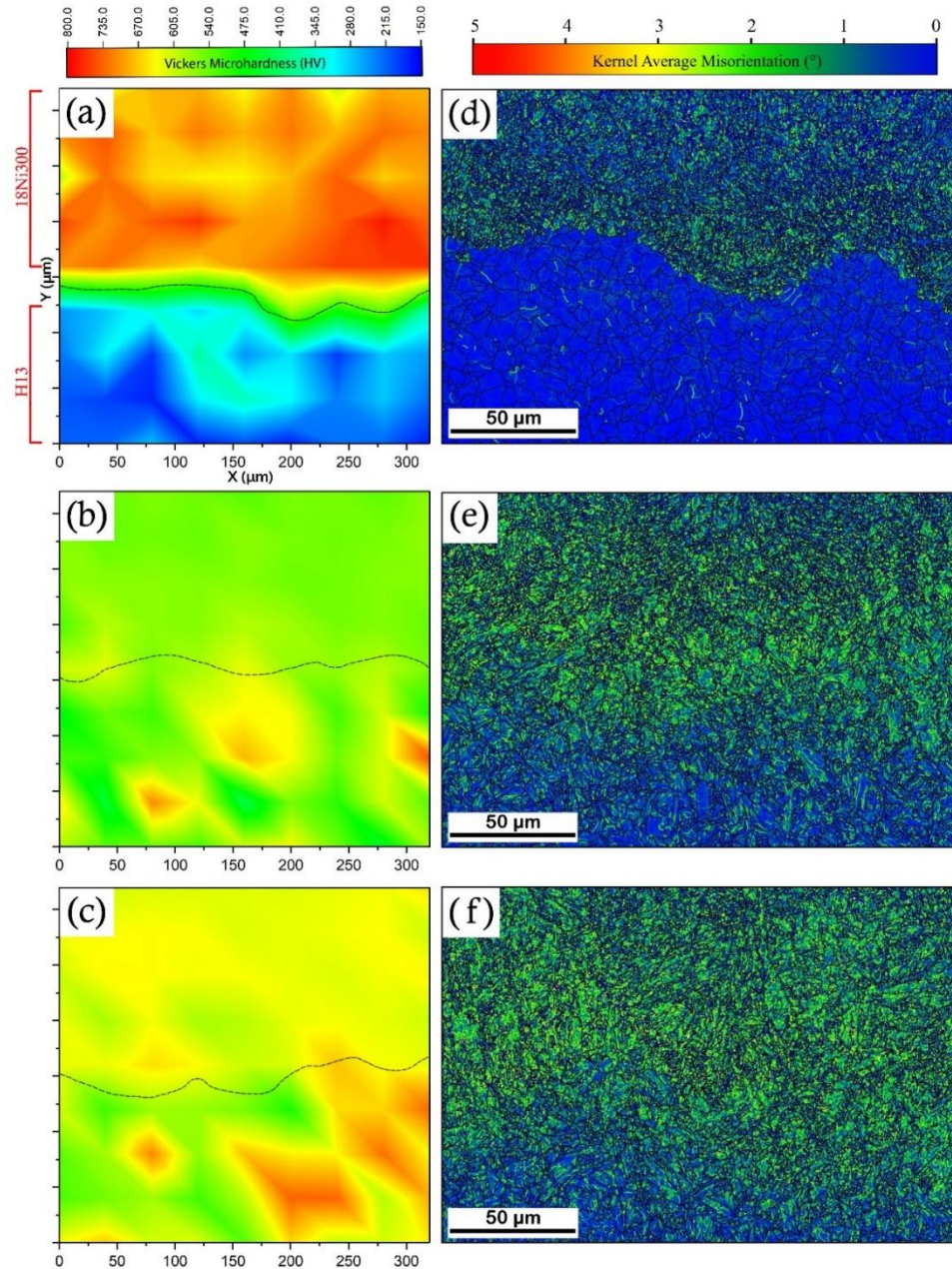


Figure 4-5: Vickers microhardness mapping across the interface: a) as-built b) HT-I c) HT-II; Kernel Average misorientation (KAM) mapping: d) as-built e) HT-I f) HT-II.

It is noteworthy that fracture occurred within the soft substrate of the as-built sample, while the HT-I and HT-II samples failed from the interface (see Figure 4-6 (b)). In Figures 4-6 (c)-(e), all three samples contain mainly fine dimples indicative of ductile fracture; however, some brittle facets were developed in the HT-I and HT-II samples due to the fracture of hard martensite laths. The strengthening effect of heat treatment sequences inevitably reduces ductility; however, fracture remains in the ductile mode at the microscopic level due to the dominance of dimples in the heat-treated samples.

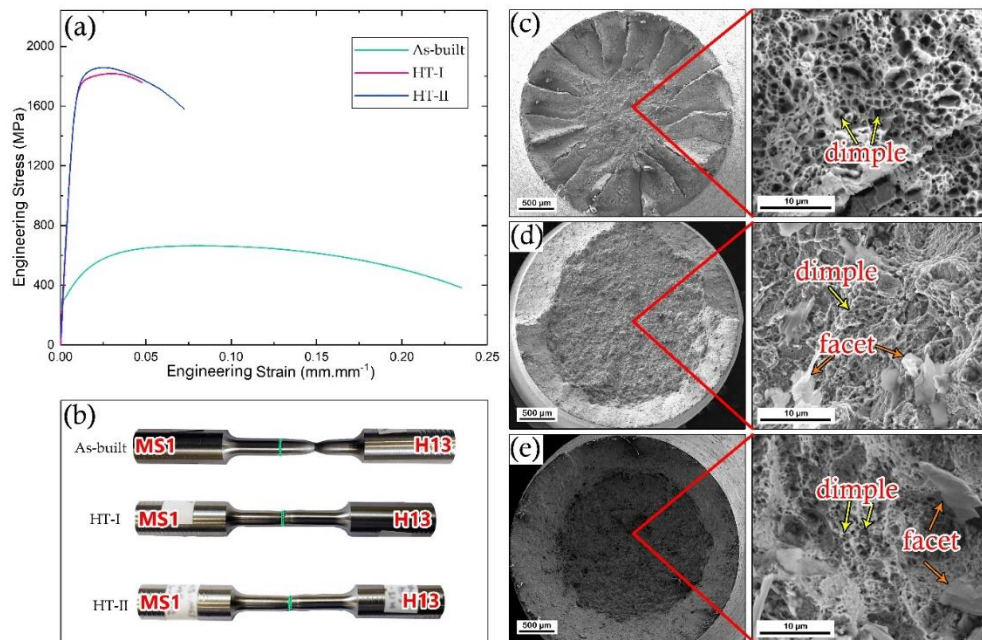


Figure 4-6: a) engineering stress-strain curves b) fracture tensile samples; SEM fracture surface: c) as-built d) HT-I e) HT-II

Table 4-4: Mechanical properties of 18Ni300-H13 bimetals.

	As-built	HT-I	HT-II
Yield (MPa)	300.85 ± 5	391.53 ± 8	467.21 ± 7
UTS (MPa)	664.41 ± 13	1818.89 ± 15	1857.54 ± 22
Fracture Strength (MPa)	382.54 ± 9	1733.82 ± 12	1694.89 ± 14
Ductility	0.24 ± 0.003	0.05 ± 0.001	0.06 ± 0.004
Toughness (J/cm³)	135.85 ± 7	83.47 ± 5	100.90 ± 8

4.3 Conclusion

The HT-I and HT-II sequences effectively homogenized the microstructure across the interface, and the following results were achieved:

- A uniform martensitic lath structure and identical grain sizes were developed on both sides of the interface, resulting in a less distinguishable interface upon the heat treatment sequences.
- No dominant crystallographic texture was developed in the heat-treated samples.
- The yield strength and UTS increased at the expense of ductility in HT-I and HT-II samples.
- HT-II obtained higher ductility, UTS, and fracture toughness compared to HT-I.

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5 Hybrid Manufacturing of MS1-P20 Tool Dies by Powder Bed Fusion

5.1 Introduction

Additive manufacturing (AM), the recent innovative manufacturing technique, is capable of producing any material from polymers, ceramics, metals, and composites with a wide range of versatility in terms of geometry and outstanding properties [1]–[7]. This method is referred to as “3D printing” since the products are 3D objects manufactured by stepwise deposition of a feedstock material. Metal additive manufacturing (MAM) has been the area of interest for many researchers due to finer microstructural features and relatively superior yield strength as well as UTS [8]. Concerning the feedstock material and the heat source, MAM encompasses two main techniques as powder bed fusion (PBF) and direct energy deposition (DED) [9], where the latter seems unrivaled in terms of design flexibility and dimensional accuracy [10]. In PBF, the layer upon layer of powder feedstock is selectively melted via a high-power laser beam, and finally, the virtual CAD design emerged as a real metal part with all the required geometrical complexity and desired properties [11]–[13].

Since both PBF and DED techniques involve melt pool formation and subsequent solidification, they are relatively identical to fusion welding in terms of melting and solidification [14]. This feature motivates many researchers to develop the idea of utilizing AM as a joining process [15]–[26]. They use a conventionally made metal as the build plate and carry out the layer-by-layer deposition of feedstock material, and finally, a bimetallic component is fabricated. This procedure is also called hybrid additive manufacturing (HAM) since the final part's manufacturing process engages with both

conventional and additive manufacturing techniques [27]. This hybrid approach can generate the bimetallic structures out of many alloys and compounds, including aluminum, titanium, nickel alloys, and steels [17], [19], [20], [24]–[26], [28], [29]. For example, the IN625-SS316L bimetal was produced using the DED process for nuclear reactors and the aerospace industry, where high mechanical strength at elevated temperatures and high corrosion resistance are essential factors for materials selection [16]. Zhai et al. [21] deposited γ -TiAl on Ti6Al4V using electron beam melting (EBM) and fabricated a bimetallic component for turbine blades. The bimetallic structures are intended to stimulate several properties, including thermal, mechanical, and corrosion properties at the same time; thus, leading to superior performances in severe working conditions.

Additive manufacturing of bimetals requires a feedstock material with proper printability, dilution with the substrate, and satisfactory mechanical properties. The control of porosity level and other defects is also a crucial factor for forming a cohesive bonding between the two sides. In this regard, maraging steels have been frequently fabricated using the PBF technique since they satisfy the criteria above to a great extent [30]–[32]. High strength along with good ductility, low susceptibility to cracking during solidification, and good corrosion resistance are the main advantages of using maraging steels in additive manufacturing [15], [33]–[35]. For instance, Shamsdini et al. [36] fabricated a highly dense maraging steel (>99.95%) with an ultimate tensile strength (UTS) of over 1200 MPa and total elongation of 14%. The high strength of maraging steels has been attributed to a very fine martensite structure achieved by rapid cooling during the PBF process [37]. Therefore, maraging steels can be used as the feedstock material for hybrid additive manufacturing (HAM) processing. Recently, many researchers utilized maraging steels to fabricate

bimetal components using PBF. Tan et al. [38], [39] worked on depositing maraging steel powder on a copper block and produced a defect-free AM bimetal with a high-strength bond and superb fatigue properties, which can be used in heat transfer and cryogenic components.

The PBF processed maraging steels exhibited remarkable bonding with many steel grades, including CrMn, stainless steels, TRIP steels, and tool steels [18], [22], [23], [40], [41]. Excellent metallurgical bonding, high mechanical performance, and a negligible level of defects (i.e., porosity) were generally the specifications of maraging steel bimetals. For instance, the MS1-H13 bimetal, consisting of maraging stainless steel and H13 tool steel, contained a very narrow interface promoting a noticeably cohesive bonding between the two materials [42]. This interface showed higher strength than the substrate and did not fail under tensile loadings [42].

Besides the deposition parameters, it was shown that proper heat treating procedures could also significantly improve the interfacial morphology and the resultant mechanical properties of the produced bimetal [27]. However, AM bimetals lack proper case studies, and more investigation should be done to prove the capability and reliability of additive manufacturing of bimetals.

The maraging steel-tool steel bimetal has shown excellent potentials for the tool and die industry [27], [42]. As an illustration of bimetal application, advanced dies can be made of bimetallic component consisting of a conventionally-made bulk side and an AM material on the contact surfaces with pre-designed cooling channels. This can be applied to many conventional tool steels such as AISI P20, mainly used in plastic injection molding

applications. This material features excellent mechanical properties and satisfactory fatigue lifetime characteristics; however, the poor wear resistance of P20 can ease plastic deformation, resulting in early deterioration and failure of this material under compressive loading service conditions [43], [44]. Thus, additive manufacturing of bimetals can be an approach to address this problem. In this work, for the first time, high strength MS1 maraging steel powder was printed on top of an AISI P20 block using the PBF technique to manufacture dies for the automotive industry. Microstructural investigations were then performed to characterize the interface between the two sides using advanced electron microscopy techniques; and finally, the performance assessment of the MS1-P20 hybrid steel was evaluated using various mechanical tests.

5.2 Experimental Procedure

Virgin maraging steel powder (MS1) manufactured by EOS GmbH with a particle size of 15-45 μm and an AISI P20 block of 150 $\text{mm} \times 70 \text{ mm} \times 100 \text{ mm}$ dimensions were used as the raw materials for manufacturing the MS1-P20 hybrid steel. The chemical composition of both materials is listed in Table 5-1. No heat treatment was carried out on the P20 build plate, and it was sand blasted prior to the installation inside the printer to avoid the laser reflection effect. An EOS additive machine model M290 equipped with a Yb-fiber laser of 400 W power was used for PBF.

An argon gas with 99.9% purity was blown to the printer chamber to reduce the oxygen level below 0.1% throughout the process. The P20 build plate was preheated to 40 $^{\circ}\text{C}$. A thin layer of maraging steel (MS1) with 40 μm thickness was spread uniformly over the P20 build plate, and the process was initiated using a laser with the laser spot size of 100 μm , a scan speed of 960 mm/s , and hatch distance of 0.11 mm . Each MS1 powder

layer was deposited by a stripe-like scanning strategy and a 67° rotation with respect to the underneath layer. Finally, round bar specimens were prepared for further characterizations. These specimens had the height of 10 cm on the MS1 side. In addition, a plastic mold forging die was fabricated using the CAD file shown in Figure 5-1, demonstrating the feasibility of making real parts via the hybrid approach.

The round bar samples were then sectioned perpendicular to the building direction, mounted, and polished for characterizing the interface. The primary characterization was carried out using a JEOL 6400 scanning electron microscope (SEM), and the micrographs were then taken both in back scattered and secondary electron modes. The SEM was also used for fractography of samples after the tensile test. An EDS elemental distribution map was also captured using a Genesis energy dispersive X-ray (EDS) detector.

Table 5-1: Chemical composition (wt%) of MS1 powders and the P20 substrate.

MS1 maraging steel powder									
Fe	C	Ni	Co	Mo	Ti	Mn, Si	Al	Cr, Cu	P, S
Bal.	≤0.03	17- 19	8.5- 9.5	4.5- 5.2	0.6- 0.8	≤0.1	0.05- 0.15	≤0.5	≤0.01
P20 cold work tool steel									
Fe	C	Mn		Cr		Si		Mo	
Bal.	0.28-0.40	0.60-1.00		1.40-2.00		0.20-0.80		0.30-0.55	

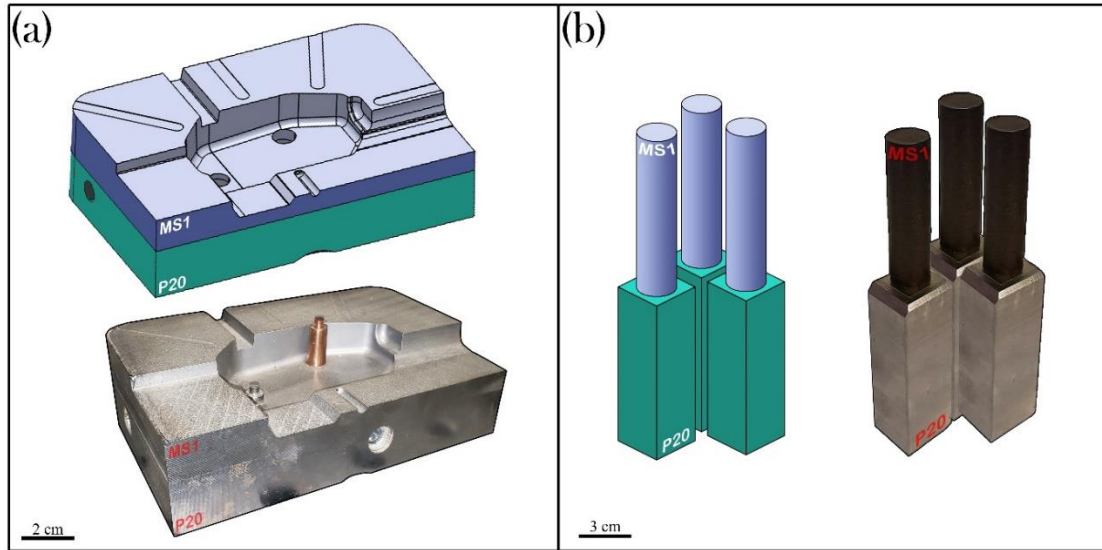


Figure 5-1: a) hybrid die for plastic mold forging b) hybrid MS1-P20 specimens.

For electron back scatter diffraction (EBSD), a very fine polishing method was conducted by polishing the mounted samples down to 0.05 μm colloidal silica. The EBSD was performed using a Hikari EBSD system along with AZtec data acquisition software. Step sizes of 400, 150, and 20 nm were selected for low, medium, and high magnification mappings across the interface. EBSD data was processed via HKL Channel 5 software, where various analyses were extracted, including IPF-Z, grain boundary, recrystallized grains, kernel average misorientation (KAM), and phase maps. At high magnification, the KAM maps were presented in an extended color scheme to better present the misorientations. To characterize the fine precipitates of P20, a FEI Helios Nanolab 650 dual-beam system was used to extract a very thin specimen from the P20 steel. Transmission electron microscopy (TEM) was carried out via a FEI Tecnai Osiris TEM equipped with a Bruker quad-core X-ray energy dispersive spectroscope (XEDS).

Two mechanical tests were conducted to evaluate the mechanical properties of

MS1-P20 hybrid steel. Hardness values were measured across the interface using a Vickers microhardness system equipped with a diamond Berkovich nanoindenter. A square of $320 \times 320 \mu\text{m}^2$ covering 9×9 spots with a $40 \mu\text{m}$ span were selected, where the nanoindenter applied a constant load of 50 mN at a strain rate of 0.2 s^{-1} at each spot.

Uniaxial tensile tests were performed together with the digital image correlation (DIC) technique to demonstrate the strain field and the tensile mechanical properties simultaneously. According to ASTM E8M [45], three dog-bone samples were machined out of the round bars, where the interface was located right at the center. A hydraulic Instron universal machine was used for the uniaxial tensile loading tests, and the strain rate was fixed at $9 \times 10^{-3} \text{ s}^{-1}$. The 10 cm tensile samples were first sprayed with speckle particles to make high contrast areas at sample surfaces. A global shutter CMOS sensor camera was aligned perpendicular to the sample surface and 100 mm away from the sprayed sample. The image recording system captured 1036 frames up to the fracture point for 257 s.

5.3 Results

5.3.1 Bulk Materials

Figure 5-2 illustrates the microstructural features of the P20 steel. The low magnification (LM) micrograph shown in Figure 5-2 (a) barely reveals the coarse grain structure. The SEM micrograph in Figure 5-2 (b) exhibits the distribution of fine particles within the grains as well as the grain boundaries. To investigate the fine particles, EDS elemental mapping in TEM was carried out on samples prepared by FIB as shown in Figure 5-2 (c), where nano-size precipitates rich in Cr and Mn can be observed. These

nanoparticles also contain high amount of carbon so they can be $(\text{Cr}_{0.6}\text{Mn}_{0.4})_{23}\text{C}_6$ intermetallic component. Identical precipitates were also reported in the as-built P20 steel [46].

The solidification structure and grain morphology of the deposited MS1 are shown in Figure 5-3. Figure 5-3 (a) shows two melt pools with different shapes as a result of changes in the scanning direction. The bottom melt pool perpendicular to the scanning direction was solidified as a semi-elliptical shape, while the upper melt pool seems elongated in Figure 5-3 (a). Thus, the melt pools are semi-elliptical in cross-section and stretched along the scanning direction.

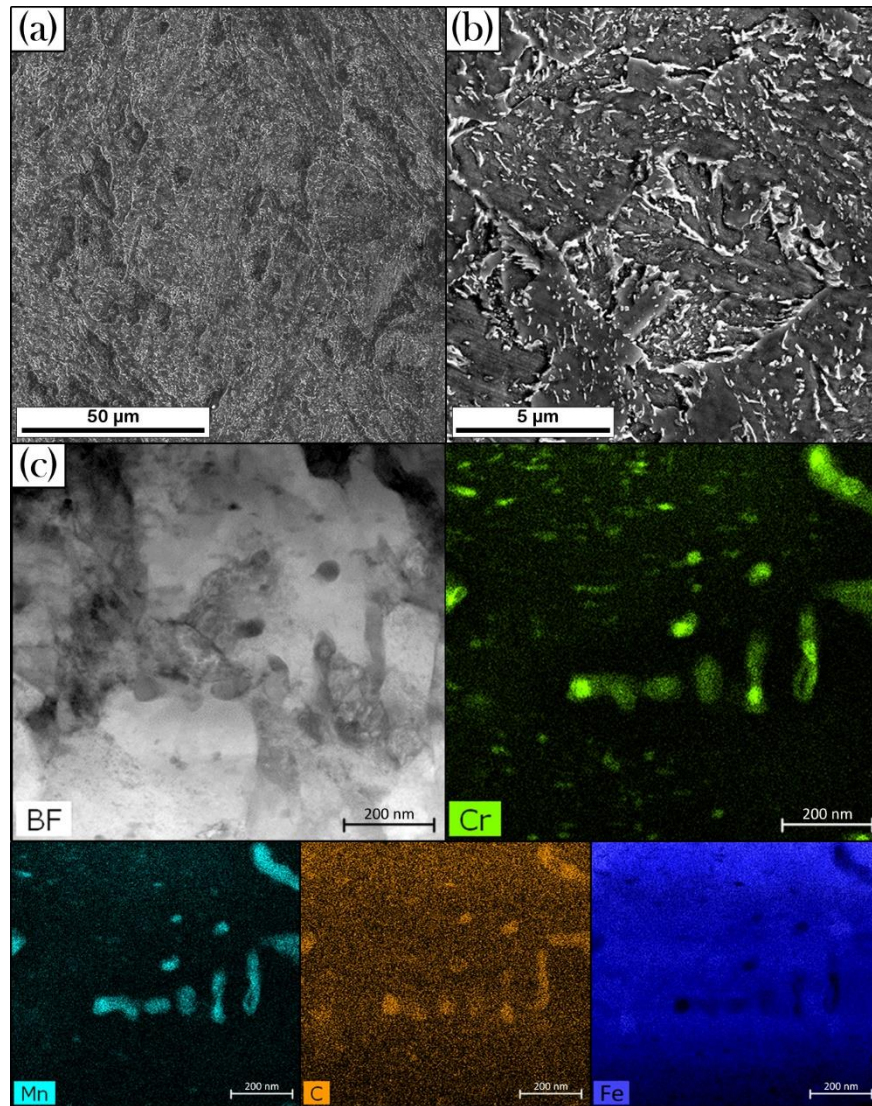


Figure 5-2: a-b) SEM micrographs of P20 mold steel c) EDS mapping of precipitates.

Inside the melt pools, a network of solidification cells can be observed containing equiaxed cells and columnar dendrites (see Figure 5-3 (b)). This is the typical solidification structure in laser-processed maraging steels [47]–[49], and it was reported that the microsegregation of alloying elements is responsible for the formation of this cellular structure [32].

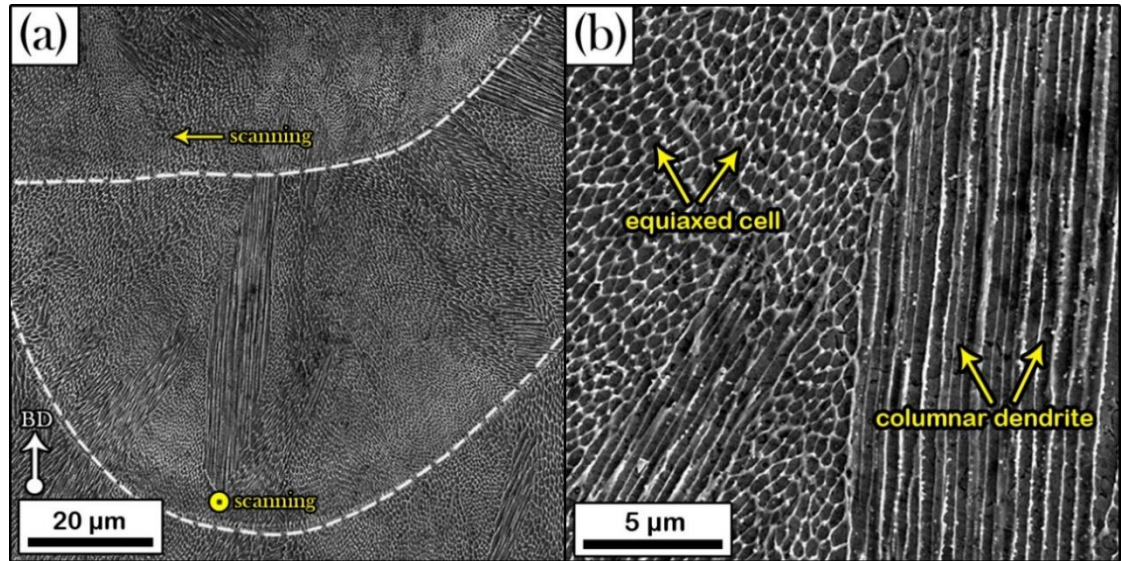


Figure 5-3: Solidification structure of as-built MS1: a) melt pools b) solidification dendrites.

The grain morphologies of bulk P20 and MS1 steels away from the interface are presented in Figure 5-4 via the electron backscatter diffraction (EBSD) technique. The grain morphology of the as-built maraging steel is illustrated in Figure 5-4 (a). The MS1 maraging steel contains a fine grain morphology with equiaxed and columnar grains mostly deviated towards the build direction. In comparison, the P20 steel consists of coarser grains than the MS1 (see Figure 5-4 (b)). For both materials, there is no preferential crystallographic orientation as indicated by the random color distribution of grains. Figures 5-4 (c) and 5-4 (d) show the grain boundary mapping within the MS1 and P20, respectively. The MS1 involves higher density of low-angle grain boundaries (LAGBs) as compared to the P20 steel. At higher magnifications, MS1 grains are shown to be grown towards the building direction, while there is no such thing for the P20 grains (Figures 5-4 (e) and 5-4 (f)). As mentioned earlier, the MS1 has no crystallographic texture; however, the grains seems to be alongside the building direction. To better prove this phenomenon, the parent austenite grain (PAG) boundaries are reconstructed and superimposed in Figure 5-4 (g)

with bold yellow lines. The PAGs were constructed by the analysis of groups of martensite with identical related variants. The full description of this method was first proposed by Gomes et al. [50]. The superimposed PAGs seem to have a columnar morphology and explicitly show the directional solidification towards the building direction. Kernel average misorientation (KAM) mapping was illustrated in Figures 5-4 (g) and 5-4 (h) as well. KAM represents misorientations up to 5° within the grains, and can be considered as the crystal distortion induced by dislocations as well as strain energy of a grain. Herein, the MS1 grains seem to have substantial misorientations with respect to the P20 grains. The grain size diameters were measured for both materials indicating finer grain sizes of the deposited MS1. The average grain diameter was calculated to be $9.05\ \mu\text{m}$ and $7.70\ \mu\text{m}$ for P20 and MS1, respectively. Figure 5-4 (i) shows the quantitative measurement of KAM along the dash lines in Figures 5-4 (g) and 5-4 (h) representing strain energy of a single grain in the MS1 and P20. On average, the P20 steel incorporates lower misorientation as well as fewer peaks with lower intensity. On the other hand, the MS1 contains higher misorientations with more frequent peaks. Consequently, the MS1 involves a finer grain morphology with higher internal strain energy, while the coarse grain of P20 is in a less energy state than the MS1 grains.

5.3.2 Interface Characterization (Low Mag)

The SEM micrographs of MS1-P20 hybrid steel at various magnifications are shown in Figure 5-5. The fish scale pattern of melt pools is evident in the PBF-MS1, and a wavy interface was formed between both sides (see Figure 5-5 (a)). The elemental contrast across the interface is presented in Figure 5-5 (b) in back scatter electron mode. There is a contrast gradient from the dark area of P20 to the brighter area of MS1, indicating

how chemical composition varies from P20 to MS1. It seems that dilution is induced by the variation of chemical composition between two materials [51], and it looks significant in the first deposited layer, where elemental color contrast is darker and less distinguishable from the substrate. However, the second and third layers are less diluted and tend to reach the color contrast of MS1 in the upper layers. These layers also contain the trace of marangoni convection, which was observed in many AM bimetals [18], [38], [42]. Figure 5-5 (c) shows a melt pool deposited on top of P20. The melt pool formed very fine cells as a result of rapid solidification. Figure 5-5 (d) presents the solidification structure of the melt pools involving dendritic cells deviated towards the build direction, which features the highest thermal gradient. Despite the Marangoni effect, the microsegregation of alloying elements occurred, leading to the formation of dendritic solidification morphology within the melt pools [42].

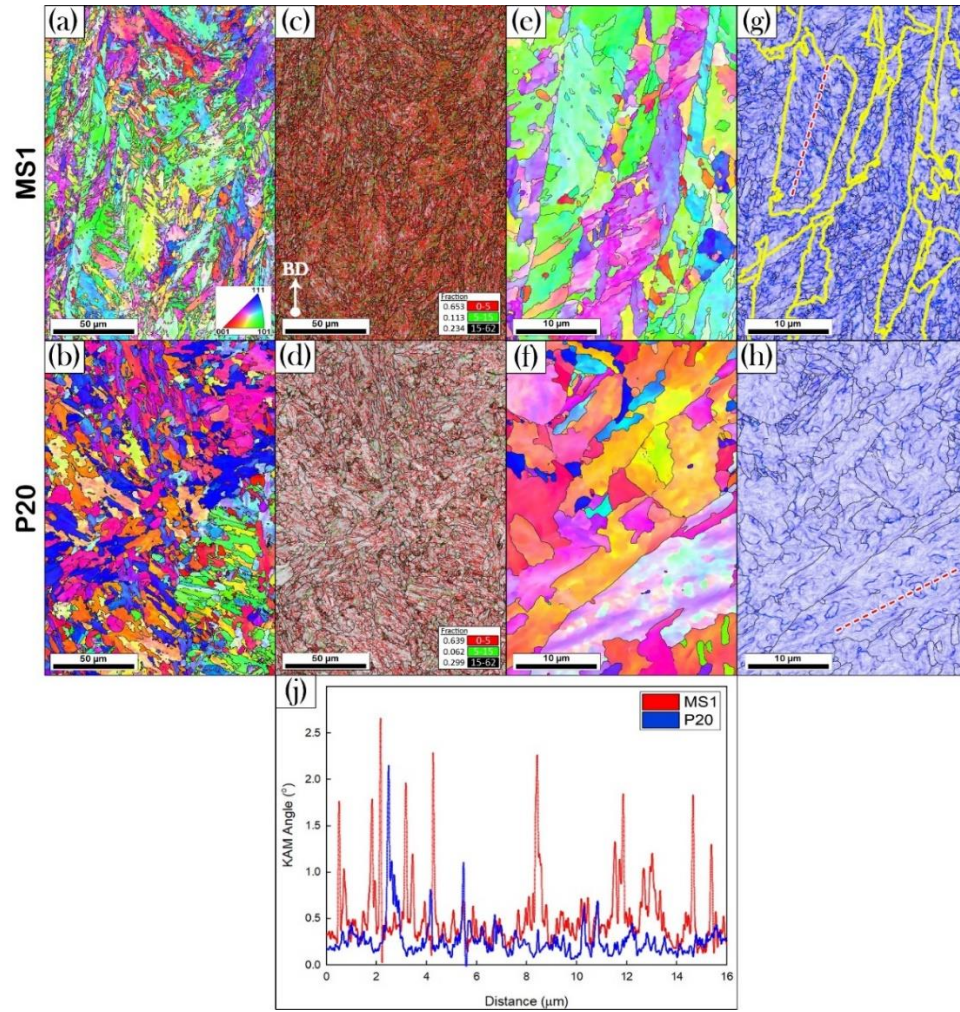


Figure 5-4: Inverse pole figure (IPF): a) MS1 b) P20; grain boundary mapping: c) MS1 d) P20; high magnification IPF: e)MS1 f) P20; KAM mapping: g)MS1 h) P20; i) KAM distribution of dash lines.

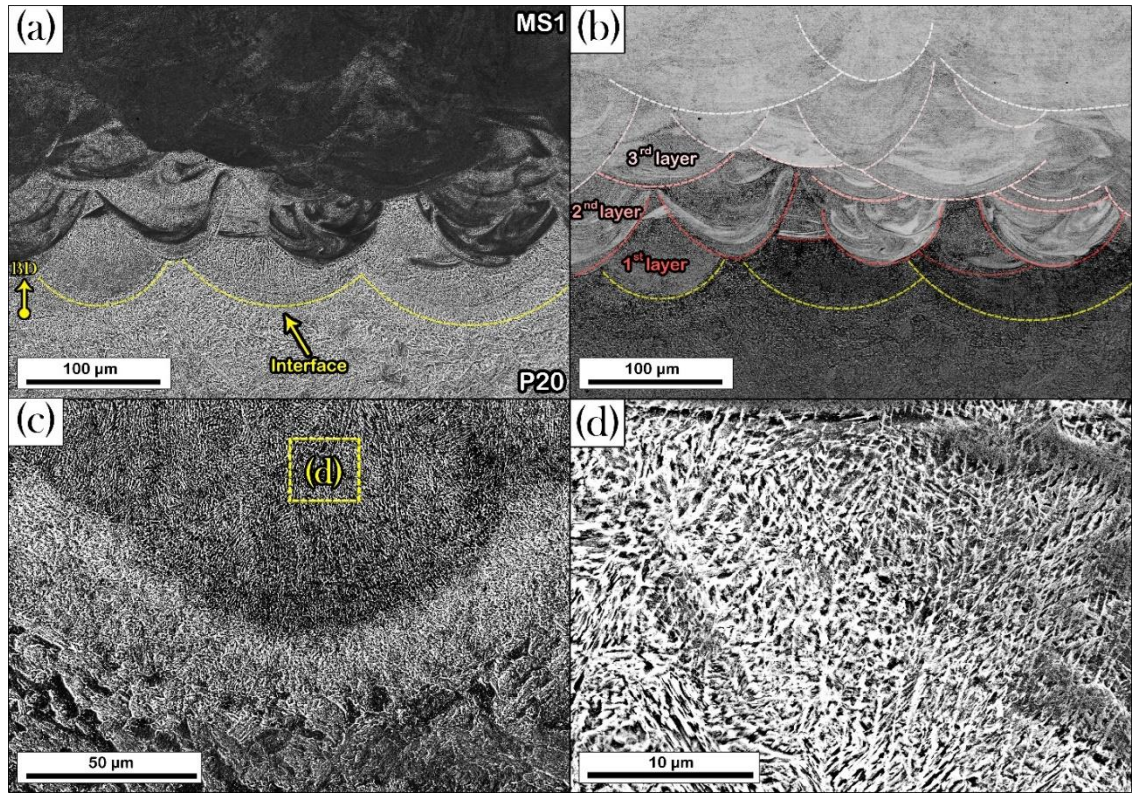


Figure 5-5: SEM micrographs of the MS1-P20 hybrid steel: a) SE mode; b) BSE mode; c) high magnification SEM of the interface; d) Solidification cells.

Figure 5-6 (a) exhibits the distribution of main alloying elements across the MS1-P20 hybrid steel. Ni, Co, and Mo are mainly concentrated in the PBF-MS1, and the P20 side is more enriched in Cr and Mn. Fe is more highlighted on the substrate side since the P20 is a low alloy steel compared to MS1. This figure shows that the chemical composition is totally different between both sides. It seems that long-range diffusion occurred between MS1 and P20 in the first few deposited layers since Mn and Cr diffused to the MS1 side as a result of dilution shown in Figure 5-5. Figure 5-6 (a) also shows the Marangoni convection, where the MS1 alloying elements (i.e., Ni and Co) were accumulated. The Marangoni convection is a result of surface tension differences between the center and the edge of the melt pool. The fluid flow induced by the Marangoni effect can boost the stirring of elements by transferring the MS1 elements to the molten substrate and P20 elements to

the melted powder; thus, leading to an interpenetration phenomenon between both sides. The variation of elements can be better understood using the EDS line analysis. Figure 5-6 (b) illustrates the quantitative analysis of elements from P20 to MS1 side, where three distinct regions were determined. The P20 side seems depleted in alloying elements with negligible amount of Cr, and Mn, and there is no Ni, Co, and Mo content as expected. From the interface, the MS1 alloying elements (i.e., Ni, Co, Mo) begin to appear and rise gradually towards the building direction, while Cr and Mn drop smoothly. These two opposing trends continue up to 150 μm from the interface forming a region with the alloying elements of both sides. Regarding the chemical composition, this region seems to be a transition from P20 to MS1. This is why it is called the transition zone (TZ), and was also reported in the MS1-H13 bimetallic structure [42]. The transition zone reflects the dilution effect in the first few stages of the process and indicates successful compatibility of the substrate with the deposited powder feedstock.

Low magnification EBSD analysis of the interface of the MS1-P20 hybrid steel is shown in Figure 5-7. The IPF map (see Figure 5-7 (a)) clearly indicates three regions with different grain sizes as: P20, TZ, and MS1. As expected, the P20 substrate contains coarse grains similar to the bulk P20 shown in Figure 5-4 (b). On top of P20, the first melt pool solidifies with an ultrafine grain structure at the interface. Further deposition of MS1 powder layer proceeds with the solidification of slightly coarser grains along the building direction, which then reaches the bulk grain morphology of MS1 at about 150 μm distance from the interface. Thus, the variation of grains can also be distinguished for the aforementioned three regions across the interface. This variation is also plotted in Figure 5-7 (c), where the grain diameter reduction is shown quantitatively across the interface.

The highest values of grain area belong to the P20, and the lowest values reach right at the interface. The values of TZ explicitly indicates the fineness of grains near the interface as compared to the bulk P20 and MS1. By reaching the MS1, the grains slightly grow with respect to TZ and forms the identical grain sizes to that of bulk MS1 shown in Figure 5-4 (a). Figure 5-7 (a) also shows that the first MS1 layer contains finer grains than the next layers, which can be related to the substrate's heat sink effect during the deposition of the first layer. Indeed, the transition zone involves finer grain than the bulk MS1. MS1 grains were randomly grown on P20 grains and did not follow the crystallographic orientation of P20 grains. Thus the solidification of the first layer was non-epitaxial without forming preferential orientation. There is a noticeable distinction between the substrate (P20) and additive side (i.e., TZ and MS1) in terms of KAM misorientation (see Figure 5-7 (b)). The P20 steel is a region with the lowest KAM intensity, while both TZ and MS1 are seemingly high in KAM angles. There is no considerable difference between TZ and MS1; however, the MS1 seems to have slightly lower intensity than the TZ region. The KAM map distinguishes the P20 steel from TZ and MS1 regions, where the density of high misorientation angles shown in green color rise remarkably from the interface. This is also proven by Figure 5-7 (d) as the average misorientation angles for P20 and TZ & MS1 are 0.74° and 1.67° respectively. It means that the additive side is more enriched in stored strain energy than the substrate.

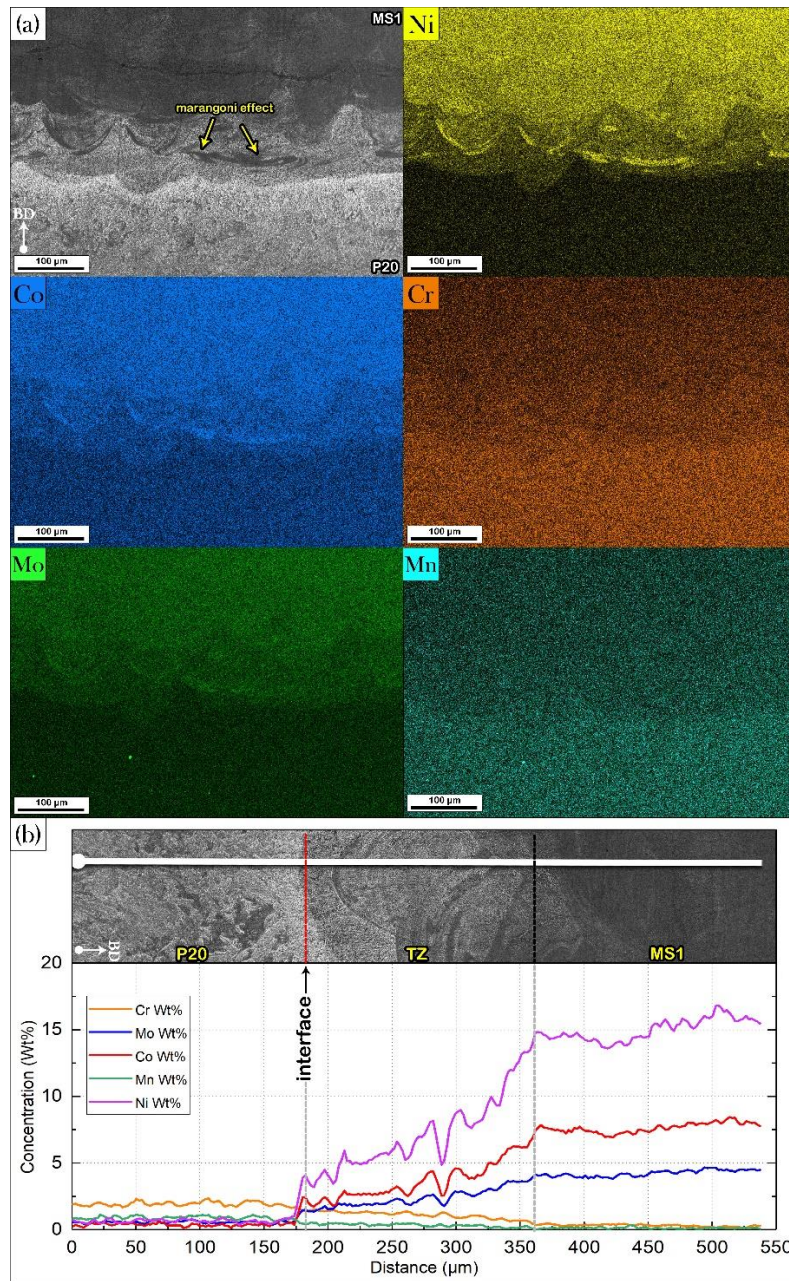


Figure 5-6: a) Elemental distribution mapping b) EDS line analysis across the interface.

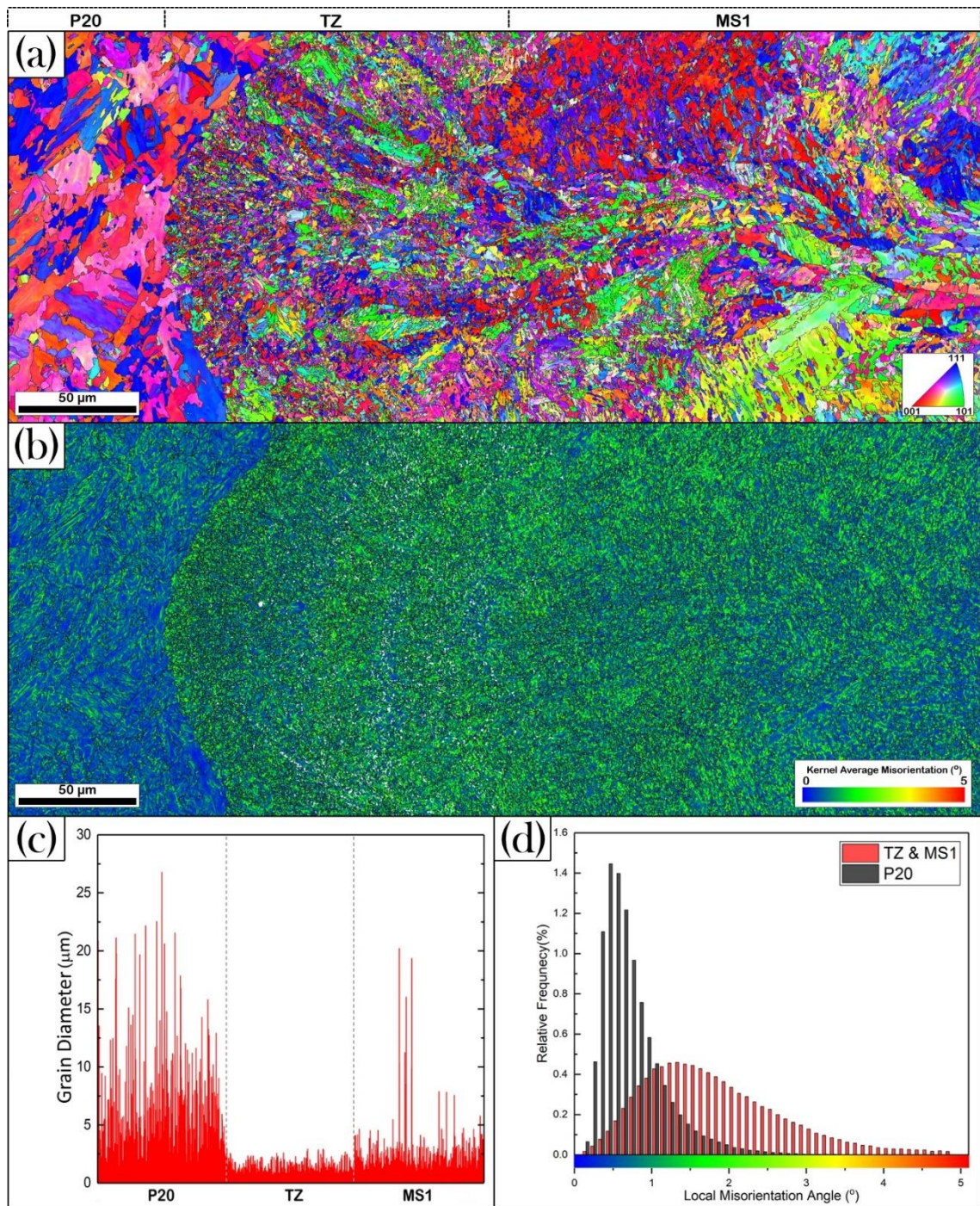


Figure 5-7: Low magnification EBSD analysis of the interface: a) IPF-Z map; b) KAM map; c) grain size variation; d) KAM distribution.

5.3.3 Interface Characterization (High Mag)

As mentioned earlier, the grain morphology across the interface can be hardly detected via SEM due to fine structure of deposited MS1; however, the grain structure can be observed via focus ion beam (FIB) milling technique. Figure 5-8 (a) shows a cross section cut at the interface and then polished for SEM microscopy (see Figure 5-8 (b)). This Figure clearly shows the exact location of the interface where ultrafine grains can be clearly seen on top of large grains of the substrate.

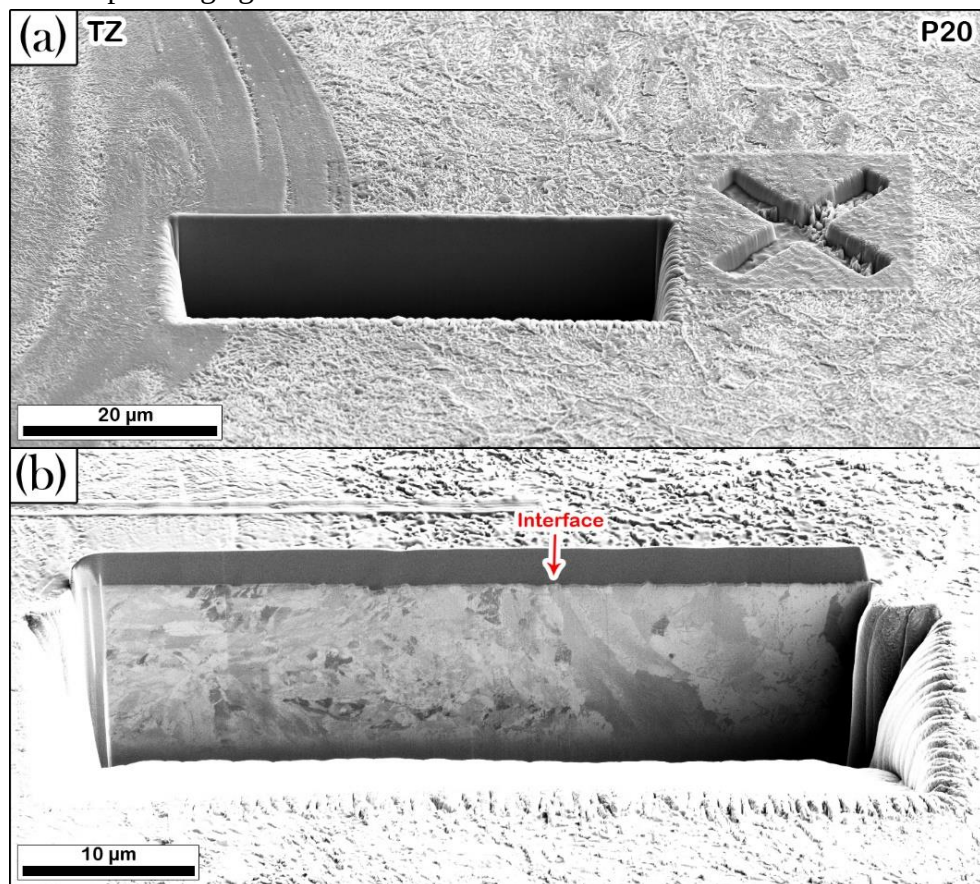


Figure 5-8: SEM micrograph of: a) FIB milling location b) Interface

Further investigation was carried out via high magnification EBSD technique to deeply characterize the interfacial features between MS1 and P20. Figure 5-9 clearly shows that the interface is a very sharp grain boundary located between the fine grains of MS1

and coarse grains of P20. Figure 5-9 (a) illustrates phase distribution across the MS1-P20 hybrid steel with dominant blue color indicating BCC phase and a negligible amount of FCC in red color. The P20 steel is covered in pure blue color, which can be associated with its ferritic matrix and hence the BCC crystal structure. The blue color in TZ, however, is indicative of a martensitic matrix since the BCT crystal structure of martensite is identical to ferritic BCC structure in EBSD [52]. In addition, the transition zone (TZ) consists of very fine grains with FCC structure, thus the retained austenite coexists within the martensitic matrix of TZ.

Figure 5-9 (b) illustrates the deformed grain mapping of MS1-P20 interface excluding the FCC austenite grains. The deformed grain mapping categorizes grains into three types based on the internal average misorientation: substructured, recrystallized, and deformed. The internal average misorientation angle is more than 1° for deformed grains, while it is less than 1° for recrystallized and substructured grains. However, the substructured grains contain subgrain misorientation of more than 1° [53]. The recrystallized grains are indeed the grains with almost no internal crystal misorientation and hence having the least strain energy. Since this analysis was carried out on the blue grains of Figure 5-9 (a), it can be concluded that the recrystallized grains with no internal misorientation are BCC structure rather than BCT structure of martensite mentioned earlier. Therefore, the recrystallized grains are ferrite with no internal misorientation.

Due to fine grain morphology across the interface, high magnification EBSD was performed and presents in Figures 5-9 (c)-(e) in order to identify the microstructural features in more detail. Figure 5-9 (c) displays the IPF map of the interface at 15000 X magnification. Apparently, it shows that the distinct boundary between the coarse grain of

P20 and fine grains of TZ can be considered as the interface of MS1-P20 hybrid steel. Moreover, an area of P20 grain seems brighter in color indicating this area is slightly tilted with respect to the P20 grain. The tilted area suggests the initiation of melting at the interface. However, since the heat input is not sufficient to fully melt the substrate grain at the interface, it is partly melted, tilted and solidified as a partially-melted grain as shown in Figure 5-9 (c). Figure 5-9 (d) shows the kernel average misorientation (KAM) across the interface in an extended rainbow color scheme. KAM is representative of micro-strain energy stored at LAGBs due to the accumulation of dislocations. In other words, KAM mapping illustrates the areas with high dislocation densities. It seems that dislocations have more density on the TZ than P20, which can be associated with finer grain morphology of TZ as well as the martensite transformation. The partially melted grain also seems to be high in KAM angles at the grain boundaries, where the misorientation between the partially melted grain and the parent grain is fitted with accumulation of dislocations. Figure 5-9 (e) illustrates the corresponding deformed grain mapping regardless of FCC grains. The blue recrystallized grains are embedded between the coarse substructured grains. Comparing Figures 5-9 (d) and 5-9 (e), the blue ferrite grains seem to be free from KAM misorientation, and hence they possess the lowest strain energy.

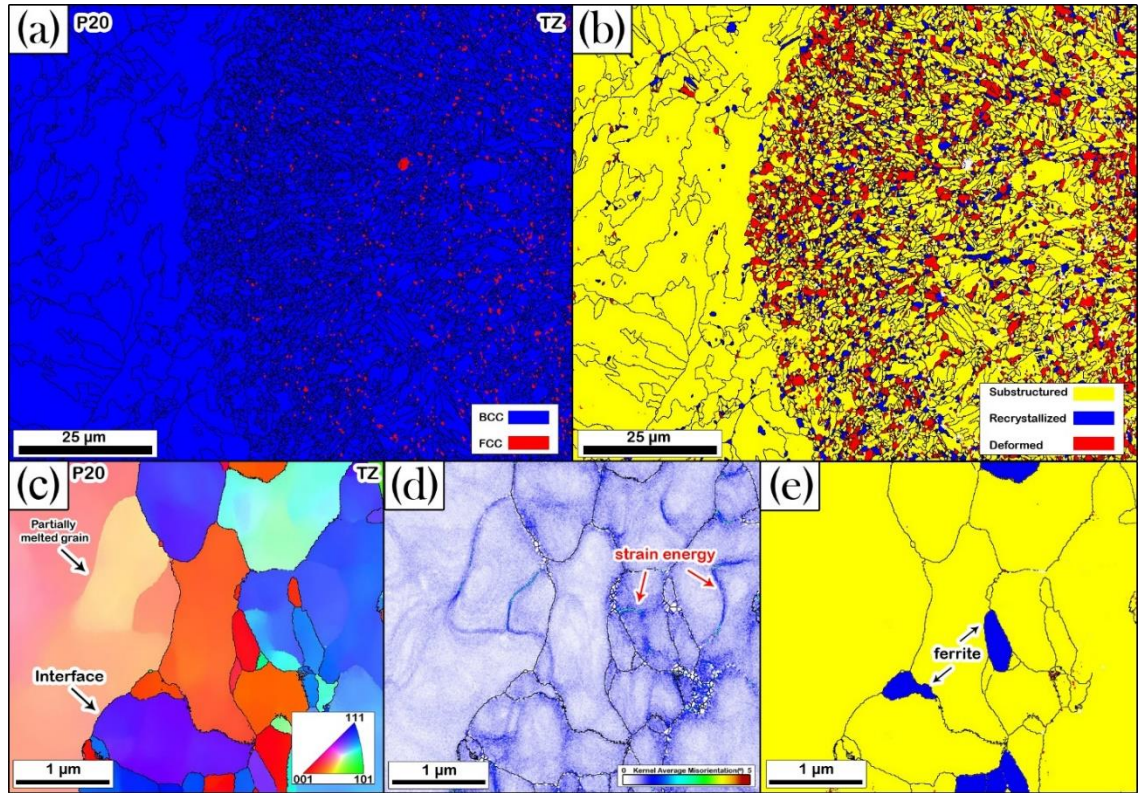


Figure 5-9: Medium magnification EBSD of the interface: a) Phase map; b) deformed grain map, High magnification EBSD of the interface: c) IPF; d) KAM; e) deformed grain map.

5.3.4 Mechanical Evaluation of the MS1-P20 Hybrid Steel

Figure 5-10 shows a 3D illustration of Vickers microhardness variations within the microstructure of MS1-P20 hybrid steel, where the Z-axis is the microhardness values. There is a steep rise in hardness values from P20 to TZ, reaching the maximum values in the TZ. The green area clearly highlights the interface. This hardness profile is typical of AM bimetal systems, where the additively manufactured side exhibits much higher hardness than the conventionally made substrate [28], [42].

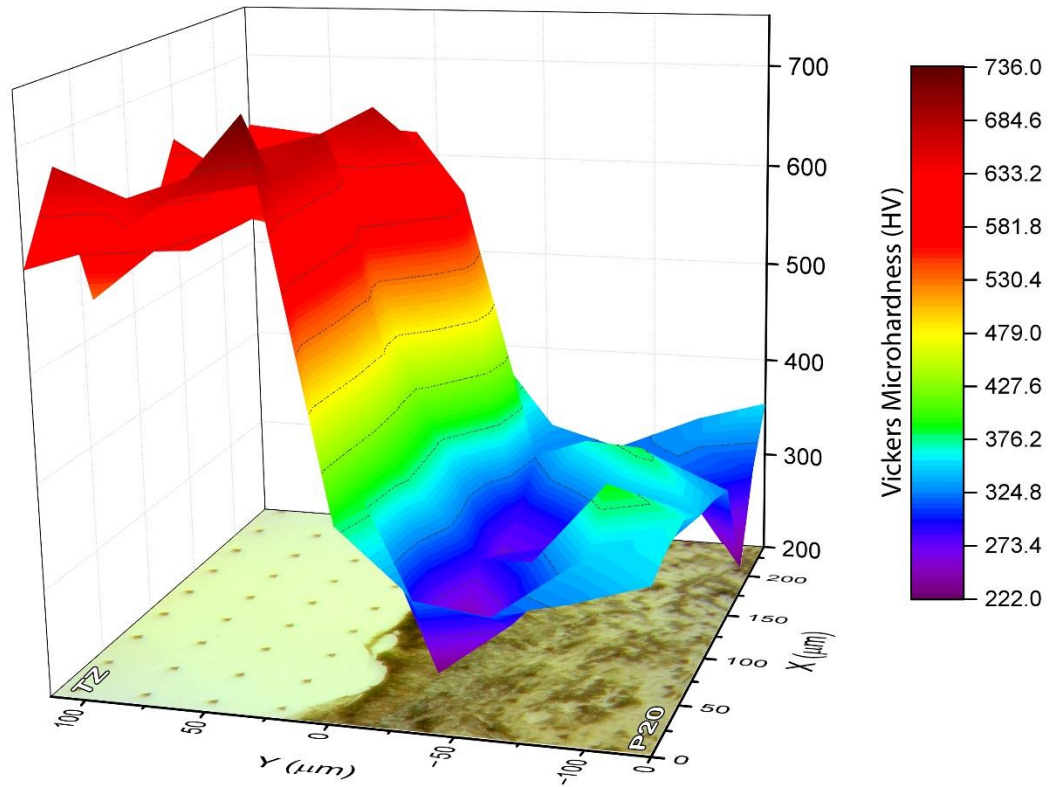


Figure 5-10: 3D illustration of Vickers microhardness values across the interface.

Figure 5-11 shows the DIC strain distribution for the MS1-P20 hybrid steel under the uniaxial tensile loading. The DIC system can measure the precise displacement of subset points on the tensile samples to calculate the strain field during the loading test [54], [55]. In the elastic region, the strain field is distributed uniformly throughout the sample ($\varepsilon = 0.005$). By moving to the initial plastic deformation region, the strain increased slightly in the P20 steel, while it remained unchanged in the PBF-MS1 ($\varepsilon = 0.020$). Further plastic deformation led to the initiation of necking with non-uniform strain distribution and increase of strain in the P20 steel ($\varepsilon = 0.038$), and finally, fracture occurred at the necking zone with the highest accumulated strain ($\varepsilon = 0.149$).

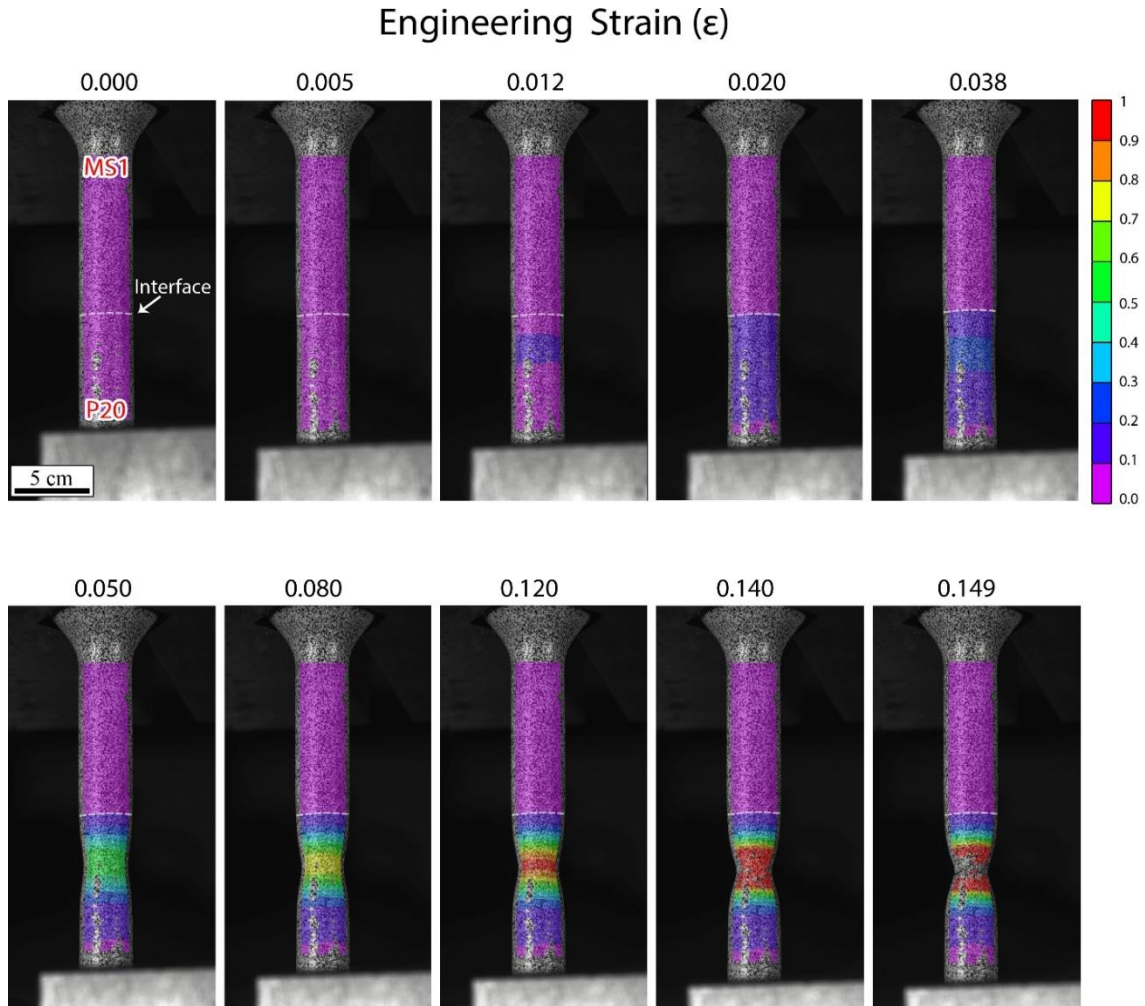


Figure 5-11: DIC representation of local strain field at different engineering strains.

The mechanical response of MS1-P20 hybrid steel under the tensile loading test is shown in Figure 5-12. The engineering stress-strain curve exhibits a ductile and robust profile with the ultimate tensile strength of 1039 MPa and fracture elongation of 14.9 % (see Figure 5-12 (a)). Thus, the MS1-P20 hybrid steel showed a relatively high toughness of 2.93×10^4 MPa. Besides, the failure occurred in the P20 steel and away from the interface, which is mainly due to the initiation of necking in P20 during the tensile loading. The true stress-strain curve up to the UTS, along with the rate of work hardening, is shown

in Figure 5-12 (b). The true stress-strain curve was obtained based on the following equations [56]:

$$\sigma = s(1 + e)$$

$$\varepsilon = \ln (1 + e)$$

where s and e are the engineering stress and strain, and σ and ε are the true stress and strain, respectively. These equations are valid up to UTS, where the specimen deforms uniformly [56], [57]. Then, the work hardening rate was calculated using the derivative $(d\sigma/d\varepsilon)$. Using the power law equation, the incident of σ and $d\sigma/d\varepsilon$ results in $\varepsilon = n$, thus the work hardening coefficient (n) is 0.035 for the MS1-P20 hybrid steel. The coefficient n indicates the hardening potentiality of the material upon plastic deformation. Since $n = 0.035$ is a low value coefficient, the MS1-P20 hybrid steel deforms with low strain hardening under uniaxial tensile loading.

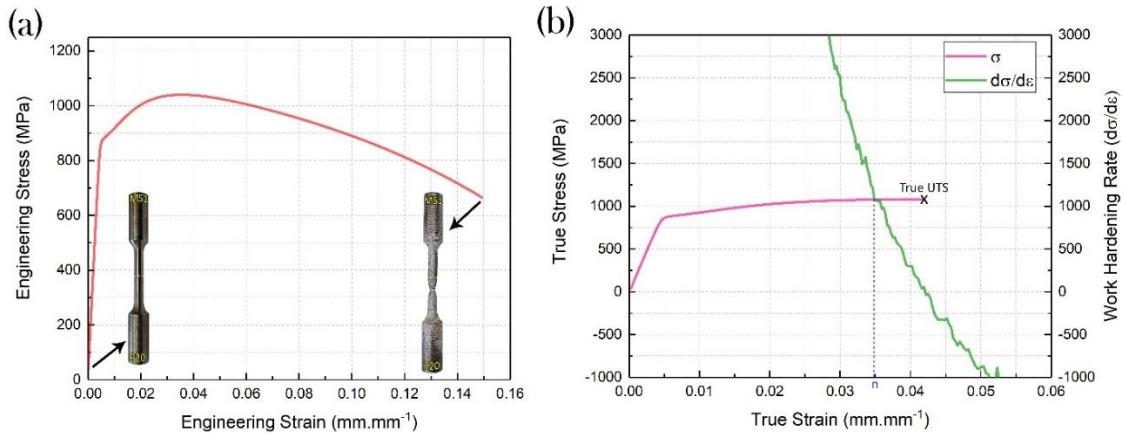


Figure 5-12: a) Engineering stress-strain curve; b) True stress-strain with work hardening curves.

Figure 5-13 shows the fracture surface of the tensile samples at different magnifications. The fracture mode looks ductile due to the presence of cup and cone configuration in Figure 5-13 (a). At higher magnifications, the central region mainly includes dimples resulting from tensile rupture (see Figure 5-13 (b)). High magnification observation revealed very fine dimples as the areas of high localized strains, where voids nucleate and aggregate to form the origin of fracture. Thus, the fracture occurred in ductile mode, either on the macroscopic or microscopic scale.

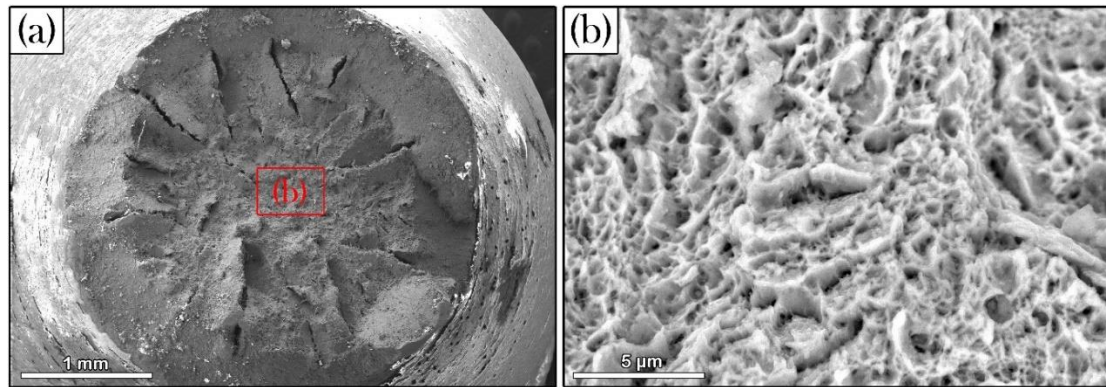


Figure 5-13: SEM fractography of the tensile samples at various magnifications.

5.4 Discussion

The results showed that printing MS1 on top of P20 is successfully feasible in terms of cohesion and mechanical strength. In this section, the mechanical behavior of MS1-P20 hybrid die is discussed regarding the evolved microstructure.

Since hybrid additive manufacturing requires bonding between two dissimilar materials, it is of great importance for the melt pool to wet the substrate. Figure 5-14 schematically shows the formation of a melt pool and the resultant microstructure at only one laser spot. Indeed, Figure 5-14 explicitly describes the formation of MS1-P20 hybrid

steel at a micro-scale. The laser's impingement on the first layer of MS1 powder forms a melt pool by melting the MS1 powder layer and a thin layer of the P20 block. In order to mix the two molten materials, a fluid flow needs to be generated. Once the melt pool is formed, the variation of solute concentration and temperature within the melt pool activates the Marangoni convection. This convection results from the temperature difference and surface tension, where the latter is related to the chemical composition. Thus the Marangoni convection can be expressed as a shear stress-dependent on two variables [14]:

$$\tau_m = \frac{d\gamma}{dT} \frac{dT}{dr}$$

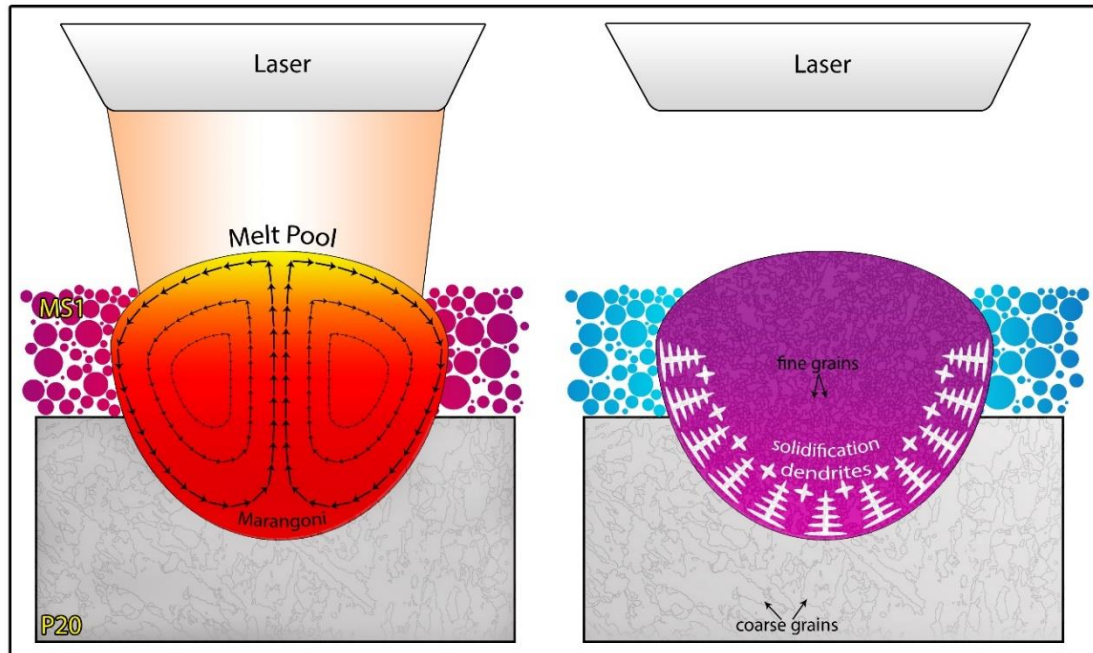


Figure 5-14: Illustration of melt pool formation in the MS1-P20 hybrid steel.

where the parameter γ is the surface tension, and $\frac{dT}{dr}$ is the radial temperature gradient from the laser beam. Because of the chemical composition difference between MS1 and P20, the surface tension (γ) is relatively high. On the other hand, the laser beam created localized

heat so that the temperature variation is considerable from the melt pool center to the edges. Therefore, the Marangoni stress is substantially high and can control the flow of molten powder within the melt pool, and mixing the alloying elements of both materials and subsequently promotes wetting the substrate.

Solidification begins with the nucleation of dendrites at the boundary between the melt pool and solid substrate. Despite epitaxial growth in additive manufacturing of bulk materials, hybrid additive manufacturing requires activation energy for nucleation at the first melt pool boundary [14]. Two compositionally different materials are involved for the MS1-P20 hybrid steel; hence there should be a constitutional supercooling for the activation of heterogeneous nucleation at the melt pool boundary [58]. Since the solidification mode is dendritic (see Figure 5-3 (b)), the constitutional supercooling seems relatively high at the melt boundary [59]. Therefore, heterogeneous nucleation is activated at the melt boundary forming equiaxed solidification dendrites on top of the P20 substrate.

By moving the laser beam, the nuclei start to grow due to constitutional supercooling, temperature gradient (G), and solidification growth rate (R). The last two parameters play a significant role in the determination of the solidification mode and the scale (size) of solidified microstructure [14], [51], [60]. The mode is a function of the ratio G/R whereas the scale is related to $G \times R$. For most bimetal steels, a structure with almost equiaxed cells was observed. An identical solidification structure was also formed in the MS1-P20 hybrid steel. A comparison was made between two materials with different heat conductivities to better understand the effect of G/R on the solidification mode. The PBF-aluminum and -maraging steel bimetals were selected for this purpose. For the aluminum bimetal, fine columnar dendrites were developed at the interface [28], while the maraging

steel bimetal contained mostly equiaxed dendrites [42]. Since aluminum is a more conductive material than steel, it creates a higher temperature gradient (G) at the interface, and hence the ratio G/R increases for the aluminum sample leading to a more columnar structure while the maraging steel with lower G/R solidifies with more equiaxed dendrites.

After solidification, the typical additive manufacturing morphology with overlapped melt pools is formed on P20, featuring a gradual transition of chemical composition from P20 to MS1. The first deposited layer is highly affected by dilution; however, this effect fades in the upper layers, as illustrated in Figure 5-6. The presence of dilution promotes wetting and subsequently bonding the substrate. Thus, a cohesive and defect-free joint is formed between MS1 and P20.

The EBSD results indicate the formation of a bimodal grain morphology across the interface, including the solidification of fine grains on top of the P20 steel with larger grains. This implies the fact that grain growth is no longer affected by the substrate morphology, and non-epitaxial grain growth occurs during the process. By comparing Figures 5-4 and 5-7, both P20 and MS1 regions seem to preserve their primary microstructure since the grain size and shapes are comparable to those mentioned in Figures 5-4 (a) and 5-4 (b). This is particularly beneficial to the P20 steel since it shows that the substrate P20 was not affected by the PBF process, and no unfavorable evolution occurred in terms of grain morphology and strain energy within the P20 microstructure. A similar feature was also reported for MS1-H13 bimetal printed using the direct metal laser sintering (DMLS) method [42].

The transition zone (TZ) adjacent to the interface forms an ultrafine and complex grain morphology. Three phases coexist at this region including martensite, retained

austenite and ferrite. The martensite is the dominant phase and contain two types of grains as substructured and deformed, where the latter one involves higher misorientations, and thus higher dislocation density. On the other hand, the ferrite grains seem to be soft and ductile due to their lowest strain energy. The versatility of phases within TZ can impede deformation due to the difference between slip systems of BCT, BCC, and FCC structures. Indeed, such difference can constrain the plastic deformation between the grains. In addition, the ultrafine grains of TZ can act as obstacles for the movement of dislocations. In other words, the finer grain morphology of TZ with respect to that of P20 actuates the Hall-Petch effect and hence adds to the strength of interface. The higher density of dislocations detected via KAM mapping, triggers forest dislocation strengthening mechanism [61]–[63], which hinders the plastic deformation in the TZ (see Figure 5-7 (b)). The aforementioned strengthening mechanisms also generate higher microhardness contours in the additive side as highlighted in red, meaning this area is highly resistant to plastic deformation. Furthermore, the soft P20 steel allows more plastic deformation to occur, leading to lower hardness values. The P20 steel was detected as the weak zone in the MS1-P20 hybrid steel due to the coarser grains with lower dislocation density.

Upon uniaxial tensile loading, relatively uniform deformation developed throughout the sample in the elastic region. The increase of loading initiated plastic deformation in P20, while MS1 still remains in the elastic region. Further loading caused the concentration of plastic deformation in P20 along with initiation of necking, while MS1 behaved elastically up to fracture. As stated earlier, P20 features a low density of dislocations with a coarse grain morphology encouraging dislocations to move more frequently, and subsequently, more plastic deformation occurs. Dislocations continue their

movement until they reach a grain boundary. At this point, they are no longer moveable and start to accumulate behind the grain boundary, leading to dislocation pile-ups. Since P20 contains very large grains, the extent of dislocation pile-ups is relatively considerable, resulting in a superdislocation within a grain [57]. The stress field of superdislocation induces a void at the grain boundary, which grows under further tensile loading as shown in Figure 5-11. The growth and coalescence of voids finally cause the ductile failure of MS1-P20 hybrid steel from the P20 side. Therefore, the failure of MS1-P20 hybrid steel is dependent on dislocation motion, pile-ups, and subsequent void formation.

It was well shown that the interface behaved stronger than the P20 steel securing the additive side of the hybrid steel from failure under uniaxial tensile loading. This achievement enables the MS1-P20 hybrid steel to be used as a new generation of dies in the tooling dies industry provided that the safety factor related to the P20 substrate is satisfied.

5.5 Conclusion

This work attempts to fabricate a hybrid steel using the powder bed fusion (PBF) technique. The soundness of the interface between the PBF-MS1 and P20 substrate was investigated, which yields the following consequences:

- A highly cohesive bonding was formed between MS1 and P20. At the first few layers, the two materials were mixed adequately as a result of Marangoni convection; successful layers were deposited without developing detrimental defects including cracks and porosity.

- A cellular-dendritic solidification structure was developed due to the microsegregation of alloying elements, the interface's temperature gradient and cooling rate. Since the latter parameter was fast, a very fine structure containing submicron cells was formed as well as a fine grain structure.
- Several phases were formed on top of the P20 comprising of mainly martensite, and small fraction of austenite and ferrite. This multi-phase microstructure plays a prominent role in strengthening the interface as a result of hindering the plastic deformation between the phases. Moreover, the classic Hall-Petch effect, together with forest dislocation hardening, positively contributed to the strength of the interface.
- The MS1-P20 hybrid steel exhibited a ductile behavior under uniaxial tensile loading and did not fail at the interface. This approach explicitly shows the unique feature of the PBF technique in the fabrication of super-strong joint between dissimilar steels.

5.6 References

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6 On the Bending of MS1-P20 Hybrid Steels Additively Manufactured Using Laser Powder Bed Fusion

6.1 Introduction

Metal additive manufacturing (MAM) technologies have been restructuring the concept of manufacturing and establishing new infrastructure for the next generation of metal part productions. Many engineering challenges are being facilitated in the production of advanced materials as well as intricate shapes. Most industries are now benefiting from the novel opportunities that MAM can offer them. As an illustration, complex alloys, functionally graded materials, and miniature structures were produced using MAM methods for marine applications [1]–[6]. In the aerospace industry, extensive research has been conducted, where lighter materials with superior efficiency were manufactured at lower costs and lead time [7], [8]. Many alloys such as titanium, nickel, aluminum alloys, and many grades of steels and stainless steels were also processed via the MAM platform, and unique microstructures with superior properties were achieved [9]–[17].

In today's manufacturing world, the concept of novel materials is becoming significant due to complex working conditions growing every day. This requires new ideas of processing raw materials to create innovative compounds. Of those innovations, bimetals have overcome the limitations of conventional alloys and brought new capabilities in engineering applications [18]. However, conventional manufacturing techniques cannot fully provide satisfactory conditions to produce bimetals. For instance, welding procedures have been extensively used, but defects such as HAZ, solidification cracking, and embrittlement are unavoidable. Instead, MAM has recently introduced a practical approach

to manufacture these materials due to the layer-by-layer buildup of feedstock materials inherent in additive manufacturing.

Steel-bronze, Inconel-stainless steel, 17-4 PH-316L, Fe-Al12Si, Ti6Al4V-IN718 are some recent examples of bimetals that were prepared fully via additive manufacturing [19]–[23]. The bimetallic systems can also be manufactured using a hybrid approach [24]. In this case, a conventionally manufactured alloy is used as a substrate, and then a powder feedstock is deposited on top of it. This is called hybrid additive manufacturing (HAM), which combines subtractive manufacturing or forming methods with the MAM techniques [24]. Several HAM bimetallic systems were studied recently, including Ti6Al4V-316L, AlSi10Mg-AA2618, NAB-316L, and steel-steel [25]–[30]. One of the most investigated bimetals is maraging steel bimetals since maraging steel is a material of interest in laser powder-based MAM techniques [31]–[34]. It was reported that a free-defect interface with excellent mechanical strength is the key characteristic of maraging steel bimetals, making them ideal candidates for high-temperature applications [30], [35]–[38].

Since bimetals usually involve two dissimilar metal components, various alloy combinations have been studied for this purpose. Thus, the bimetallic systems of interest can become widely diverse, and each case requires detailed research. Of particular importance is an MS1-P20 system as a new maraging steel bimetal with potential applications in the tool and die industry for the automotive sector. This bimetallic system involves a conventionally made P20 steel and an additively manufactured maraging steel called the MS1-P20 hybrid steel. Bimetals involve dissimilar alloys with vastly different chemical compositions, and microstructures can possess unbalanced mechanical properties negatively affecting their performance. Recently, MS1-H13 bimetals were produced using

the laser powder bed fusion (LPBF) technique, where different post-processing techniques were employed to homogenize their interfacial characteristics [35], [39]. The effect of different heat treatment procedures on maraging steel-based bimetal was studied by other researchers as well; see, e.g. [40], [41].

Most maraging steel-based bimetal used in the tool and die application are associated with severe working conditions, where complex loadings exist. Most studies available in the open literature on these AM bimetal were focused on their typical uniaxial tensile behavior, which involves the simplest loading condition far from realistic service environments. Only a few examples can be found, including the flexural test on 316L-Hastelloy X and Al10SiMg-C18400 bimetal [42], [43]. Flexural testing regimes can generate loading conditions that both tension and compression situations that can simultaneously occur on a specimen. Therefore, both types of flexural tests in this work, including the three-point (3P) bending and four-point (4P) bending, were considered for AM MS1-P20 hybrid steels. This way, a more sophisticated evaluation of the mechanical properties using 3P and 4P bending tests were proposed. In addition, modifying the microstructural characteristics of MS1-P20 hybrid steels via proper heat treatment sequences with the knowledge learned from MS1-H13 bimetal was of particular interest. Using the heat treating procedures, the microstructural profile across the AM MS1-P20 hybrid steels interface was customized.

6.2 Experimental Procedure

Two dissimilar materials were used for manufacturing the MS1-P20 hybrid steel: an as-received P20 block as the build plate and maraging steel MS1 powder as the

feedstock material. The specifications of these two alloys are shown in Table 6-1. The hybrid steel was manufactured using the LPBF process. For this purpose, an EOS M290 additive machine was utilized, which features a Yb-fiber laser with maximum laser power of 400 W. The P20 block was screwed to the additive machine and preheated to 40 °C. The printing chamber was filled with an argon gas (99.9% purity) to avoid oxidation during the LPBF process. The first MS1 powder layer with a thickness of 40 μm was uniformly spread on the P20 build plate. The laser started to scan and fuse the powder layer at a speed of 960 mm/s. The laser scanning was pursued with a strip-like strategy, and the laser beam rotated 67° after completing each individual layer. The hatch distance and spot sizes were 0.11 mm and 100 μm , respectively. The final round bar samples were carefully extracted from the additive machine and cut for the heat treatment procedure. Two heat treatment cycles were carried out sequentially: 1) hardening at 880 °C for 1 hour and 2) aging at 500 °C for 2 hours. The MS1-P20 samples were correspondingly classified as as-built (AB), hardening treated (HT), and hardening and aging treated (HAT).

Table 6-1: Specification of the raw materials; alloying elements are in weight percentage (wt.%).

Maraging steel MS1 powder									
C	N i	Co	Mo	Ti	Mn, Si	Al	C r, Cu	P, S	Particle Diameter
≤ 0.03	$^{17}_{-19}$	8.5-9.5	4.5-5.2	0.6-0.8	≤ 0.1	0.05-0.15	≤ 0.5	≤ 0.01	1 – 45 μm
As-received P20 block									
C	Mn		Cr		Si	Mo		Dimension	
0.28-0.40	0.60-1.00		1.40-2.00		0.20-0.80	0.30-0.55		150 × 70100 mm^3	

The microstructural samples were sectioned across the boundary between P20 and MS1 and were polished for electron microscopy investigations. Table 6-2 shows three types of electron microscopy techniques used to develop a comprehensive vision of various aspects of the produced microstructure. The electron backscatter diffraction (EBSD) data was collected via Aztec software, and data processing was carried out using HKL Channel 5 and OIM softwares. The prior austenite grains (PAGs) were reconstructed from the AB, HT, and HAT samples based on the approach proposed in [44], [45]. TEM microscopy requires a particular sample preparation, so that, the focus ion beam (FIB) milling technique was carried out via an FEI Helios Nanolab 650 dual-beam system. Several microhardness tests were also conducted on the microstructural samples. A Vickers microhardness machine was utilized, in which the indenter loading and strain rate were set to 50 mN and 0.2s^{-1} , respectively.

Table 6-2: Electron microscopy techniques and equipment were used to study MS1-P20 hybrid steels.

Scanning Electron Microscope (SEM)			Transmission Electron Microscope (TEM)	
Microscope	Detector		Microscope	Detector
Thermo Fisher Scientific / FEI Scios 2	EDS	EBSD	FEI Tecnai Osiris	EDS
	Oxford Energy Dispersive Spectrometer	Oxford Symmetry		Brucker quad-core X-ray EDS

The bending tests were conducted in two different formats according to ASTM E290 Standard [46]: 1) three-point (3P) bending and 2) four-point (4P) bending tests. A specific fixture was designed and fabricated in each test as illustrated in Figure 6-1. The round bar samples were machined to cubic shapes, where the interface was located at the center. Both bending tests were performed at a constant crosshead speed of 5 mm/min, and

the data were collected as force-displacement plots. The bent samples were also sectioned at the interface and used for further microscopy via EBSD and TEM.

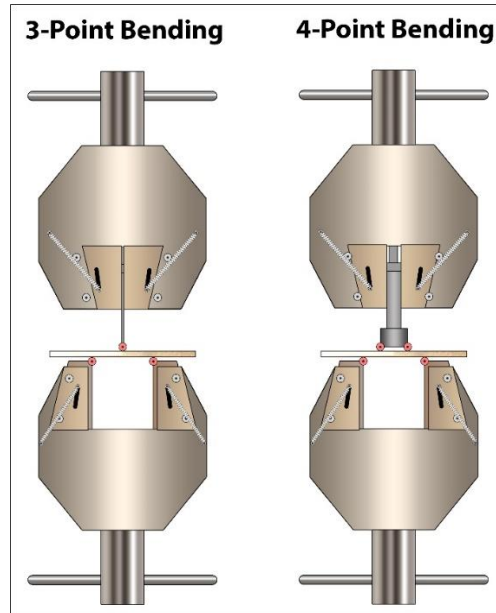


Figure 6-1: Schematic of 3P and 4P bending tests

6.3 Results

6.3.1 Heat Treatment Selection

The fabricated MS1-P20 hybrid steel consists of two different materials, and this difference can lead to more complicated criteria for heat treatment. Thus, the following strategy was used as the selection criteria. The typical heat treatment cycles for both MS1 and P20 were first evaluated, and identical cycles were designed and carried out on both materials. The microhardness test was then conducted to select the appropriate heat treatment for the MS1-P20 hybrid steel. P20 mold steel can be commonly hardened within the temperature range of 850-880 °C and tempered within 200-600 °C [47]. For MS1, the temperature ranges of 800-900 °C and 400-600 °C were reported as the solution and aging treatments, respectively [48]. Thus, the P20 hardening temperature of 880 °C and MS1 aging temperature of 500 °C were considered for the MS1-P20 hybrid steel.

Figure 6-2 illustrates the microhardness variation for the as-built and heat-treated samples. The as-built sample involves a very soft substrate of 325 HV as expected and a very hard area of 475 HV right above the interface. The hardness then decreases below 400 HV, reaching the hardness value of as-built MS1. In HT condition, the average hardness surprisingly escalated to 625 HV in P20, and then a sharp drop occurred from the interface to MS1, which was softened with values around 300 HV. In HAT, the hardness values drop in the substrate, while there is a remarkable rise in hardness of the additive side with respect to the HT-sample. Despite the extreme variation of hardness in AB- and HT- samples, the HAT-sample exhibited a relatively homogeneous hardness gradient from P20 to MS1. The hardening followed by aging treatment (HAT) can produce more uniform properties than HT treatment; therefore, the HAT-sample was selected for further investigations.

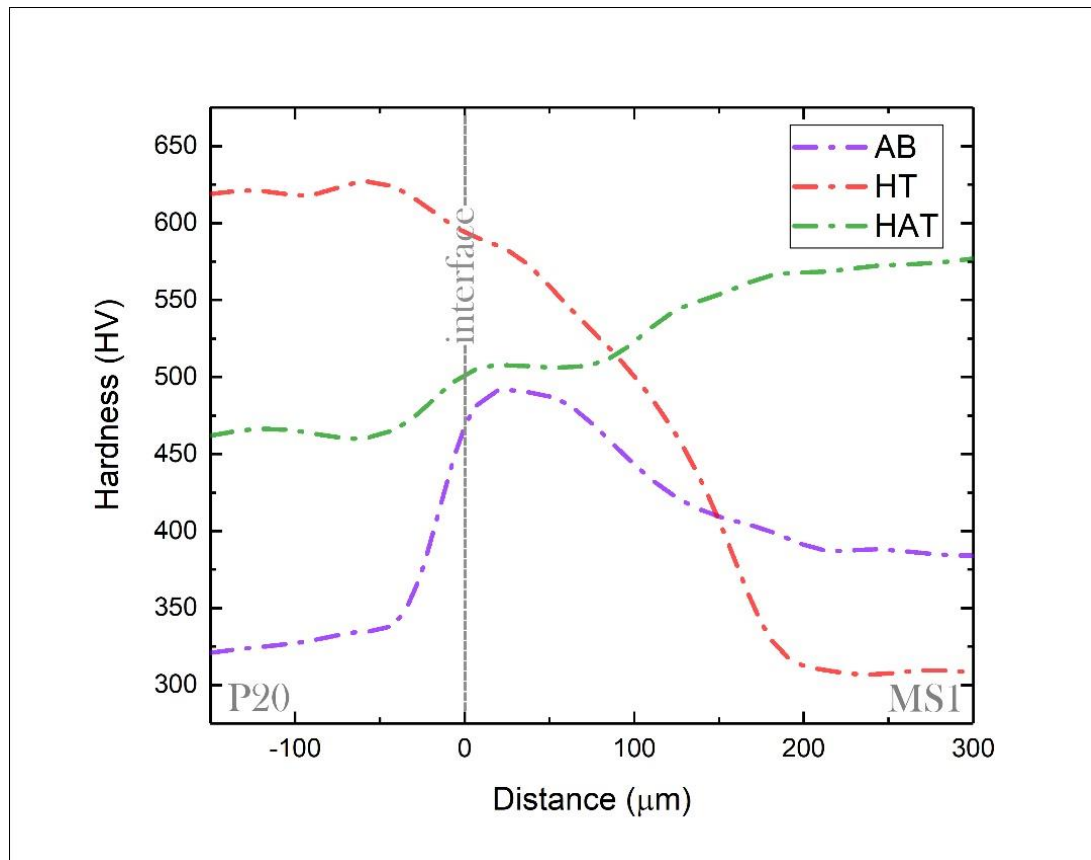


Figure 6-2: Microhardness gradient across the MS1-P20 interface

6.3.2 Heat-treated bulk materials

The microstructural effect of HAT sequence on both MS1 and P20 bulk materials is first investigated via SEM and EBSD techniques and presented in Figures 6-3 and 6-4. The as-built MS1 contains a hierarchical structure, where groups of ultrafine solidification cells form a grain (see Figure 6-3 (a)). However, the HAT sequence removed the solidification structure and seemed to form some fine precipitates (see Figure 6-3 (b)). Figures 6-3 (c)-(d) illustrate the BD-IPF maps of MS1 before and after the heat treatment. The additive MS1 did not exhibit any dominant color in the IPF maps. The corresponding pole figures confirm that there is no preferred orientation; thus, the heat treatment did not cause any texture in MS1. The IPF maps also indicate that a considerably coarse grain morphology can be developed via the HAT sequence in MS1. The grain boundary maps are also shown in Figures 6-3 (e)-(f) to illustrate the grain boundary evolution. The grain boundaries can be categorized as very low angle boundaries (VLAB) with rotation angles less than 5° , low angle boundaries (LAGB) of $5\text{-}15^\circ$ misorientation angle, and high angle grain boundaries (HAGB) with misorientations above 15° [49].

The VLABs represent strain energy inside grains, and the total length of VLABs decreased from 8.02 cm to 5.42 cm upon the HAT sequence, indicating that MS1 is relatively in less strain energy state than the as-built condition. LAGBs mark the subgrain structure in green color, where a higher fraction of LAGBs was formed in the heat-treated sample. The HAT sequence increased the total length of LAGBs almost six times the as-built value from 1.43 cm to 9.97 cm. The as-built MS1 contains longer HAGBs of 3.06 cm

compared to 1.94 cm in the HAT-MS1, which is due to more refined grains of the as-built MS1. Figures 6-3 (g) and 6-3 (h) demonstrate the grain size distribution and aspect ratio, respectively. The quantitative values of these two figures are also presented in Table 6-3. In Figure 6-3(g), the AB profile shows an uneven distribution of grains, while the grain area distribution seems to be uniform since the HAT profile shows a typical Gaussian distribution. Thus, the HAT sequence caused a homogenous grain morphology in MS1. In addition, this heat treatment sequence caused a grain growth from $98.50 \mu\text{m}^2$ to $106.03 \mu\text{m}^2$ in the printed MS1. The aspect ratio graphs shown in Figure 6-3 (h) indicate that most grains are equiaxed in both conditions. There is not much difference in fraction values of columnar to equiaxed grains calculated in Table 6-3.

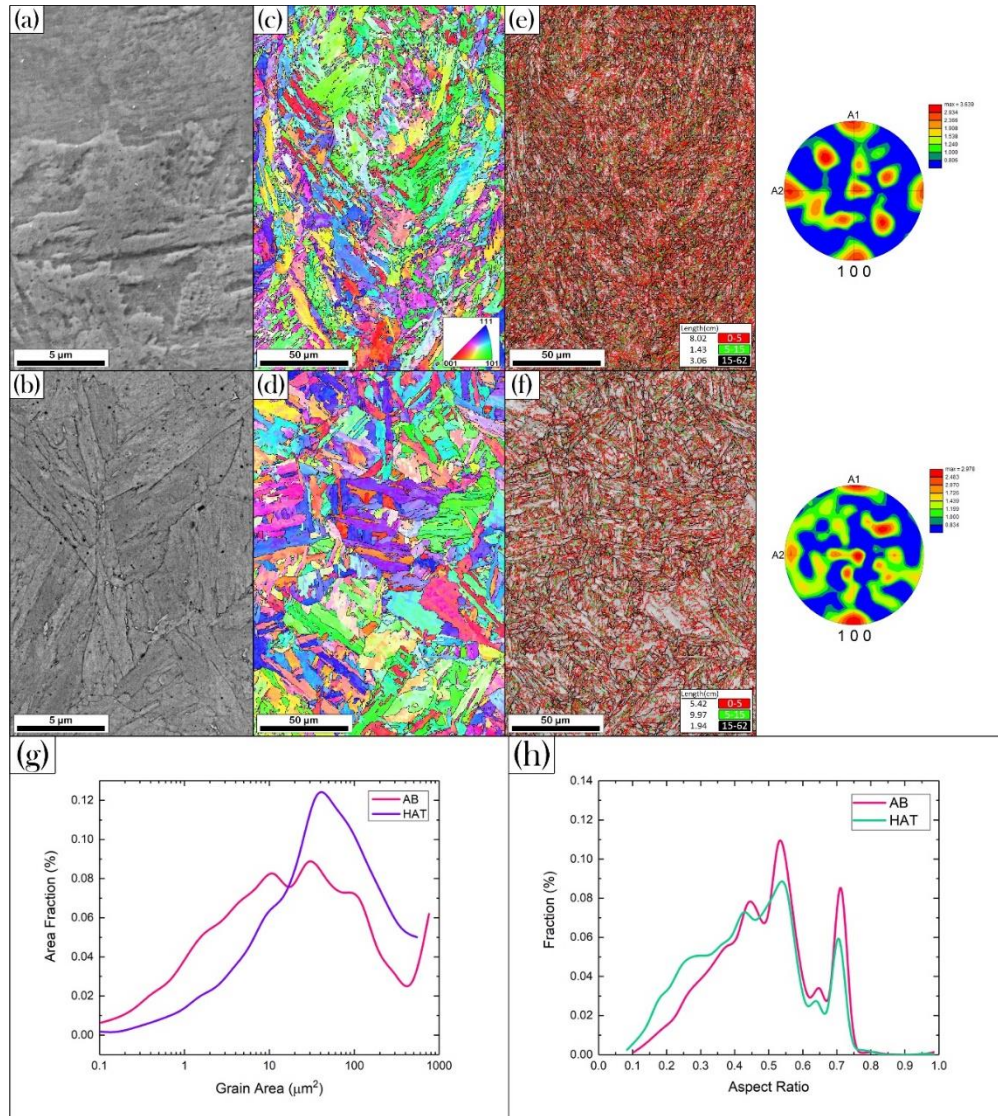


Figure 6-3: SEM micrographs: a) AB b) HAT; BD-IPF of MS1: c) AB d) HAT; Grain boundary mapping and corresponding pole figure of MS1: e) AB f) HAT; g) MS1 grain size distribution; h) MS1 Aspect ratio

Table 6-3: MS1 grain specification before and after heat treatment

Sample	Grain Area(um2)	Aspect Ratio (%)		KAM(°)
		Columnnar	Equiaxed	
AB	98.50	12	88	1.17
HAT	106.03	16	84	0.86

The same analysis was carried out on P20 in AB and HAT conditions, as presented in Figure 6-4. The as-received P20 was filled with rough precipitates, mainly Cr-rich and

Mn-rich carbides [50]. These precipitates seem to become refined upon the HAT sequence within the P20 grains (see Figure 6-4 (b)). The BD-IPF map of as-received P20 shows a relative dominance of blue colors in [111] direction, and the corresponding pole figure proves the high density of poles for [111] planes. It seems that the P20 substrate contained a texture in the as-received condition. This texture, however, was removed when the HAT sequence was applied on the P20 steel since the IPF map of Figure 6-4 (d) shows a random color distribution, and the corresponding pole figure indicates a low value of 1.916 MRD (multiples of random density) as the maximum intensity. Regarding grain boundaries, HAGBs, as well as LAGBs, almost doubled after the HAT sequence, whereas VLABs remained the same (see Figures 6-4 (e) and (f)). The HAT sequence refined the grains and formed a more profound subgrain structure within P20.

Figure 6-4 (g) clearly shows that the as-received P20 contains two main peaks, including coarse and very large grains, while the HAT-P20 peak is located within much lower values. In addition, the HAT profile is demonstrated as a normal distribution; thus, the grains seem to be uniform in P20. In other words, Figure 6-4 (g) is indicative of grain refinement and grain size homogenization upon the HAT sequence in P20. The average grain area for the AB and HAT conditions was calculated to be $160.29 \mu\text{m}^2$ and $8.88 \mu\text{m}^2$, respectively (see Table 6-4). In terms of aspect ratio, no tangible change was observed after heat treatment, and the majority of grains remained equiaxed within P20 (see Figure 6-4 (h)).

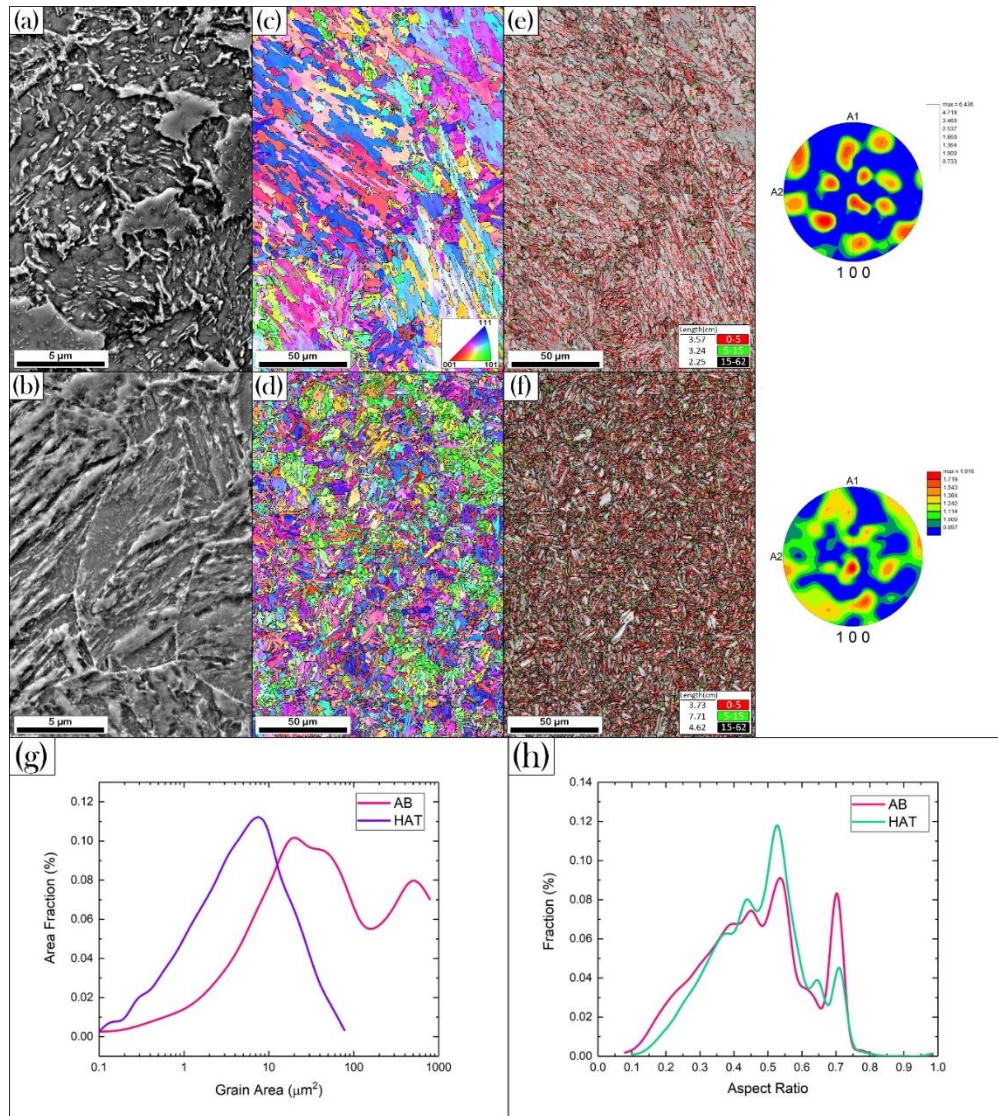


Figure 6-4: SEM micrographs of P20: a) AB b) HAT; BD-IPF of P20: c) AB d) HAT; Grain boundary mapping and corresponding pole figure of P20: e) AB f) HAT; g) P20 grain size distribution; h) P20 Aspect ratio

Table 6-4: P20 grain specification before and after heat treatment

Sample	Grain Area(um2)	Aspect Ratio (%)		KAM(°)
		Columnar	Equiaxed	
AB	160.29	13	87	0.69
HAT	8.88	12	88	0.71

6.3.3 Heat-treated interface

Figure 6-5 shows the SEM micrograph of the MS1-P20 interface in the as-built and heat-treated conditions, as well as EDS line analysis across the samples. The AB-sample contains coarse ferritic grains on the substrate side, and a few fractions of solidification cells with the trace of the Marangoni effect can only be seen on the additive side (see Figure 6-5 (a)). Figure 6-5 (b) shows the MS1-P20 interface after the HAT sequence, where the substrate underwent martensite transformation. Although the microstructure of the additive side is no longer visible, it can be concluded that the HAT sequence removed the Marangoni effect above the interface. Figure 6-5 also presents the profile of main alloying elements across the interface area. Ni and Co are the MS1 alloying elements, and Cr and Mn are the main alloying elements of P20.

Overall, there is an ascending gradient for Ni, Co, and a descending gradient for Cr, Mn at the interface indicating the transition zone (TZ). The elemental gradients look identical in both AB and HAT conditions and do not seem to vary significantly. However, there is a remarkable rise in Ni and Co concentrations along with a Cr decrease in the HAT-interface. This is the explicit indication of diffusion associated with heat treatment at the interface, where the elemental variations are prominent.

Figure 6-6 shows the band contrast and BD-IPF maps of MS1-P20 interfaces before and after heat treatment. The band contrast maps distinguish different areas across the interface. Based on grain size variation, three main areas can be observed: substrate, transition zone (TZ), and fully MS1 zone. In as-built condition, the interface emerged by very fine grains of TZ solidified on top of large grains of the substrate. The transition zone

(TZ) continued to grow about 150-200 μm , and then the maraging steel smoothly appeared above TZ, as highlighted in Figure 6-6 (a).

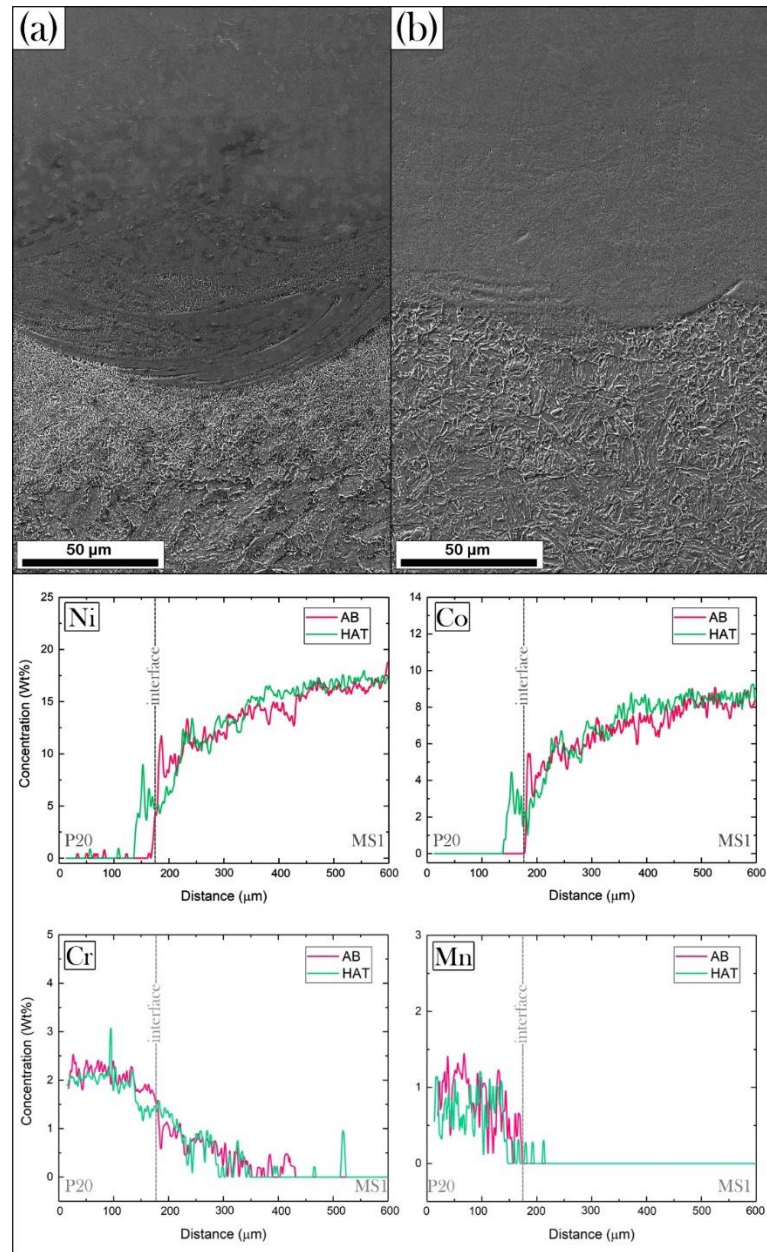


Figure 6-5: MS1-P20 interface micrographs: a) AB b) HAT and corresponding elemental variation

Since very fine grains were developed on the substrate, the interface seems highly distinguishable in the as-built condition. The heat-treated interface, however, seems less

detectable due to remarkable grain refinement of the substrate, as can be seen in Figure 6-6 (b). The heat-treated TZ seems to be coarser than the as-built one, but the grain growth is more noticeable on MS1 after heat treatment. Figures 6-6 (c) and (d) demonstrate the grain orientation in terms of inverse pole figures along the building direction. Random color distribution can be easily detected across the interface in both conditions, so no texture was developed in the HAT sequence. Interestingly, the grain contrast seems to be faint at the HAT-interface since the grain size difference is subtle between P20 and TZ (see Figure 6-6 (d)).

Figure 6-7 represents the strain analysis of the interface in terms of kernel average misorientation (KAM) and grain orientation spread (GOS). The KAM maps demonstrate the stored strain energy inside grains associated with dislocation density [51], [52]. In contrast, GOS measures an average value and assigns a color to an individual grain as an indication of strain [53]–[55]. Figures 6-7 (a) and (b) reveal the apparent distinction between the substrate and additive side. The additive side is usually highlighted in a brighter color; thus, KAM mapping can be a good candidate to recognize the interface area.

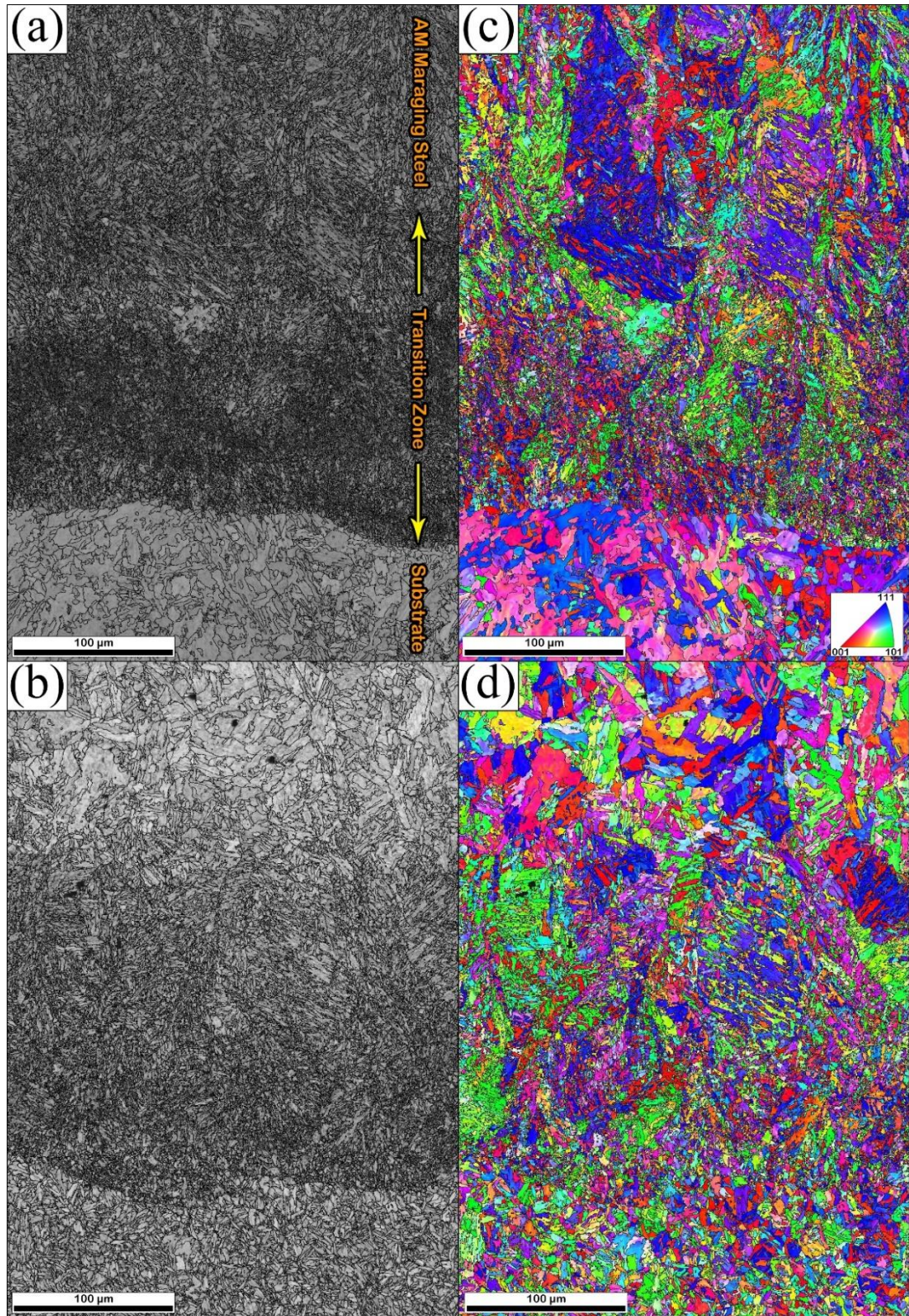


Figure 6-6: Band contrast: a) AB b) HAT; BD-IPF map: c) AB d) HAT across the interface

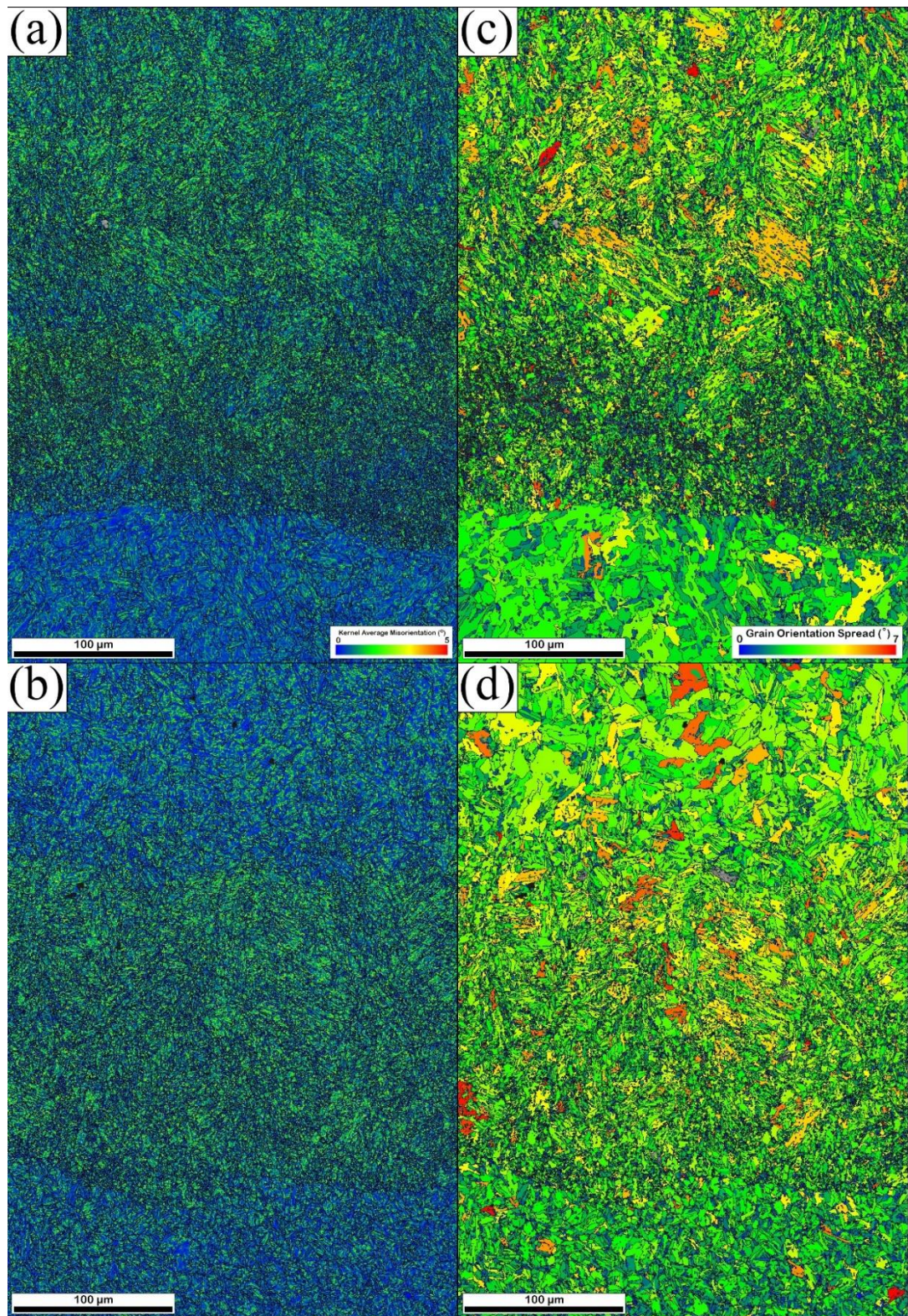


Figure 6-7: KAM mapping: a) AB b) HAT; GOS mapping: c) AB d) HAT across the interface

The GOS maps also display the average misorientation value of each grain. In the AB-interface area, TZ contains lower GOS values than P20 and MS1 since the blue grains are considerable in this area. There exists a non-uniform distribution of GOS misorientation across the interface (see Figure 6-7 (c)). The heat-treated interface, however, seems more homogeneous in this regard (see Figure 6-7 (d)).

The MS1-P20 interface features a very fine morphology; thus, further investigations are necessary at higher magnifications. Figure 6-8 (a) shows the SEM micrograph of the HAT-sample at 5000X, where the morphology contrast between the transition zone and P20 forms the interface. The P20 microstructure is clearly detected with martensitic grains, and very fine precipitates are embedded within the grains. The transition zone (TZ) no longer involves the solidification structure of as-built condition, and interestingly very fine precipitates can be observed. High magnification EBSD mapping of the HAT-interface is shown in Figure 6-8 (b), where the grains are randomly orientated across the interface. A smooth transition can also be seen in terms of grain size, in which coarser grains turn to finer grains by approaching TZ. Figure 6-8 (c) also shows the grain boundary morphology of the HAT-interface. Likewise, the coarse-to-fine transition of grains can also be viewed in this figure since more VLABs, LAGBs, and HAGBs were accumulated in TZ rather than P20.

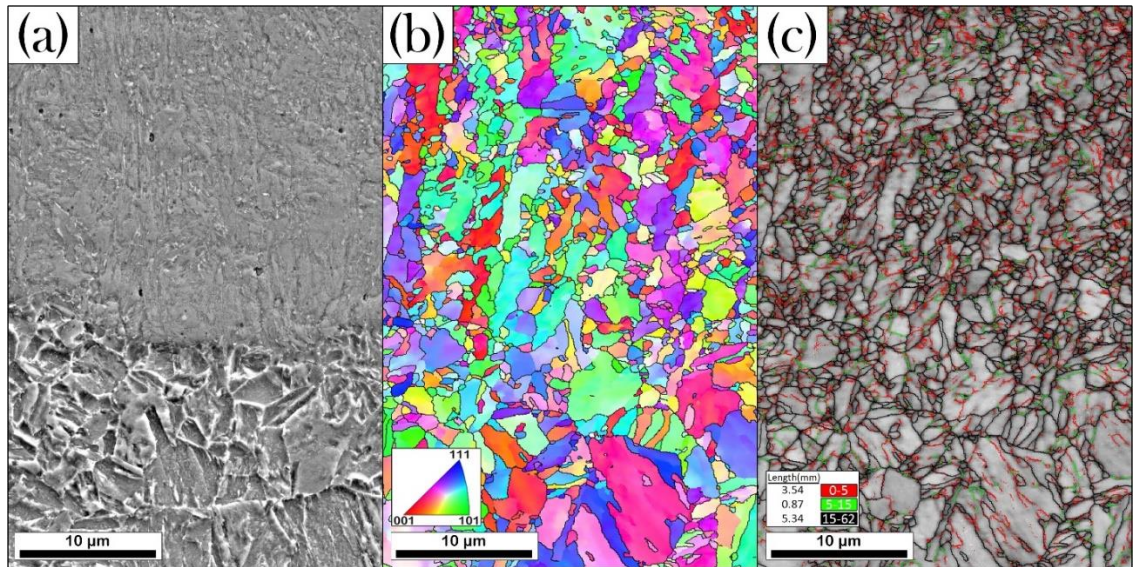


Figure 6-8: a) SEM micrograph b) BD-IPF map c) grain boundary map across the interface at high magnification.

The very fine precipitates of HAT-interface detected in high-magnification SEM imaging require precise analysis using the TEM/EDS technique. Figure 6-9 illustrates the elemental mapping of the HAT-sample right at the interface. This figure reveals the ultrafine details of the interface on the nanoscale. The HAT-interface constitutes a very narrow band of few nanometers between P20 and TZ. Ni and Co, as the main alloying elements of MS1, coexist on both sides of the interface, which confirms the diffusion induced by heat treatment and, moreover, it caused the enrichment of the interface with these two elements. Hence, the HAT-interface is a Ni-and Co- rich solid solution. In addition, nano precipitates rich in Cr, Mn, and C were accommodated along the interface; thus, Cr and Mn carbides can be considered the other constitution of the HAT-interface. Another type of precipitate can also be seen near the interface, mainly in TZ. These precipitates are AlTi intermetallic compounds reported in AM maraging steel [56]. It seems that the MS1 alloying elements constructed the matrix of the interface, and the P20 alloying

elements pinned the interface by forming nano precipitates. Therefore, superior mechanical properties can be expected for the HAT-interface.

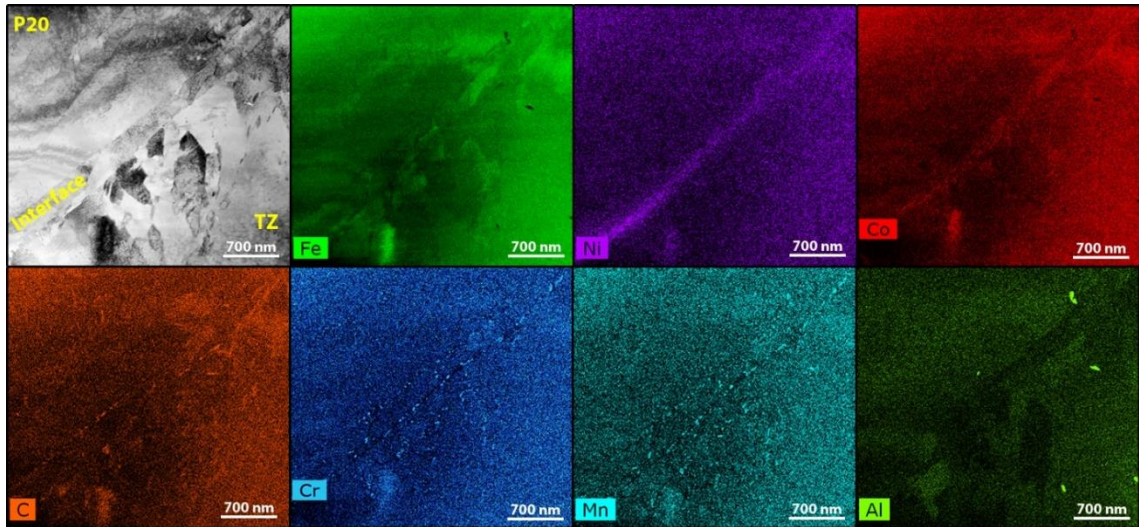


Figure 6-9: STEM-EDS elemental analysis of HAT-interface

6.3.4 Flexural performance of MS1-P20 hybrid steel

The mechanical performance of the MS1-P20 hybrid steel was assessed under complex loading conditions. Accordingly, three-point (3PB) and four-point bending (4PB) tests were applied on both AB and HAT specimens. Figures 6-10 (a) and (b) demonstrate the load-displacement curves for 3PB and 4PB tests, respectively. The three-point test was performed until the specimen failed at the bottom. In 3PB, the AB-sample started to deform plastically at lower load (750.86 MPa) and underwent more displacement before failure at 1405.94 MPa. In contrast, the HAT-sample yielded at 993.38 MPa, failed at higher loading (2022.98 MPa), and showed lower displacement with respect to the AB-sample. Thus, the bending strength increased upon heat treatment. Figure 6-10 (b) similarly shows an identical trend in the 4P-bending test in which the hybrid steel performed stronger after the HAT sequence. Apparently, the HAT sequence was effective in enhancing the MS1-P20

hybrid steel under bending conditions. The lower strength values were obtained in 4P-bending than 3P-bending, which can be attributed to more complex loading conditions of the 4P-bending test.

Since deformation is associated with the movement and interaction of dislocations, TEM images were taken to study the deformation mechanism at very high magnification. Figure 6-11 shows The TEM micrographs of the HAT-sample underwent the four-point bending test. The TEM sample was extracted from the bottom of the bulk specimen and far from the neutral axis. Figure 6-11 (a) shows the martensitic lath structure of the HAT-sample as well as the accumulation of dislocations. The dislocations tend to pile up and accumulate through the martensite laths. The individual dislocations can be detected at higher magnification, as shown in Figure 6-11 (b). The individual dislocations tend to interact with each other and interlock as deformation proceeds. Moreover, the surrounding areas of nano precipitates are depleted in dislocations, which means the strain field of precipitates inhibits the movement of dislocations.

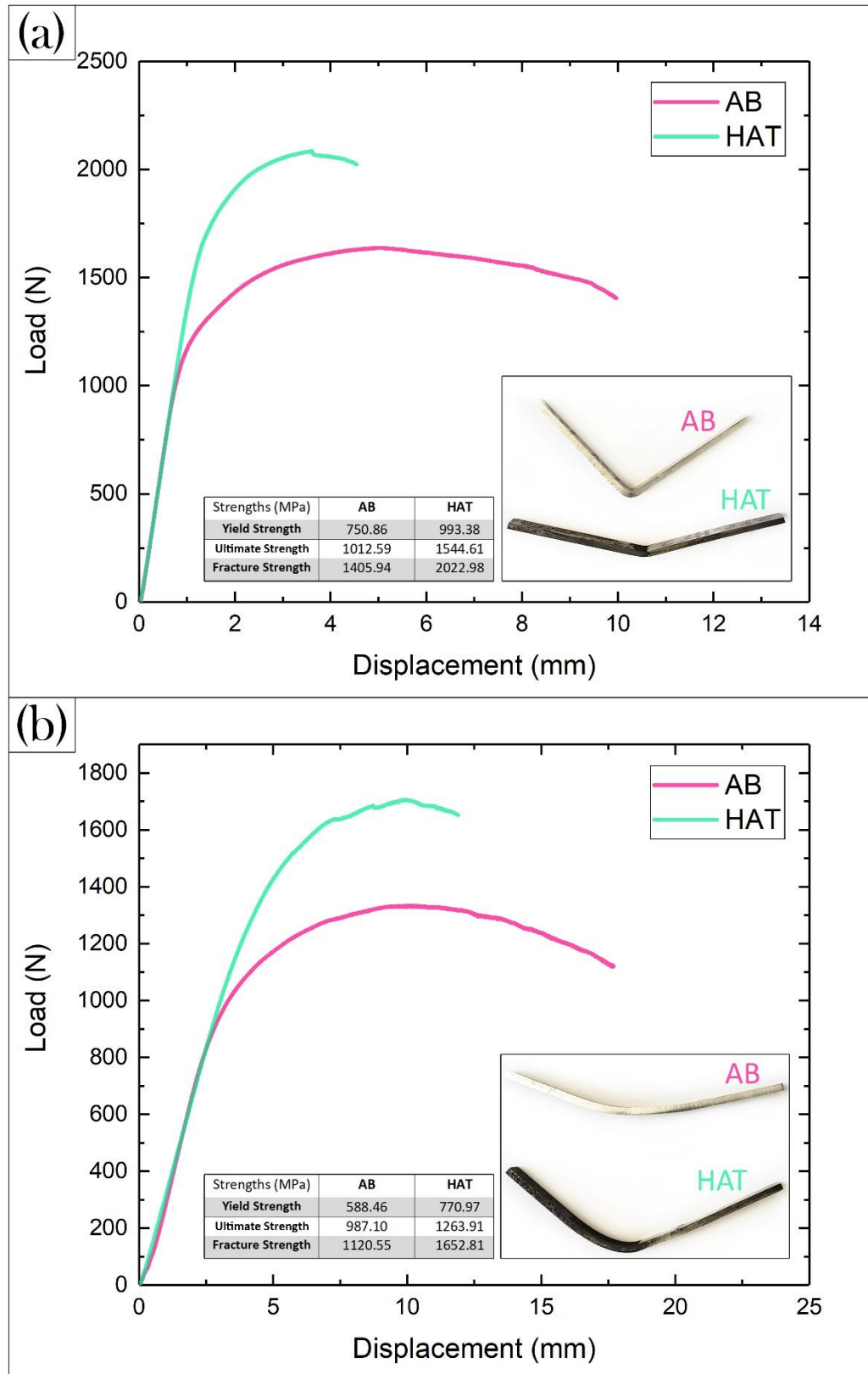


Figure 6-10: Load-displacement profile for a) 3P bending b) 4P bending tests

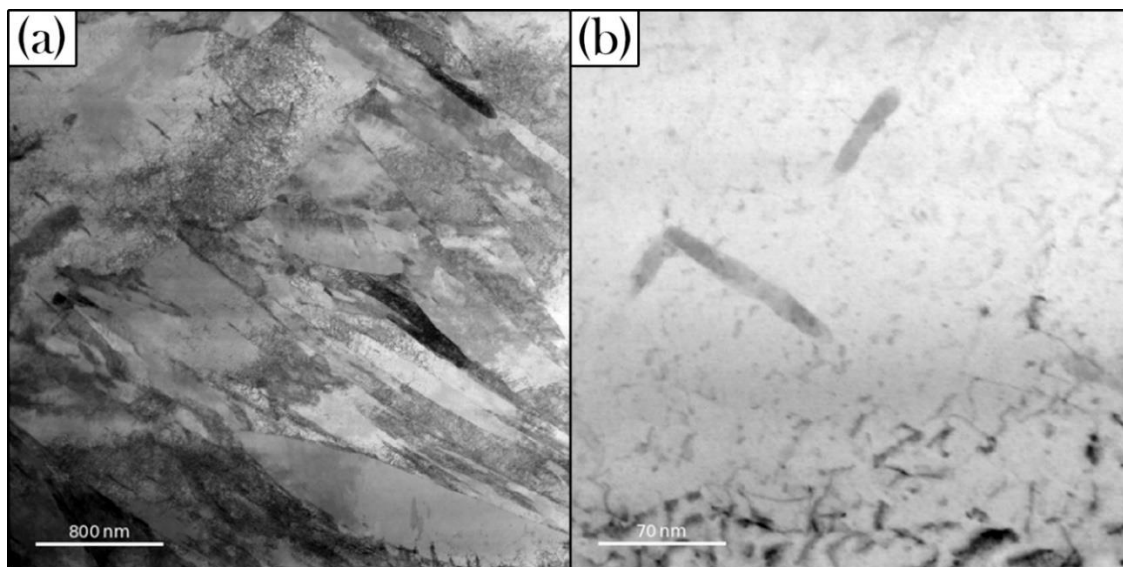


Figure 6-11: STEM bright-field images showing the general microstructure and the dislocation arrangement in HAT-sample after 4P bending test

6.4 Discussion

The heat treatment procedure was proved to significantly impact the MS1-P20 hybrid steel. This section discusses the microstructural modifications which led to the enhancement of mechanical properties.

The MS1-P20 hybrid steel was presented as two-phase steel; martensitic MS1 on ferritic substrate P20 in the as-built condition. Since the martensite phase is harder than ferrite, hardness varies substantially from low values of P20 to high values of MS1 across the interface. However, the hardening treatment at 880 °C (HT treatment) reversed the hardness profile, where a very hard substrate was obtained, followed by a drastic decrease in MS1 hardness. The HT temperature was extremely high to cause softening in the MS1. Despite the hardness increase in P20, the HT hardness profile is no longer favorable for mechanical performance since the MS1 side is abnormally softer than the substrate, which

can cause early failure upon loading. Therefore, a complementary treatment should be carried out to level off the hardness profile across the interface. For this purpose, the hardening treatment was followed by the aging treatment at 500 °C for 2 hours. This heat treatment sequence corrected the hardness variation and resulted in a relatively straightened hardness profile across the interface. Thus, more uniform mechanical behavior can be expected in the HAT condition.

It was stated that the HAT treatment caused grain growth in MS1, while grain refinement occurred in P20. The following assessment was presented to better understand how the heat treatment caused the aforementioned morphological evolution in MS1 and P20. First, the PAGs were reconstructed for each step of the HAT sequence (i.e., hardening and aging), and then they were compared with their original EBSD maps. The PAG maps can be regarded as representative microstructures at high temperatures. Figure 6-12 indicates step-by-step morphological changes for both MS1 and P20 and clearly shows the evolution of grains in terms of grain size and shape during the HAT sequence.

In MS1, the hardening treatment caused an abnormal grain growth by a factor of 15, where the as-built grain area of $98.49 \mu\text{m}^2$ (step I) raised to $1527.20 \mu\text{m}^2$ at 880 °C (step II) as shown in Figure 6-12 (a). Since this temperature is within the range of solution temperature of maraging steels [57], [58], the as-built grain boundaries, as well as solidification cells, dissolved, and more equiaxed austenite grains with coarser size appeared. The subsequent quenching turned the coarse austenitic morphology into a finer martensitic structure (step III). In the next step, the resultant PAG grain size was measured to be $567.17 \mu\text{m}^2$ (step IV), which is much finer than the grain size at 880 °C. This can be related to the finer martensite structure of step III and lower temperature of aging, which

can lead to finer PAGs upon heating. Finally, coarser martensite grains ($106.03 \mu\text{m}^2$) were developed during cooling to room temperature (step V). The larger grain area of step V compared to step III can be attributed to a slower cooling rate of aging, resulting in a coarser martensite structure in MS1. Although the precipitation during aging can inhibit grain growth, the holding time of 2 hours might not be sufficient to trigger this effect. In terms of grain geometry, the hardening treatment was successful in creating more equiaxed grains (steps I-II); however, the martensite transformation during cooling resulted in more columnar grains (steps II-III and IV-V). This is mainly due to the martensite structure, in which columnar martensite packets were developed as the dominant microstructure.

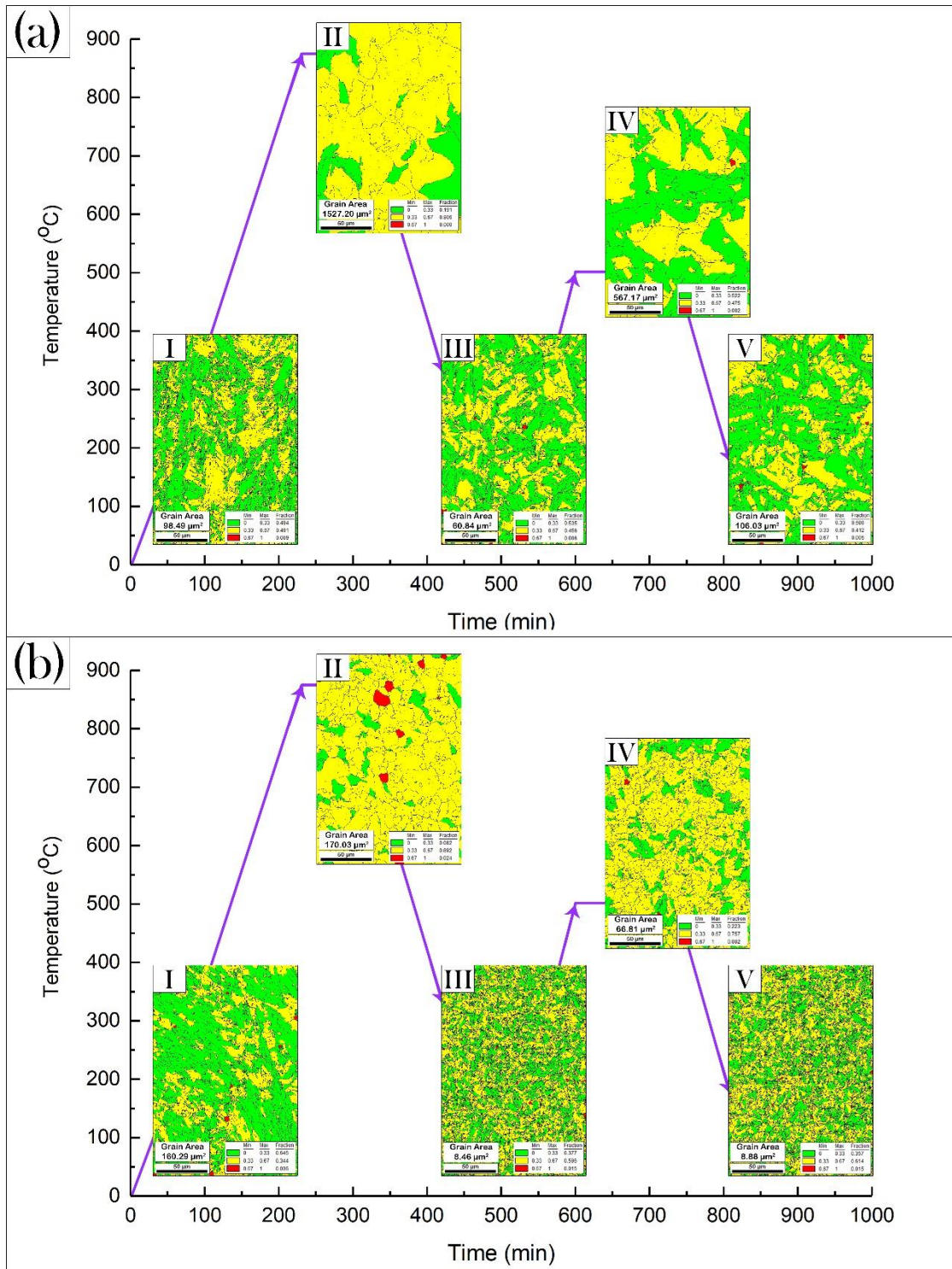


Figure 6-12: Morphological variation of a) MS1 b) P20 during HAT sequence. It should be noted that II and IV maps have been reconstructed based on the EBSD maps of III and V, respectively

Figure 6-12 (b) shows the corresponding step-by-step morphological map for P20. The first heating cycle from room temperature to 880 °C did not alter the grain sizes, and only a negligible grain growth occurred, where grain area slightly raised from 160.29 μm^2 to 170.03 μm^2 . On the other hand, the columnar grains of as-received P20 were evolved to more equiaxed grains at this temperature since 92% of PAGs involved an aspect ratio of ≥ 0.33 (step II). The hardening treatment, indeed, rearranged the P20 grains along and dissolved the carbide precipitates. Upon quenching, a very fine martensite structure was formed (Step III). Comparing steps I and III, a remarkable grain refinement can be seen as a result of the martensite transformation which led to very high hardness values of P20 in the HT condition. In the next heating cycle to 500 °C (step IV), finer PAGs were also constructed compared to 880 °C. Likewise, finer martensitic grains of step III and lower temperature of aging can cause smaller PAGs in step IV. In the final step V, the P20 grains evolved as small as the grains in step III, so the main contributor to the grain refinement is the hardening treatment at 880 °C. Regarding the grain geometry, it seems that both heating cycles (steps I-II and III-IV) reformed the grains in equiaxed shapes, while the subsequent martensite transformation upon cooling resulted in more columnar grains. The grain refinement of P20 diminished the grain size difference between P20 and TZ and promoted an indistinguishable interface in the HAT condition. Moreover, lower hardness was achieved in the HAT condition. The aging treatment at 500 °C acted as a tempering treatment on P20 and encouraged stress relief in step IV. Subsequently, lower hardness values were measured in step V, which was favorable since a more uniform hardness profile was formed across the HAT-interface.

The PBF process involves intensive and localized heating followed by rapid solidification, so the grains can absorb residual stresses and contain high strains. In addition, the HAT sequence might affect the grains in this regard. Consequently, strain variation should be significant across the interface and plays an important role in the mechanical performance of the hybrid MS1-P20 steel. Therefore, quantitative strain analysis was carried out using GOS and KAM values, and the changes were evaluated before and after the HAT sequence. Figure 6-13 compares the distribution of GOS misorientations for each area of the interface before and after the heat treatment. To distinguish the high-strain grains, a threshold of $GOS > 2^\circ$ was selected and used as the criteria to calculate the percentage of high-strain grains [16], [59], [60]. The as-received P20 contains the lowest GOS values, and only 29% of grains are high in strain. However, the HAT sequence raised this value to 39% for P20. Both TZ and MS1 areas are associated with higher values of GOS and contain 53% and 63% high-strain grains, respectively. The HAT sequence led to higher high-strain grains in TZ (62%), while the opposite trend occurred in MS1 (58%). Quantitative measurement of KAM-based misorientation is also included in Figure 6-13, which simply shows the misorientation evolution of each zone across the interface. The average misorientation of P20 slightly increased during the HAT sequence with a minute increase from 0.64° to 0.72° . The same trend also occurred in TZ, where the average misorientation of 0.89° raised to 0.94° . KAM misorientation, however, reduced from 0.96° to 0.77° upon heat treatment in the MS1 area. This reduction can clearly be seen in Figure 6-7 (b). Both GOS and KAM analyses revealed that the fluctuation of strain values tends to be subtle across the interface after heat treatment (HAT). This effect was also observed in Figure 6-7 (d).

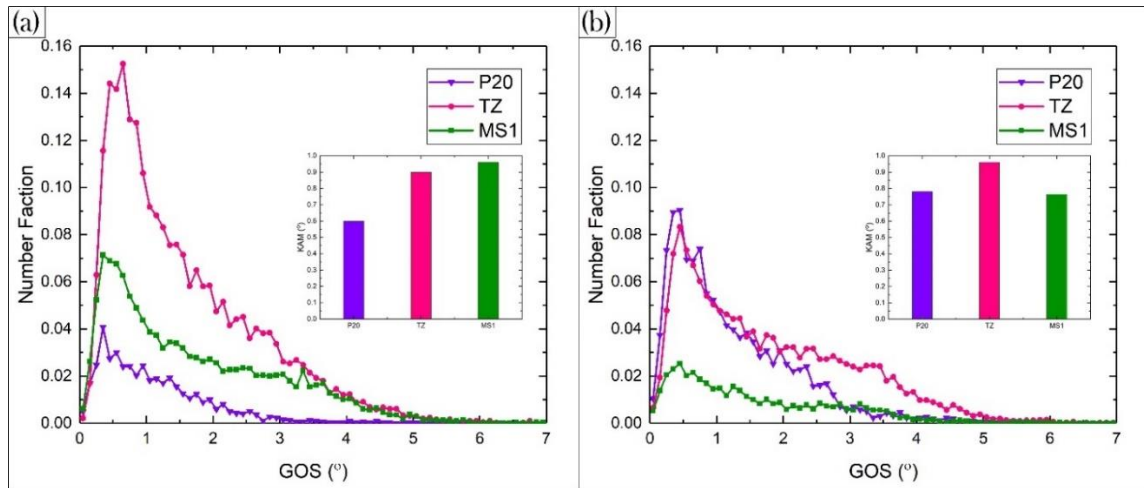


Figure 6-13: GOS & KAM strain analysis of a) AB and b) HAT

The HAT sequence effectively modified the microstructure of MS1-P20 hybrid steel in terms of hardness, grain morphology, and strain values. As a result, the bending properties were also affected by this modification, as indicated by Figure 6-10. The HAT sequence enhanced the ultimate strengths in three-point and four-point bending conditions by a factor of 1.53 and 1.28, respectively. The enhancement of bending strength can be highly entangled with the deformation mechanism of the hybrid steel during the bending tests.

The three-point bending samples were studied using the EBSD technique to correlate the bending deformation to the microstructure. Strain analysis was investigated across the interface and shown in Figures 6-14 and 6-15 for AB- and HAT- samples, respectively. Figure 6-14 (a) illustrates the GOS mapping of the as-built specimen after the three-point bending test. This figure suggests that the P20 edges are highlighted in warm color, which means that the majority of strains are stored at the P20 edges, where the highest compression and tension stresses were applied at the top and bottom of the bending sample, respectively. The KAM mapping is also shown in Figure 6-14 (b), which can be

considered as the distribution of dislocations throughout the sample. On the P20 side, the edges look saturated with KAM values indicating a high density of dislocations because of the stress conditions. In the middle of the bending sample, the variation of KAM seems identical to that of Figure 6-7 (a) and (c), showing lower strain values in P20 while higher values on the MS1 side. This is mainly attributed to stress conditions in which the least stress concentrated in the middle of the sample in the 3-point bending test.

The GOS mapping as well as KAM distribution were also extracted for the HAT-sample and presented in Figures 6-15 (a) and (b). In this condition, both maps evolved uniformly, and lower color contrast can be observed across the interface. The P20 edges are still areas of higher strains; however, the KAM contrast between the edges and the middle area seems less intense than the corresponding areas in Figure 6-14. Therefore, the dislocations were distributed homogeneously in the HAT-sample.

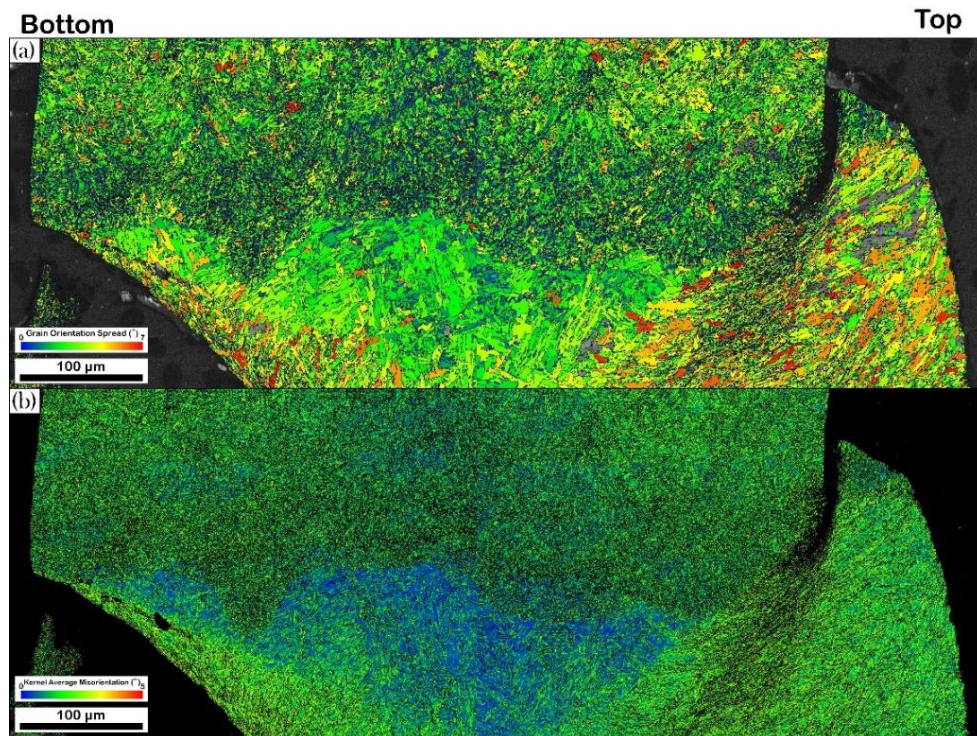


Figure 6-14: Strain analysis of AB-interface after 3P bending: a) GOS b) KAM

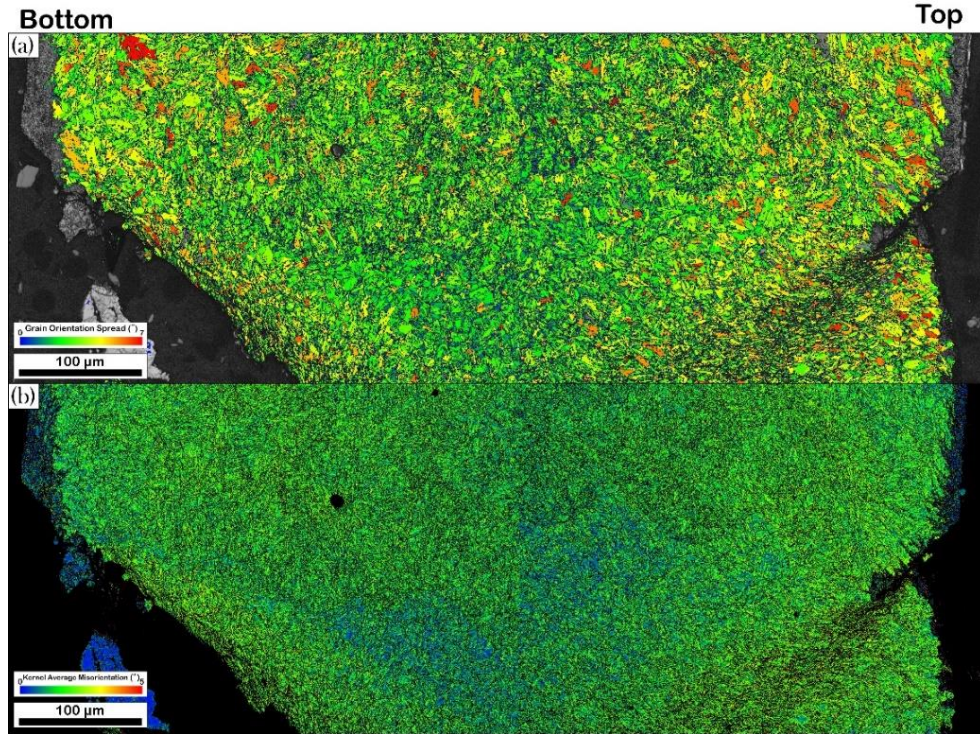


Figure 6-15: Strain analysis of HAT-interface after 3P bending: a) GOS b) KAM

The deformation mechanism can also be evaluated using the Schmid factor mapping. The $\{110\}\langle 111 \rangle$ and $\{112\}\langle 111 \rangle$ as the main slip systems of BCC and BCT crystallographic structures [61]–[63] were selected and used to extract the Schmid factors. Figures 6-16 (a) and (b) suggest that both slip systems were active and contributed to the deformation of the AB-sample. The average value of Schmid factor for $\{110\}\langle 111 \rangle$ and $\{112\}\langle 111 \rangle$ slip systems are 0.48 and 0.45, respectively. Although the Schmid factor was measured to be relatively identical for both slip systems, the $\{110\}\langle 111 \rangle$ slip system seems to be more active than the $\{112\}\langle 111 \rangle$ slip system in AB-sample. These values; however, were calculated to be 0.46 and 0.45 for the HAT-sample (See Figures 6-17 (a) and (b)). There is also a contrast between P20 and MS1 in the as-built condition, implying the fact that the deformation activity is different between these two materials. The P20 side seems brighter than the MS1 side, which means the slip systems are relatively less active in P20

than MS1. In HAT-sample, both slip systems exhibit indistinguishable mapping across the interface, and no contrast can be observed between P20 and MS1 sides. Therefore, uniform deformation occurred in the HAT condition across the MS1-P20 hybrid steel.

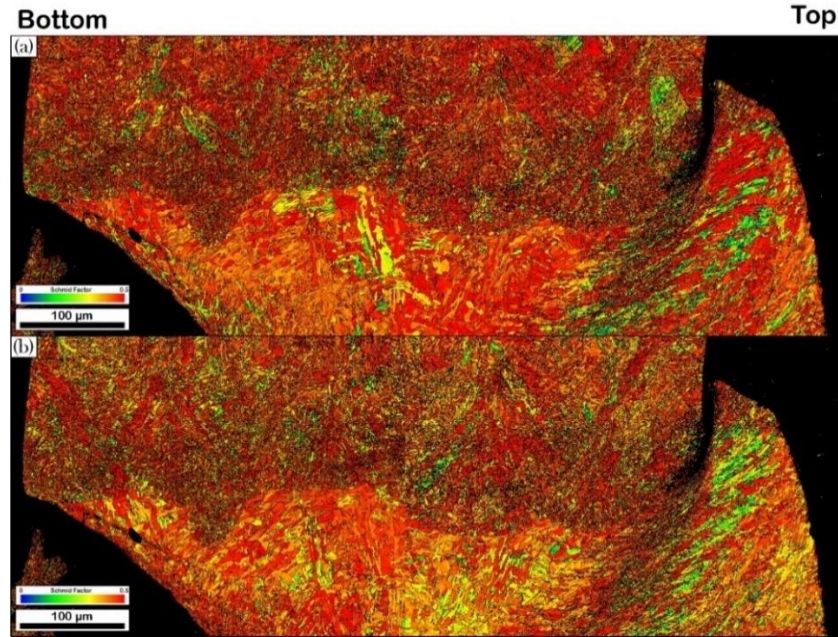


Figure 6-16: Deformation maps of AB-interface after 3P bending: a) $\{110\}\langle 111 \rangle$ b) $\{112\}\langle 111 \rangle$ slip systems

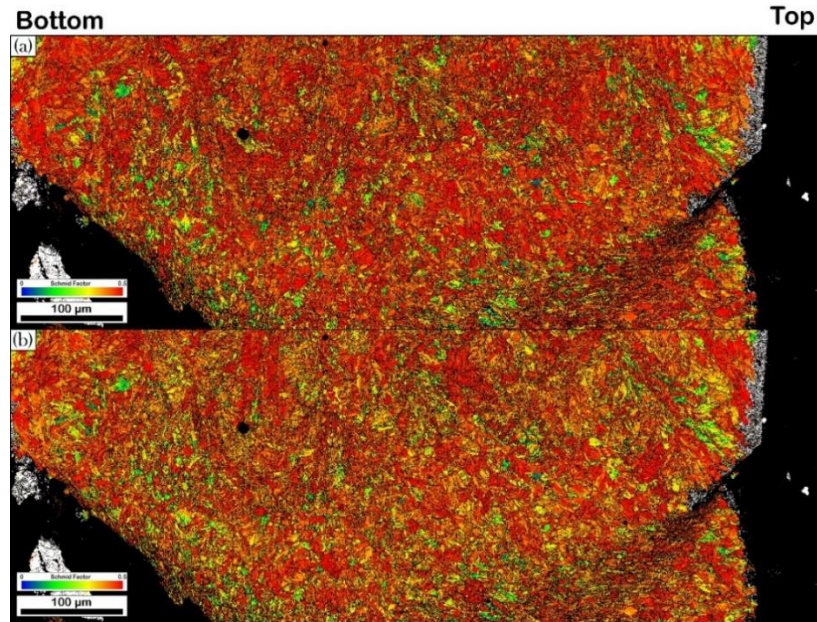


Figure 6-17: Deformation maps of HAT-interface after 3P bending: a) $\{110\}\langle 111 \rangle$ b) $\{112\}\langle 111 \rangle$ slip systems.

It was shown that the as-received P20 was weaker than the additive side in the AB-sample (see Figure 6-2). Plastic deformation initiated from P20 at lower loading values. In the 3P bending test, the top and bottom of the MS1-P20 sample are the areas with the highest stresses, as indicated. So plastic deformation mostly occurred at the P20 edges. The dislocations were generated and moved in the slip systems; however, the P20 grains could not tolerate further movement of dislocations at some point, and the dislocations began to accumulate. Excessive accumulation of dislocations leads to the formation of voids and propagation of cracks at the edges, and finally, failure of the AB-sample. The dislocation accumulation is evident in Figure 6-14 (b) at the bottom and top edges of P20. This figure also proved that dislocations could not move and propagate to the middle of P20, and dislocations were blocked at the edges. Moreover, the P20 edges contain some green color grains in Figure 6-16. This means that the slip systems had the lowest activity in these grains, which is well consistent with the impeded dislocation at the edges (see Figure 6-14 (b)). Therefore, the existence of the least active grains at high strain areas of the sample suggests that the as-received P20 grains possess very low deformability, and the AB-sample consequently failed at a lower strength.

The KAM map evolved as a homogenous profile throughout the HAT-sample upon the HAT sequence. This is particularly important from the bottom to top areas of the sample, where the highest stresses were applied. Indeed, dislocations distributed more uniformly and hence had more mobility to move and propagate throughout the sample (see Figure 6-15 (b)). The uniform distribution of dislocations definitely indicates that the plastic deformation took place homogeneously in the HAT condition. This is well demonstrated in Figure 6-17, where there is no contrast either across the interface or from

the bottom to top areas. Upon the HAT sequence, a comparable martensitic structure was developed between P20 and MS1, and the resultant interfacial microstructure generated identical hardness and strength on both sides of the interface. Therefore, the MS1-P20 hybrid steel featured monotonous deformation properties and exhibited higher strength under complex loading conditions of bending tests. Consequently, the MS1-P20 interface was favorably engineered using the HAT sequence.

6.5 Conclusion

The current research investigated the heat treatment and mechanical behavior of additively manufactured MS1-P20 hybrid steels under complex loading conditions. An effective heat treatment sequence was proposed, and the following outcomes were produced:

- The hardening treatment at 880 °C/1 hour followed by the aging treatment at 500 °C/2 hours was designated as the HAT sequence. This heat treatment sequence generated the martensite phase on the P20 side and reformed the martensitic matrix of MS1.
- The HAT sequence caused two opposing grain evolutions: grain growth in MS1 and grain refinement in P20. This effect rearranged the grain morphology across the interface.
- Both AB- and HAT- samples consisted of three main zones, including P20, TZ, and MS1. And the interface was defined as the boundary between P20 and TZ. The AB-

interface was clearly distinctive in EBSD maps; however, the HAT sequence removed this distinction and made a less distinguishable interface.

- The HAT-sample featured almost constant hardness properties across the interface, while wide fluctuation of hardness was detected in the AB-sample.
- The HAT sequence successfully homogenized the strain energy across the interface and promoted uniform deformation in bending conditions.
- The HAT-interface did not fail under 3P- and 4P- bending tests, and cracking initiated and grew with P20.
- The HAT-sample yielded and failed at higher bending strengths than the AB-sample. Thus this sequential heat treatment can be a practical approach to modifying MS1-P20 hybrid steel.

6.6 References

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7 Summary

The goal of this thesis was to use additive manufacturing in production of reliable bimetals for practical applications, particularly in tool and die industry. Two different tool steels were selected including H13 and P20 as the representatives of hot work and cold work tool steels, respectively. Maraging steel (MS1) was successfully deposited on both tool steels which means a good bond was made between MS1 and the tool steels (i.e. H13, P20). Both MS1-H13 and MS1-P20 showed good cohesion at the interface area. This result was considered the prerequisite for a reliable AM bimetal.

Although sound bimetals were fabricated, they exhibited weak behavior under loading conditions, and relatively poor mechanical properties were recorded. The in-depth characterization also proved the microstructural inhomogeneity of interface area as the main contributor to low strength values. Since the root of weak mechanical performance was detected, it was concluded that modification of microstructure can recover good mechanical properties in these AM bimetals.

Heat treatment- based modification procedures were designed regarding the specific heat treatment cycles of base materials in each bimetal systems (i.e. MS1-H13 and MS1-P20). In this step, heat treatments were applied on the as-built bimetals in order to promote identical microstructural features between both base materials. Either MS1-H13 and MS1-P20 systems evolved as more homogenized bimetals upon the appropriate heat treatments. In other words, the interface was less detectable because of microstructural similarities of base materials.

Finally, the modified bimetals obviously behaved stronger than the as-built samples, and exhibited superior mechanical performance even under more complex loading conditions. Thus, the modified bimetals can be considered successful candidates for practical applications.

8 Conclusion

In brief, this thesis investigated additive manufacturing of tool steel bimetals using maraging steel. It consists of two main alloying systems and four different targets. The two main alloying systems were MS1-H13 and MS1-P20. For each alloying system, four targets were set, including i) fabrication of a sound bimetal, ii) characterization of the interface, iii) modification of interfacial microstructure and iv) evaluation of modified bimetal. The first target was achieved via depositing maraging steel (MS1) on top of H13 and P20 substrates. A laser powder bed fusion (LPBF) printer was used for this purpose. The second target was reached using comprehensive microscopic techniques as well as mechanical testings. Optical and scanning electron (SEM) microscopes were used to observe the initial microstructure of fabricated bimetals. In-depth characterizations were followed using the electron backscatter diffraction (EBSD) technique, transmission electron microscope (TEM), and energy dispersive spectroscopy (EDS). Modification of bimetals was carried out using heat treatment, and the modified bimetals were assessed via the advanced characterization techniques mentioned earlier.

The first paper entitled “*Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique*” was published in the journal of “*Additive Manufacturing*”. This paper discussed the fabrication of MS1-H13 bimetal and the microstructural and mechanical properties of this tool steel bimetal. A successful bond was made between the additive side (MS1) and substrate (H13) with no porosity, cracks, or discontinuity at the interface. The interface between MS1 and H13 was a narrow area of 2 μm ; however, the interfacial area involved four regions as substrate-H13, partially melted zone (PMZ), transition zone (TZ), and MS1. The grain morphology of MS1-H13 bimetal

consisted of ultrafine grains of MS1 and coarse grains of H13. The additive side (MS1) exhibited much higher hardness values than the substrate-H13, and the as-fabricated MS1-H13 failed from H13 under uniaxial loading. This was mainly due to the softer matrix and coarser grains of H13 resulting in lower hardness and tensile strength of the substrate. Although the MS1-H13 bimetal failed at a lower strength, this result was a success since the interface did not fail.

The low performance of the as-built MS1-H13 motivated us to work on enhancing the mechanical properties through heat treatment. Accordingly, we evaluated phase transformations of both MS1 and H13 and conducted different heat treatments in the format of a single cycle or sequences. This work includes two papers: our second and third papers. The second paper was published in the journal of “*Materials Characterization*”, under the title of “*Interface engineering of additively manufactured maraging steel-H13 bimetallic structures*”. This paper investigated the effect of two heat treatment cycles on the as-built MS1-H13 bimetal. The main purpose of heat treatment was to generate a homogeneous microstructure between MS1 and H13. Striking results were achieved with the solution treatment, in which a uniform martensitic structure was developed entirely in the MS1-H13 bimetal. The grain morphology and dislocation density were also homogenized between MS1 and H13, leading to uniform hardness properties as well. The assessment of mechanical properties was accomplished via uniaxial tensile loading, and this result indicated a stronger MS1-H13 bimetal with strength values three times higher than the as-built condition. The third paper was submitted to the journal of Materials Engineering and Performance as “*Microstructure control of hybrid additively manufactured 18Ni300-H13 bimetal by sequential heat treatments*”. This work discussed the sequential heat treatment

procedure for the MS1-H13 bimetal, in which almost identical results with the second paper were obtained. No texture was developed across the MS1-H13 interface upon sequential heat treatment, and slightly higher ductility was achieved in this condition.

The significant role of P20 mold steel in the die industry motivated us to pursue our investigation on the MS1-P20 alloying system. The four targets were accomplished and presented in the fourth and fifth papers. “*Hybrid Manufacturing of MS1-P20 Tool Dies by Powder Bed Fusion*” was submitted to the journal of “Additive Manufacturing” as the fourth paper. This work explained the reliable fabrication of MS1-P20 bimetal, the characteristic features of the MS1-P20 interface, and the mechanism of interface formation between MS1 and P20. Interestingly, a real industrial part was fabricated out of MS1-P20 bimetallic structure due to the successful results of this work, where a very cohesive interface was formed between MS1 and P20. The mechanical properties were further analyzed using the digital image correlation (DIC) method, in which the strain field was measured on the tensile sample during loading. The result showed that the interface was strong enough to tolerate the loading stresses due to Hall-Petch effect and forest dislocation hardening at the interfacial area.

Despite the strong joint of as-built MS1-P20, we aimed at obtaining higher strength and evaluating the performance of this tool steel bimetal, which formed our motivation for the last paper of this thesis. The fifth paper entitled “*On the bending of MS1-P20 hybrid steels additively manufactured using laser powder bed fusion*” was submitted to the journal on “Materialia”. It was found that a heat treatment sequence can be effective in modifying the MS1-P20 bimetal. Microstructural homogeneity and uniform hardness properties were achieved because of heat treatment. The performance evaluation was conducted using two

different bending tests. The MS1-P20 bimetal showed uniform deformation and higher strength along with lower localized strain in the heat-treated condition. This paper also emphasized the EBSD technique as a sophisticated tool to analyze the characterization, modification, and deformation of tool steel bimetals.

9 Future Works

The current study proved that the powder bed fusion (PBF) technique has a great potential for producing dissimilar components called bimetallic structures. Although defect-free joints were generated in both MS1-H13 and MS1-P20 bimetallic systems, the effect of process parameters or different strategies were not evaluated. It might be possible to optimize the PBF process parameters to produce more efficient bimetals in terms of microstructure and properties. The bimetal samples made in this study had simple geometries; however, complex geometries can be more challenging. This is particularly important in the fabrication of industrial parts from a bimetallic structure. In future works, such real bimetallic parts can be investigated in collaboration with industries.

This study presented standard mechanical testing methods, including hardness, uniaxial tensile loading, and bending loading. These tests provided us with important information about the standard mechanical properties, including hardness, yield strength, UTS, ductility, toughness, and fracture strength. This information can be used to develop numerical or analytical models which enable us to predict the behavior of the bimetallic structure in working conditions. Moreover, industrial parts are normally designed to work in more severe service conditions, where complex stress conditions such as fatigue, creep, high temperature or corrosive environments coexist. In this case, more investigations should be conducted to determine the mechanical, thermal, and corrosion performance of bimetallic components. These tests can be designed based on the application of fabricated bimetals. For instance, the compression test at high temperatures can be conducted on tool steel bimetals since they can be utilized as forging dies.

A significant part of this study was dedicated to the modification of tool steel bimetals through heat treatment, and very interesting results were obtained, including the noticeable increase of strength after heat treatment. This study showed that the tool steel bimetals could be manipulated using heat treatment. However, this is relatively a new field of study since phase transformations of additively manufactured materials and bimetallic systems have not yet been well understood. Therefore, more in-depth research should be carried out to broaden our knowledge in the post-processing of additively manufactured bimetals. One of the greatest methods to study the effect of heat treatment on any material is the in-situ characterization techniques, in which the microstructural evolution is being monitored in every stage of heat treatment. This will definitely help us to better discover the phase transformation mechanism and better select the proper heat treatment cycle.

Our approach to exploring the tool steel bimetal was based on experimental studies, which is an essential part of research in the field of mechanical engineering. This approach can be better completed by simulating the interface formation in the additive manufacturing bimetals. This requires thorough knowledge of the solidification and solid-state transformation during the AM techniques. The simulation methods can enable researchers to anticipate the interfacial microstructure of AM bimetals and predict the corresponding properties. These predictions can then be validated with advanced EBSD techniques such as the three-dimensional (3D) EBSD acquisition. In addition, the outcome of the 3D-EBSD technique can be used as the input to predict the performance of AM bimetals in working conditions. As a result, simultaneous experimental and theoretical investigations can offer the most comprehensive perspective on the design and fabrication of practical bimetallic components.

Appendix A



Additive Manufacturing

Volume 29, October 2019, 100797



Full length article

Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique

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Materials Characterization

Volume 170, December 2020, 110728



Interface engineering of additively manufactured maraging steel-H13 bimetallic structures

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- **S. Shakerin**, A. Hadadzadeh, BS. Amirkhiz, S. Shamsdini, J. Li, M. Mohammadi, *Additive manufacturing of maraging steel-H13 bimetals using laser powder bed fusion technique*, Additive Manufacturing 29 (2019) 100797.
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Submitted under review or under preparation publications:

- **S. Shakerin**, H. Pirgazi, M. Sanjari, D. Chalasani, M. Mohammadi, Microstructure control of hybrid additively manufactured 18Ni300-H13 bimetals by sequential heat treatments, submitted in Journal of Materials Engineering and Performance.
- **S. Shakerin**, M. Sanjari, H. Pirgazi, BS. Amirkhiz, A. Lloyd, M. Mohammadi, Hybrid Manufacturing of MS1-P20 Tool Dies by Powder Bed Fusion, submitted in Additive Manufacturing.
- **S. Shakerin**, M. Sanjari, H. Pirgazi, BS. Amirkhiz, A. Lloyd, M. Mohammadi, On the Bending of MS1-P20 Hybrid Steels Additively Manufactured Using Laser Powder Bed Fusion, submitted in Materialia.