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ORIGINAL REPORT



Laser deposition additive manufacturing of 17-4PH stainless steel on Ti-6Al-4V using V interlayer

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ABSTRACT

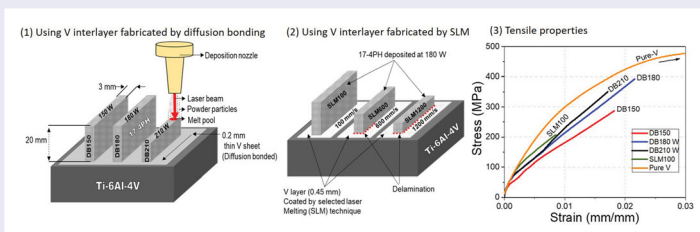
For the first time, additive manufacturing was used to obtain a strong joint (bonding strength > 200 MPa) between stainless steel and Ti-6Al-4V using a V interlayer. The V interlayer applied via solid-state joining prevented the formation of brittle intermetallic phases more effectively than a V interlayer deposited by a liquid-state process. The microstructure and strength of the joint interface depended on the laser power and scan speed. The joint strength decreased with increasing annealing time, especially after 4 and 24 h, demonstrating the presence of the Fe-V-Cr σ -phase.

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Additive manufacturing; bonding strength; stainless steel; Ti-6Al-4V; σ -phase



IMPACT STATEMENT

For the first time, an additive manufacturing process using a V interlayer successfully produced a hybrid structure of stainless steel and Ti-6Al-4V alloy.

Introduction

Additive manufacturing (AM) has attracted significant worldwide attention in recent years because it produces complex-shaped, functionally graded, and customized parts [1,2]. For instance, the solidification and cooling path from laser AM can provide a uniform fine lamellar $\alpha + \beta$ microstructure in Ti-6Al-4V [3]. In addition, by exploiting the intrinsic heat treatment capabilities of AM, the microstructural properties of alloys such as Ti-6Al-4V and maraging steels can be improved [4,5]. In most popular metal AM processes, including selective-laser melting (SLM) and direct-energy deposition (DED), layers of powder material are fused sequentially to previously fused layers on a base plate. Generally, similar powders or metals are used for both building and the base plate, because dissimilar material pairs can develop

considerable residual stresses and intermetallic compounds. Hence, AM using two different materials is very difficult to achieve. In particular, AM between Ti alloys and steels is extremely difficult because the mixing of molten Ti and Fe during processing readily yields undesirable Fe-Ti intermetallic compounds [6], which facilitate delamination fracture. Even with these difficulties, highly dissimilar joining between Ti alloys and high-strength steel remains of interest because their combined properties would provide many technological advantages in the chemical, nuclear, and spacecraft industries [7]. For instance, a high-speed projectile should be very light and simultaneously resistant to frictional heat with air, which could be achieved by a hybrid structure of Ti and steel. However, because direct Ti deposition on steel inevitably forms intermetallics, the introduction of an

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interlayer material that is metallurgically compatible with both Ti and steel is required. Materials such as Cu [8], Ni, and alloys thereof [9] fail as interlayers because they form unfavorable intermetallic phases. V, however, is a promising interlayer metal [10], forming a continuous solid solution in all temperature ranges with Ti [11]. With Fe, V shows large regions of continuous solid solutions at temperatures $> 1219^{\circ}\text{C}$ and a controllable metastable σ -phase within the temperature range $400\text{--}1219^{\circ}\text{C}$ and composition range 19–78 wt.% [12].

Limited studies describe the use of AM techniques to effectively join Ti and stainless steel. Li et al. [13] employed a transition composition route ($\text{Ti-6Al-4V} \rightarrow \text{V} \rightarrow \text{Cr} \rightarrow \text{Fe} \rightarrow \text{SS316}$) to join Ti-6Al-4V to SS316 stainless steel. However, this multiple-interlayer process is limited in practicality and its bonding strength was not reported. Reichardt et al. [14] used a multi-hopper laser deposition system to manufacture functionally graded 304L stainless steel to Ti-6Al-4V with the subsequent addition of V, mixed with stainless steel as an interlayer. Mid-fabrication cracking occurred, attributed to the precipitation of Fe-Ti and Fe-V-Cr (σ -phase). The mixed V interlayer facilitated the precipitation of σ -phase and caused the direct interaction of steel and Ti.

The current work therefore focuses on the complete segregation of steel and Ti-6Al-4V with pure V as the interlayer. The interlayer will thus enable the practical usage of the component and prevent possible intermetallic compound formation. The mechanical properties and microstructures near deposition interfaces are analyzed. Solid-state cladding (diffusion bonding) is compared to liquid-state deposition (SLM) for V interlayer production. The effects of the laser power, scan speed, and post-weld heat treatment (PWHT) of the bonded metals are examined.

Experimental

The materials used in this experiment were a Ti-6Al-4V plate, 17-4PH stainless steel powder, and pure V (99.9 at.%) in the form of a sheet (0.2 mm thickness) and powder. The V interlayer was bonded to Ti-6Al-4V in two separate ways for comparison: (1) through diffusion bonding (DB) and (2) by SLM.

In DB, the surfaces of Ti-6Al-4V and V were ground with 2400-grit SiC papers and cleaned ultrasonically with acetone. They were then clamped together with abutting surfaces. A hydraulic press was applied to the components for 4 h at 1100°C . A successful bond was obtained between the Ti-6Al-4V and V. Thereafter, a DED machine (DMT 3D printer) from InssTek Co., Ltd.

was used to deposit the 17-4PH powder on the V surface of the Ti-6Al-4V/V bar. During DED, three different laser powers were utilized for three different samples. The sample designations DB150, DB180, and DB210 were assigned to samples processed with laser powers of 150, 180, and 210 W, respectively.

For the SLM process of interlayer application, V powder was deposited directly on the surface of the Ti-6Al-4V plate under an Ar atmosphere using an SLM machine (Concept LaserTM, MLab). The laser power of 90 W and scan spacing of $80\text{ }\mu\text{m}$ were used to build all samples to a layer thickness of $\sim 0.40\text{ mm}$. Different scanning speeds of 100, 600, and 1200 mm/s were applied to specimens denoted SLM100, SLM600, and SLM1200, respectively. Similar to the DB process, the 17-4PH stainless steel powder was later deposited on the V surface of the Ti-6Al-4V/V bar via DED at the laser power of 180 W. A schematic and images of the bonded joint are shown in Figure 1. The experiment is further described in the supplementary material.

Results and discussion

Microstructure of V interlayer fabricated by DB

Figure 2 shows the microscopic images and X-ray diffraction (XRD) patterns of the V/17-4PH joint interfaces of samples DB150, DB180, and DB210. The scanning electron microscopy (SEM) image, energy-dispersive X-ray spectroscopy (EDS) line scan, electron backscatter diffraction (EBSD) phase map, and XRD pattern of the V/17-4PH fusion zone (FZ) of DB150 are shown in Figure 2(a) and (b). The microstructure is characterized as a partially mixed FZ with a small interface area (marked yellow) and a connected polygonal structure comprising a Fe + FeV solid solution [(FeV)ss]. The EBSD results show that the primary phase in the FZ is bcc with negligible precipitated amounts of retained austenite from the Fe-rich zone. The V concentration decreases from the fusion interface (region 1) to the center of the FZ (region 2), which mainly comprises (Fe)ss (Table S2). The low laser power provides insufficient energy and less solidification time, which causes less melting and mixing of the V substrate with the deposited 17-4PH, yielding smaller bonding area. The micro-XRD pattern in Figure 2(b) identifies the V/17-4PH FZ interface as mainly (FeV)ss. The points of XRD measurement for all samples were at the FZ near the V interlayer.

For DB180 with the V/17-4PH joint interface depicted in Figure 2(c) and (d), the joint shows a serrated structure at the FZ interface, as seen in region 3 (marked yellow), which grows toward the center of the FZ or in the buildup direction. This structure appears uniform with a wider

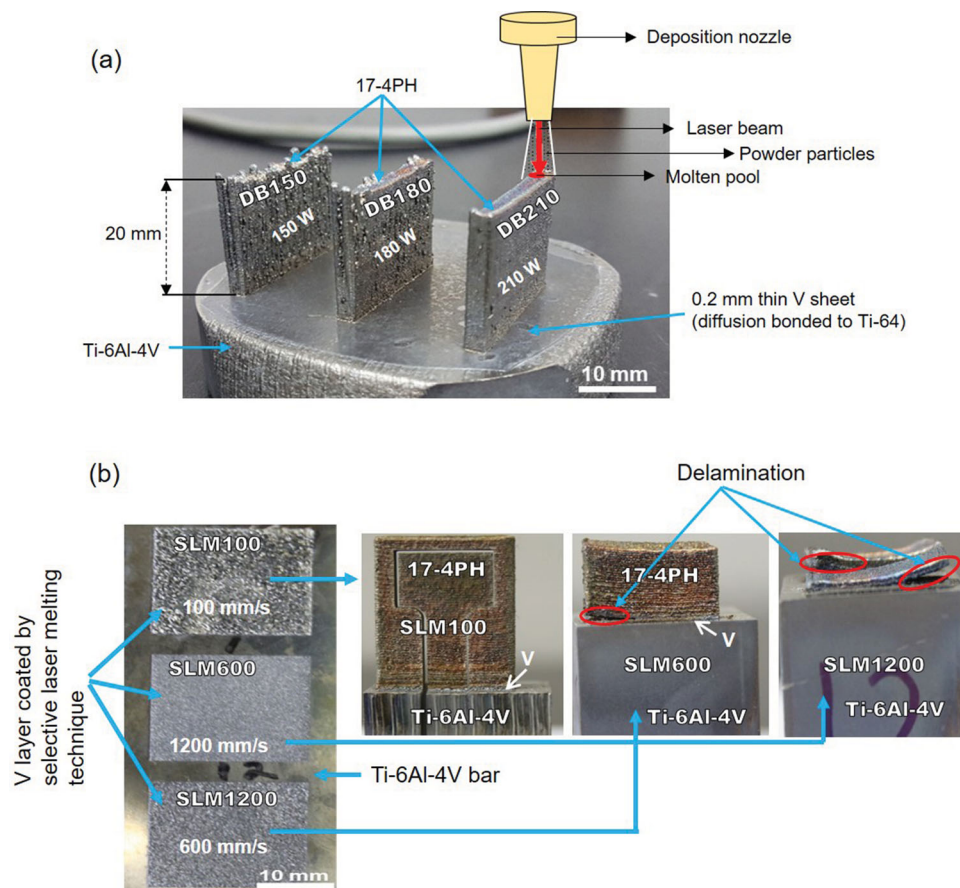


Figure 1. Optical images of (a) DB150, DB180, and DB210 after DED of 17-4PH. (b) SLM100, SLM600, and SLM1200 after DED of 17-4PH.

area, comprising Fe + (FeV)ss and a much higher V content than that of DB150. The observed composition is given in Table S2. Similar to DB150, the EBSD phase map shows the primary phase in the FZ to be mainly bcc. The XRD observation at the FZ (Figure 2(d)) confirms the presence of (FeV)ss. Accordingly, the laser power applied (180 W) is sufficient to melt more of the V substrate into the molten pool, yielding a higher V concentration in the FZ. In addition, the molten pool has a relatively longer time for solidification, yielding a fully-grown serrated structure as compared to that of DB150.

Figure 2(e) and f display the microstructure of the V and 17-4PH bonding interface of DB210 fabricated with the laser power of 210 W. It shows features similar to those of DB180. The FZ (Figure 2(f)) also appears as a serrated structure comprising (FeV)ss at the interface and columnar grains growing in the direction parallel to laser build-up. The growth direction of the serrated structure and columnar grains is attributed to the directional solidification of the molten pool because the substrate acts as a heat sink. However, the content of V in the FZ (FeV)ss is high, reaching 35.4 at.% in region 5, compared to ~ 28.0 at.% V measured in DB180. EBSD reveals the primary phase in the FZ as bcc with small V-rich

phases. The FZ is further analyzed by Transmission electron microscopy (TEM; see supplementary material) to identify the V-rich phase. From the corresponding XRD pattern (Figure 2(f)) of the V/17-4PH FZ interface of DB210, (FeV)ss is present in the FZ.

For all samples, since Fe and V were majorly dispersed in the FZ, the bcc phase is an (FeV)ss and most importantly, the (FeV)ss composition for all three joining conditions corresponds to the σ -phase homogeneity range according to the pseudo-binary phase diagram of 17-4PH and V. However, since XRD patterns did not indicate the presence of the σ -phase and EBSD/TEM barely showed its presence, it implies that the very rapid cooling from the AM process after each laser pass greatly suppressed (Fe-V-Cr) σ -phase formation.

The cooling rate is important in controlling σ -phase formation because high cooling rates can bypass the dangerous temperature range (300–900°C) wherein σ -phase precipitates, as observed in the calculation of phase diagram (CALPHAD) results shown in Figure 3(a) and (b). The CALPHAD technique is used to calculate the phase boundary and fraction of σ -phase formation in the 17-4PH/V FZ under equilibrium solidification (slow cooling). The equilibrium thermodynamic calculation

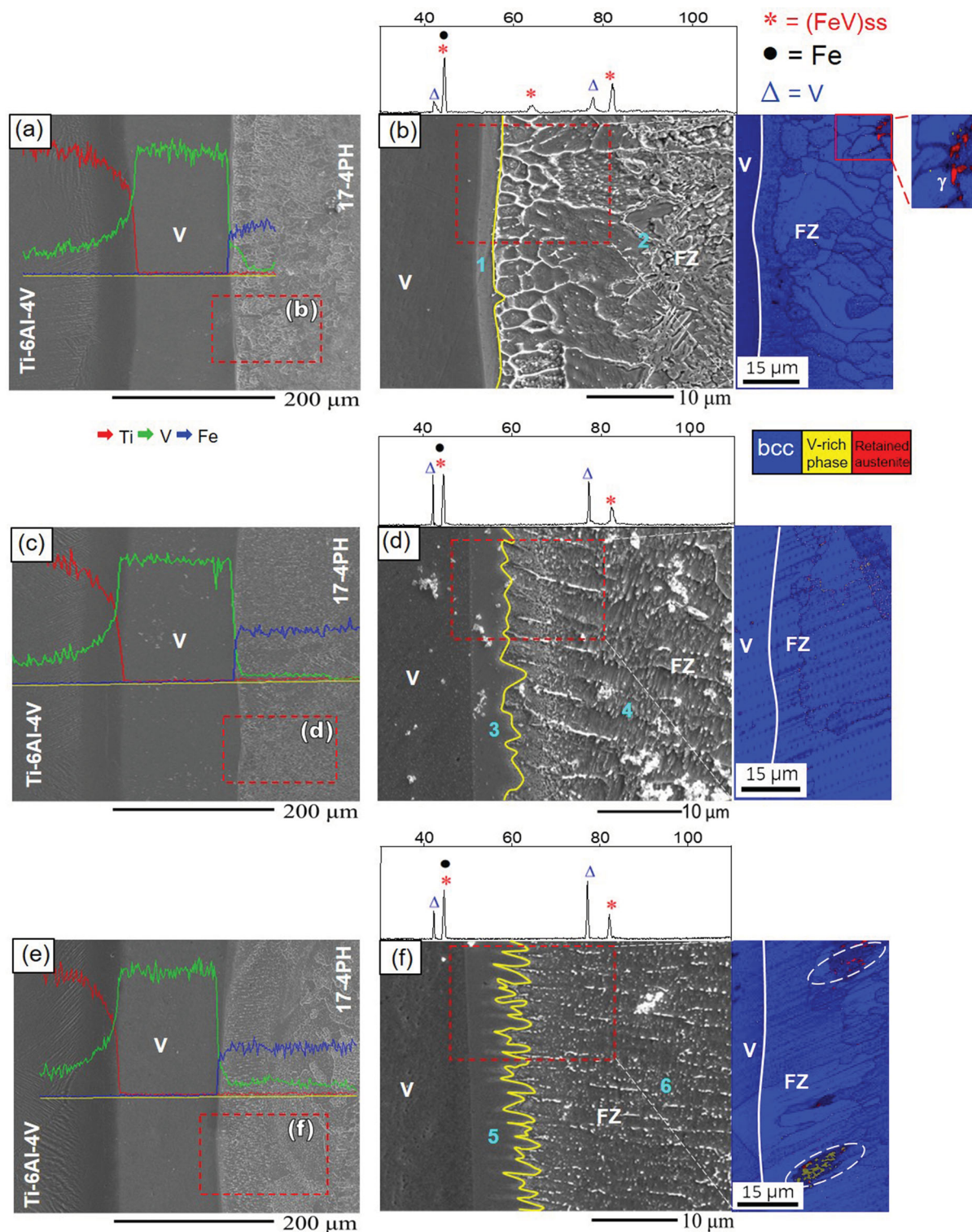


Figure 2. (a) SEM images, XRD patterns, EBSD images, and EDS line scans of (a, b) DB150, (c, d) DB180, and (e, f) DB210.

predicted that σ -phase would precipitate in the compositional range 20–60 wt.% V. Non-equilibrium solidification (fast cooling) calculation, as employed by the Scheil model, did not confirm the presence of σ -phase as shown in Fig. S1. In reality, the solidification speed

of the DED process is much faster than that assumed in typical Scheil modeling [15]; hence, because σ -phase formation was not predicted by the Scheil model, the high cooling rate from the DED process is concluded to effectively suppress σ -phase precipitation. The

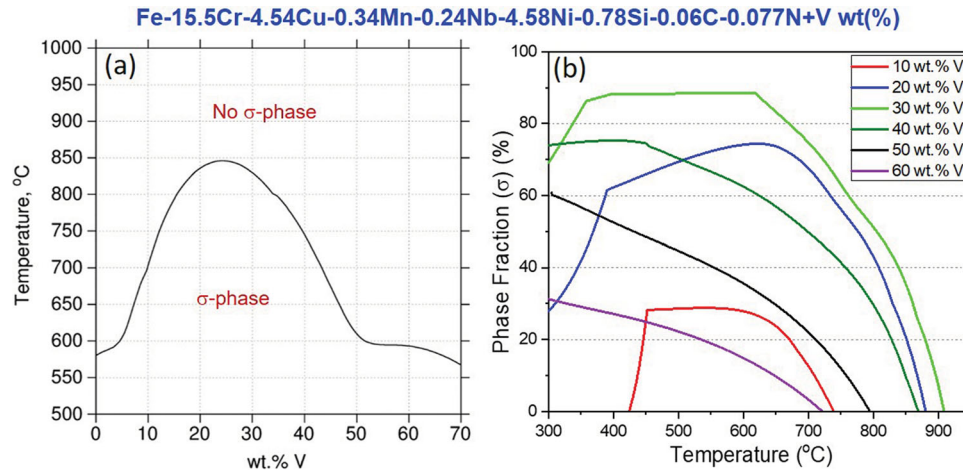


Figure 3. (a) Pseudo-binary phase diagram of 17-4PH showing the stability of σ -phase as a function of V concentration. (b) Phase fraction of the σ -phase in 17-4PH with V addition under equilibrium solidification (calculated by JMatPro software).

CALPHAD model is further described in the supplementary materials.

Microstructure of V interlayer fabricated by SLM

Figure 4 shows the microstructures and XRD patterns of the bonding interfaces of the samples when V interlayer was fabricated by SLM. Ti diffuses considerably into the V interlayer because the melting process is involved in V deposition, as shown by the EDS line scans in Figure 4(a) and (b). The scanning speed significantly affects the FZ nature. Obviously, a high scan speed permits less time for laser heating/melting, causing incomplete powder melting coupled with rapid solidification, thus leading to pore formation. Qiu et al. [16] measured the surface roughness and area fraction of porosity as a function of laser scan speed. They noted that unstable melt flows, especially at high laser scan speeds, increased the porosity and surface defects, consistent with our experimental results. The subsequent deposition of 17-4PH infiltrated the pores of the V layer with Fe, causing Fe-Ti intermetallic phase formation. SLM100, with a lower scan speed of 100 mm/s, displays fewer pores in the interlayer region than SLM600, which shows numerous macroscale pores. The crack and delamination regions in SLM600 after the deposition of 17-4PH stainless steel are attributed to the formation of Fe-Ti intermetallic compounds, as confirmed by the corresponding XRD pattern (Figure 4(b)). SEM observation of the bonding interface of SLM1200 with the highest scan speed was not performed because the joint experienced total delamination. XRD phase observation (Figure 4(c)) indicates the presence of Fe-Ti at the delaminated surface.

Tensile properties before and after PWHT

The joint strengths of DB150, DB180, DB210, and SLM100, as measured by tensile testing, are compared to that of pure V in Figure 5(a). Tensile results of SLM600 and SLM1200 are not presented because of the severe delamination of these joints. The difference in the tensile strengths of DB150, DB180, and DB210 is likely influenced by the nature of the melted zone between V and 17-4PH, because failure occurred at that joint interface. In the DED process, the nature of the molten pool and the grain structures formed during solidification significantly affect the mechanical properties of the part [2,17]. The low strength of 286 MPa is shown by DB150. The poorly bonded joint is attributed to the small partially mixed interfacial/bonding area at the molten pool by the insufficient melting of the substrate [18]. For DB180 and DB210, with laser powers of 180 and 210 W, higher strengths of 398 and 350 MPa are recorded, respectively. The laser powers for both samples ensure sufficient melting and adequate cooling, yielding a wider, well-structured, and fully formed FZ interface. However, the higher strength recorded from DB180 compared to that from DB210 can be attributed to the higher V content in the FZ ((FeV)ss) of DB210 than in DB180. Recent studies have demonstrated that FZs/(FeV)ss with higher V contents are more brittle and likely to demonstrate lower joint strength than those with lower V contents [10]. SLM100 shows the lowest tensile strength of 200 MPa, attributed to the insertion of Fe and Ti in the interlayer. The use of solid-state cladding for joining V to Ti-6Al-4V successfully prevents the incorporation and mixing of Ti and Fe in the V interlayer, as demonstrated by the higher strengths of the DB specimens (DB150, DB180, and DB210) compared to those using liquid-state deposition (SLM100, SLM600, and SLM1200).

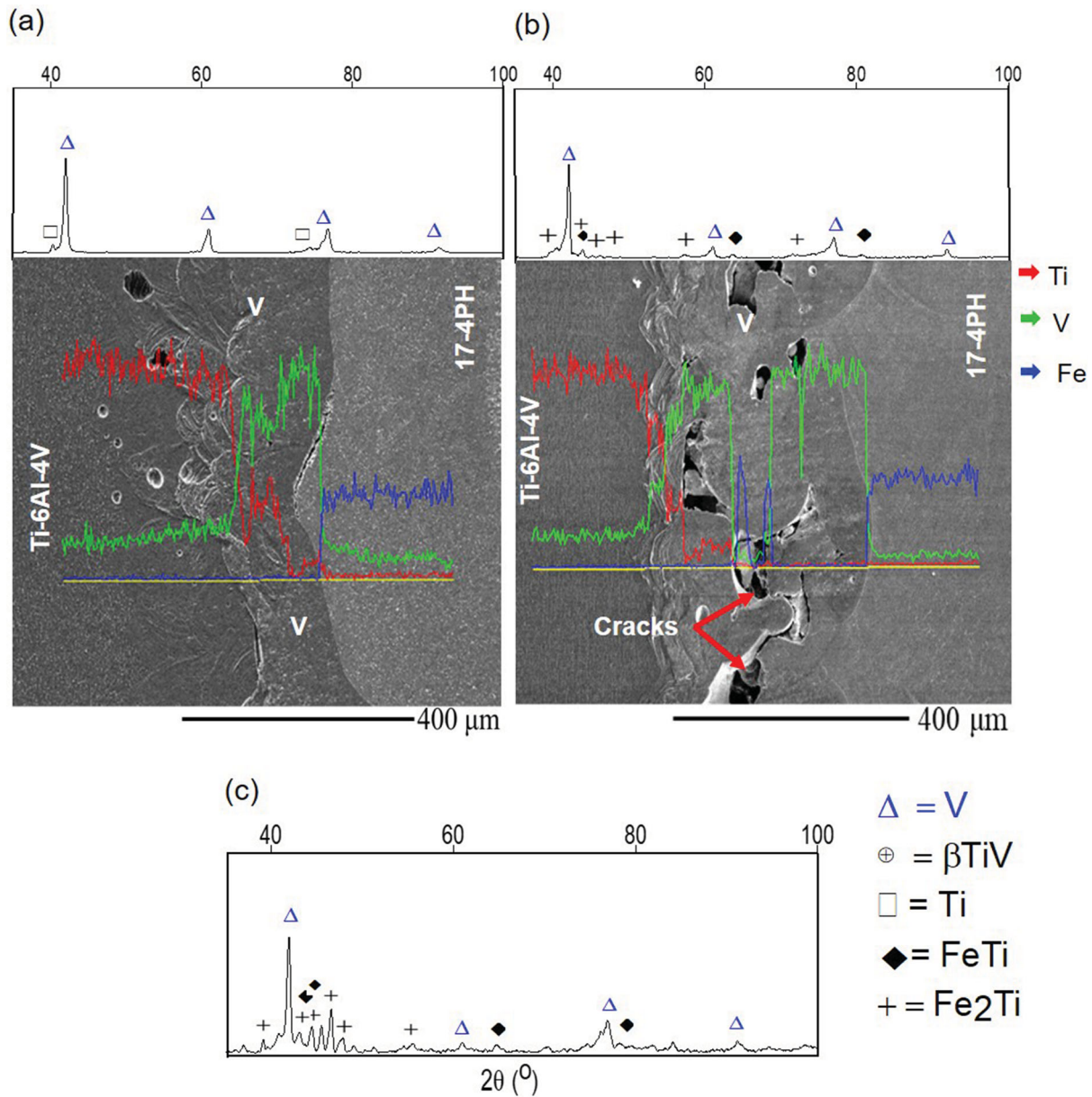


Figure 4. SEM image, EDS line scan, and XRD pattern of the bonded interface of (a) SLM100, (b) SLM600. (c) XRD pattern of the joint interface of SLM1200.

Considering these results, the V interlayer must be free of Fe and Ti; even limited mixing can form cracks or otherwise lower the bonding strength. The liquid-state deposition method has a very high likelihood of inducing the mixing and incorporation of Fe and Ti with the molten V interlayer. In the work conducted by Reichardt et al. [14], the observed mid-fabrication cracking in the buildup upon depositing adjacent layers was caused by the interaction of Fe and Ti. Moreover, the V gradient-composition interlayer already contained some Fe before deposition, facilitating intermetallic and σ -phase formation through the continuous melting and re-melting of layers. Here, even for pure V in laser

deposition, the incorporation of Ti and Fe still occurs, as confirmed by results from the SLM samples. The small V interlayer thickness (0.4 mm) appears to partially contribute to the easy mixing of Ti and Fe. It is therefore suggested to increase the interlayer thickness (to about 1 mm) and to optimize the laser scanning parameters to avoid pore generation and Ti/Fe mixing. An excessively high interlayer thickness, however, can generate detrimental buildup strain and residual stresses, thus reducing strength.

The effect of PWHT on the joint properties was studied. Tensile test specimens were taken from DB180 because it showed the highest strength. Figure 5(b) shows

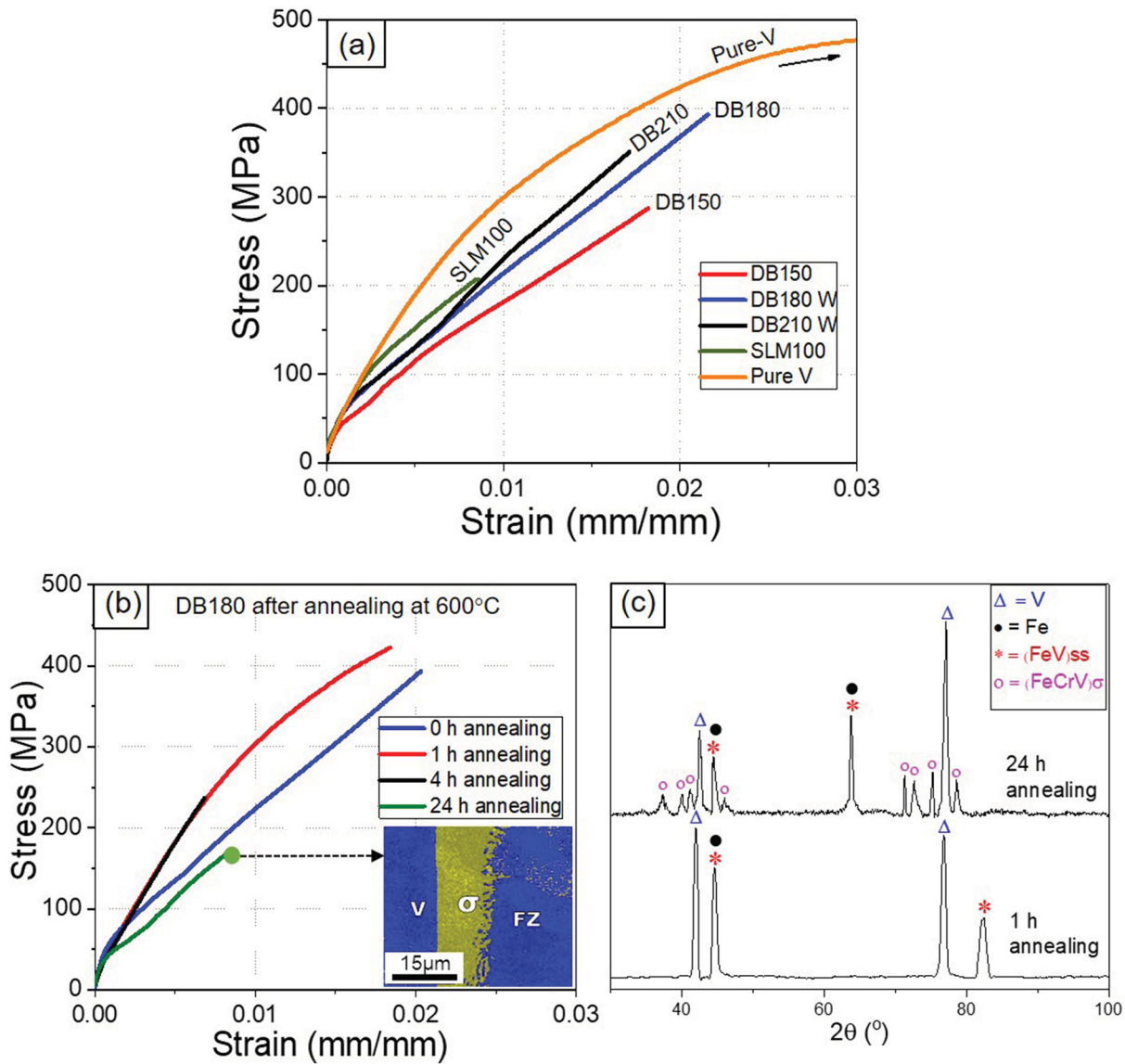


Figure 5. (a) Tensile stress–strain curves of DB150, DB180, DB210, and SLM100 as well as pure V at room temperature. (b) Tensile stress–strain curves of DB180 after annealing at 600°C for 0, 1, 4, and 24 h. Inset shows EBSD phase map at joint interface after 24 h. (c) XRD pattern of the V/17-4PH joint of DB180 after 1 and 24 h annealing.

the stress–strain curves of DB180 annealed at 600°C for 1, 4, and 24 h compared to the curve of the sample without annealing. No significant change in the strength of the joint is observed after 1 h annealing. Precipitation of σ -phase is not observed at the annealing time of 1 h, as evidenced in the XRD pattern in Figure 5(c). However, upon increasing the annealing time to 4 h and then 24 h, noticeable decreases in the joint strengths are observed. This can be attributed to the precipitation of the (Fe-V-Cr) σ -phase, which causes embrittlement of the joint interface and a dramatic reduction in strength. XRD phase identification of the bonded joint after 24 h of annealing is shown in Figure 5(c), verifying the presence of the (Fe-V-Cr) σ -phase. The EBSD phase identification map, inset in Figure 5(b), further confirms the

presence of a continuous σ -phase layer, unlike the EBSD phase maps in Figure 2. The amount of σ -phase precipitation increases with increasing annealing time [19]. This further explains why the specimen annealed for 24 h experienced a higher drop in strength than the specimen annealed for 4 h.

Conclusions

For the first time, AM was applied to obtain a strong joint between stainless steel and Ti-6Al-4V using a pure V interlayer. The melt zone characteristics and joint strength were dependent on the laser power; high powers of 180 and 210 W produced high-strength joints, while that of 150 W did not. The joint strength decreased with

increased annealing time, especially at 4 and 24 h, which demonstrated the presence of σ -phase. In addition, SLM of V at different scanning speeds on the surface of Ti-6V-4Al inevitably caused Ti incorporation in the deposited V interlayer, which affected the dissimilar joint formed between the interlayer and steel.

Disclosure statement

No potential conflict of interest was reported by the authors.

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