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Microstructures and tensile properties of Inconel 718 formed by high deposition-rate laser metal deposition

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The aim of this study is to characterize microstructures and tensile properties of Inconel 718 (IN718) formed by high deposition-rate laser metal deposition (LMD), and furthermore to verify that the properties of the material are equivalent to those obtained by conventional manufacturing processes, such as casting and forging, and therefore satisfy the specifications for industrial applications. Initially, the powdery additive was characterized in terms of chemical composition, morphology, and porosity. Afterward, blocks for producing tensile specimens were deposited by applying the newly developed high deposition-rate LMD process that has a deposition rate of approximately 2 kg/h. Finally, microstructures and tensile properties of directly deposited and heat-treated material were analyzed, respectively. From the results, precipitation of an irregular shaped phase, which is believed to be Laves phase, and segregation of Nb and Mo were found at interdendritic regions of the directly deposited material. The directly deposited material exhibited relative low tensile strength and 0.2% yield strength but high elongation. Moreover, due to recrystallization that occurred in heat treatment, columnar grains in the directly deposited material transformed to equiaxed grains. By heat treatment, Laves phase was dissolved, and three extra phases, which are believed to be δ phase, strength phases γ' and γ'' , were precipitated. After heat treatment, tensile strength and 0.2% yield strength of the material were significantly enhanced, whereas the plastic elongation decreased by approximately 38%. In comparison to conventional manufacturing technology, the heat-treated IN718 presented superior tensile strength, 0.2% yield strength, and plastic elongation to aerospace material specifications for casted and wrought IN718. © 2016 Laser Institute of America. [<http://dx.doi.org/10.2351/1.4943290>]

Key words: laser metal deposition, Inconel 718, high deposition-rate LMD, microstructures, tensile properties

I. INTRODUCTION

Laser metal deposition (LMD) is a process that uses a laser beam to form a melting pool into which powder is fed on a metallic substrate. The powder melts to form a deposit track that is fusion-bonded to the substrate. One layer is formed track by track, and a desired geometry can be built up layer by layer. LMD has various advantages such as lower

material waste and tooling costs compared to conventional manufacturing technologies, so there has been a growing interest in its development in recent years.^{1–5} Tensile properties are commonly used to validate the LMD technology and to determine if it is applicable to the manufacturing of desired components. Because microstructures of material have strong influence on tensile properties, studies on tensile properties are usually associated with microstructure investigations.^{6–12}

Inconel 718 (IN718) is a niobium-modified nickel-based super alloy, which has a high creep-rupture strength even at high temperatures to about 700 °C (1290 °F).¹³ IN718 has been the most widely used nickel-based superalloy in the aircraft engine industry over the past 40 years, and it has been used in many aircraft engine components like critical

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TABLE I. Chemical composition of IN718 in wt. %.

	Ni (+Cr)	Cr	Fe	Nb (+Ta)	Mo	Ti	Al	Co
Requirement	50.00–55.00	17.00–21.00	Balance	4.75–5.5	2.80–3.30	0.65–1.15	0.20–0.80	<1.00
Tested	53.51	19.08	17.51	4.89	2.98	0.99	0.68	0.16

rotating parts, airfoils, supporting structures, and pressure vessels, accounting for over 30% of the total finished component weight of a modern aircraft engine.^{14–16}

In 2005, Blackwell¹⁷ argued that prior to HIPing (Hot Isostatically pressing) the IN718 deposit exhibited anisotropic properties that appeared to be associated with nonoptimized processing conditions, and HIPing led to a reduction in anisotropy within the deposit, but generated considerable grain growth within the (IN718) substrate. In 2008, Zhao *et al.*¹⁰ studied the microstructure and mechanical properties of IN718 formed by LMD and showed that the ultimate tensile strength of heat-treated material is comparable to that of the wrought IN718, which is 1.5 times of that of the directly deposited samples. As reported by Zhang *et al.*¹⁸ in 2011, Nb, Mo, and Ti concentrated in the columnar crystal trunk in directly deposited IN718, but after heat treatment, the segregation was minimized, and the material was strengthened by precipitated Nb- and Mo-rich phases. In the same year, Tabernero *et al.*¹⁹ found that the directly deposited IN718 presented high anisotropy. In 2013, Lambarri *et al.* proved that the heat-treated IN718 formed by LMD satisfy the industrial requirement for aeronautic applications, with mechanical properties well above the minimum specified values at room temperature and with no detrimental phases or precipitates left after heat treatment.

For several years, effort has been devoted to the study of microstructures and tensile properties of IN718 formed by conventional LMD. However, studies on this area for high deposition-rate LMD are lacking. On the other hand, most of the previous studies were based on conventional LMD processes whose deposition rates are normally lower than 0.3 kg/h, so the key limitation of these studies is that their conclusions may not be practical in high deposition-rate LMD. In this respect, the objective of the current study is to characterize

microstructures and tensile properties of IN718 formed by high deposition-rate LMD with a deposition rate of approximately 2 kg/, and furthermore to verify if the properties of the material formed by the developed high deposition-rate LMD process are equivalent to those obtained by conventional manufacturing process such as casting and forging and therefore satisfy the specifications for industrial applications.

II. MATERIALS AND METHODS

A. Materials

The powdery additive used in the current study was Inconel 718 (IN718) gas atomized (GA) powder. The chemical composition of the main elements of the used IN718 powder measured via inductively coupled plasma (ICP) emission spectroscopy as well as the limiting chemical composition (Cold-rolled sheet heat-treated in accordance with AMS 5596B) of IN718 are given in Table I.

The nominal particle size of the used powder is 45–90 μm . Metallographic prepared cross sections and SEM (scanning electron microscope) micrographs are shown in Fig. 1.

As can be seen in Fig. 1: (a) a large fraction of the powder particles feature satellites, which are formed during atomization of the powder, as smaller molten particles are solidified faster and adhered to still semifluid, larger particles, (b) a few particles with enclosed pores, which are also formed during the manufacturing process, can be located (hollow particles), and (c) a certain amount of irregular shaped particles can be found.

B. Methods

The experimental setup consists of a collimator, a standard optic, a zoom optic, and an ILT-Coax 50 powder nozzle. The

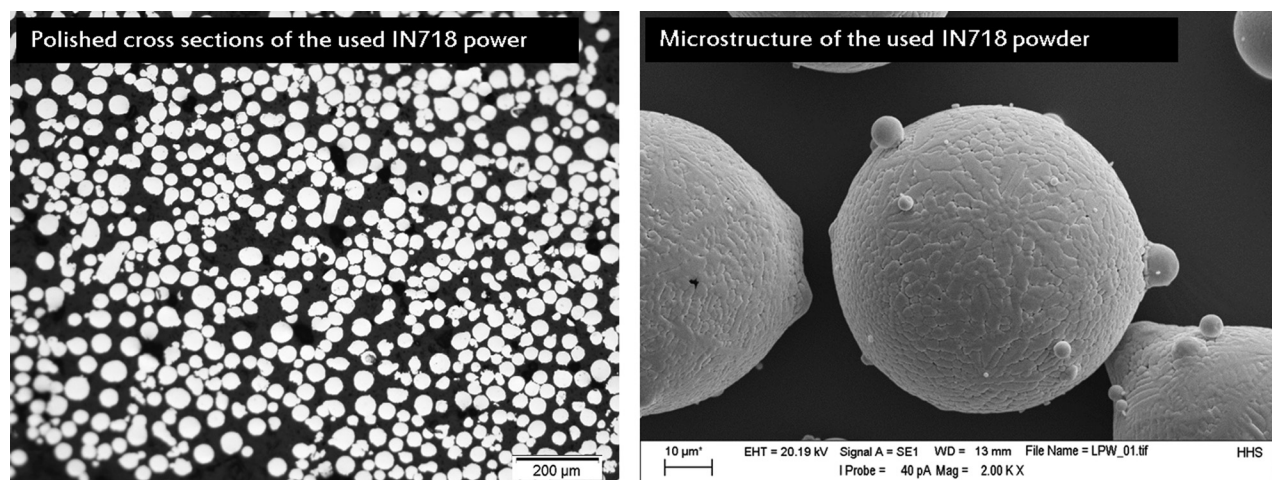


FIG. 1. Metallographic prepared cross section and SEM picture of the used IN718 powder.

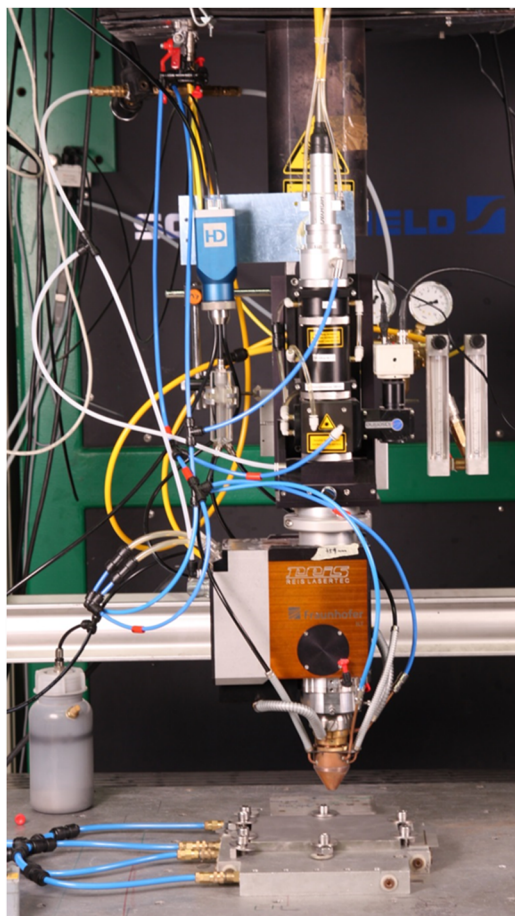


FIG. 2. Overall view of the experimental setup.

movement of the lenses in the zoom optic and the tool axis are controlled by the NC-control of a four-axes tool machine. A 12 kW diode laser source is linked via a glass fiber. The overall view of this experimental setup is shown in Fig. 2.

The powder is fed by the use of Helium (4 Nl/min) with a powder feeder. To prevent the melt pool from interacting with atmospheric gases such as O_2 and N_2 , argon is used as shielding gas (12 Nl/min). Other parameters such as laser power P_L , scanning speed v , powder flow rate \dot{m} , and laser spot diameter d_L for depositing blocks are listed in Table II, and eight identical blocks with the dimensions of $160\text{ mm} \times 28\text{ mm} \times 5\text{ mm}$ were deposited. The scanning pattern and some of the deposited blocks are shown in Fig. 3.

Refer to AMS (Aerospace Material Specification) 5583D, which states the heat treatment method for casting IN718, four deposited blocks were heat treated by a three-step heat treatment method that includes homogenization (heat to $1093 \pm 14^\circ\text{C}$, hold for 2 h, air cool or faster), solution (Heat to $954 - 982^\circ\text{C}$, hold for >1 h, air cool or faster) and

double aging (Heat to $718 \pm 8^\circ\text{C}$, hold for 8 h, furnace cool to $621 \pm 8^\circ\text{C}$ air hold for 10 h, air cool or faster).

According to the specifications of DIN (Deutsches Institut für Normung) 50125, three directly deposited and three heat-treated tensile specimens of form E with the dimension of $115\text{ mm} \times 12\text{ mm} \times 3\text{ mm}$ as shown in Fig. 3 were manufactured from the blocks by milling and wire electrical discharge machining. These six test specimens were used to characterize the tensile properties of the directly deposited and the heat-treated material.

The remaining two blocks were used for analyzing microstructures of directly deposited and heat-treated material.

III. RESULTS AND DISCUSSION

A. Microstructures

After grinding, polishing, and etching, optical micrographs showing microstructures of the cross section, the longitudinal section, and the transverse section of the deposited IN718 are presented in Fig. 4.

Mainly two kinds of regularly orientated columnar grains, namely, columnar grains oriented along deposition direction and columnar grains along track edges to track top, can be seen in Fig. 4. This indicates that there were two dominating solidification directions which were along deposition direction and along track edges to track top, and the dominant directions of heat flow were exactly the opposite of solidification directions.

The orientation of the columnar grains observed from the optical micrograph of longitudinal section is different from the previous works done by Parimi *et al.*²⁰ and Lakshmi *et al.*²¹ Both of them found that the average grain orientation angle was about 50° – 60° to the substrate in all the layers in single direction deposited wall. The different observations may due to the differences of process parameters, scanning pattern, powder characteristics, etc.

It is noteworthy that these exists quite a lot pores in the built volumes. In order to characterize the porosity, we selected randomly four cross sections and measured the porosity using image processing software that integrated to the optical microscopes. The result showed that the porosity of these built volumes is approximately 0.94%.

SEM micrographs showing intermetallic precipitates and Energy Dispersive X-ray (EDX) analysis showing interdendritic segregation of the directly deposited material are shown in Fig. 5.

As shown in the SEM micrograph of Fig. 5, the deposited material presented a dendritic microstructure, and white irregularly shaped phase was observed in the interdendritic regions. The size of this phase was about $1\text{--}4\text{ }\mu\text{m}$. EDX analysis results in Fig. 5 show that the precipitated phase is rich in Nb and Mo, which are principle elements of Laves phase, compared to the γ matrix.

Because of the use of high concentration refractory elements, especially Nb and Mo, the solidification process of IN718 alloy is often associated with segregation, resulting in the formation of Mo- and Nb-rich brittle intermetallic compound Laves phase that is located at the interdendritic regions, represented as $(\text{Ni,Cr,Fe})_2(\text{Nb,Mo,Ti})$.^{16,21,22} Qi *et al.*¹⁶ and Lakshmi *et al.*²¹ observed precipitates with

TABLE II. Range of process parameters.

P_L (W)	v (mm/min)	\dot{m} (kg/h)	d_L (mm)
3400	1500	2.05	4

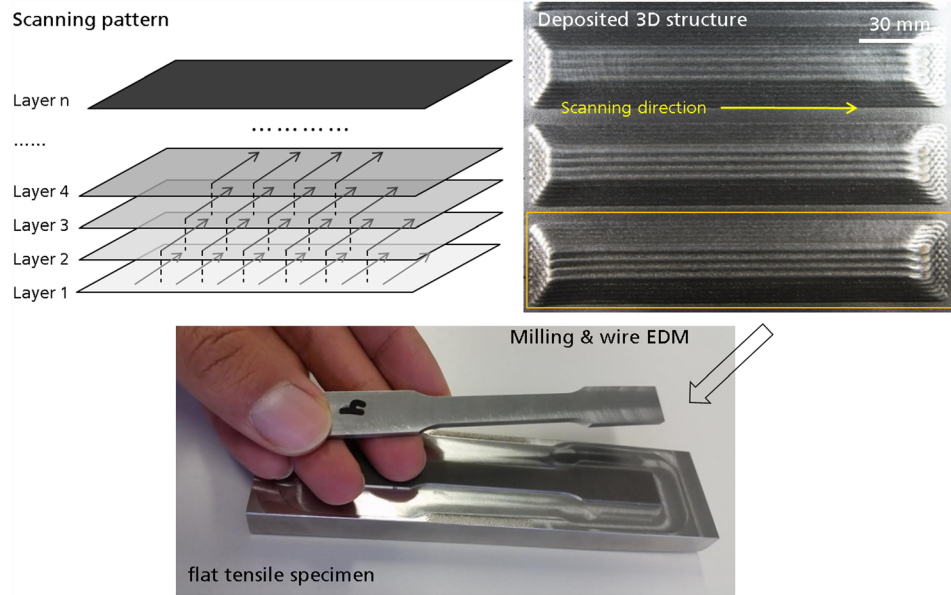


FIG. 3. Scanning pattern for the deposition of blocks for tensile specimens, three deposited volumes, and one finished tensile specimen.

similar morphology and element segregation in conventional LMD, and it was confirmed as Laves phase. Therefore, it is thought that the observed interdendritic precipitated phase in this current work is Laves phase.

No obvious strength phase was observed in the deposited material, but a great fraction of Laves phase, which is detrimental to mechanical properties, was found. It can be inferred that, the mechanical properties of IN718 formed by high deposition-rate LMD should be poor. This was confirmed by tensile test results, and these results are presented and discussed in Sec. III B.

Optical micrographs showing grain morphology and SEM micrographs showing intermetallic precipitates of the

heat-treated IN718 formed by high deposition-rate LMD are shown in Fig. 6.

Equiaxed grains and an isotropic appearance were observed from the optical micrographs of different sections of the heat-treated IN718 in Fig. 6. This should be the result of homogenization during the heat treatment process; a homogeneous recrystallization was induced. As shown in SEM micrographs, there are mainly three kinds of white precipitated phases: plate- or needlelike phase which has a longitude dimension from a few microns to tens of microns and is located in grain boundaries; two kinds of granular particles with different dimensions, which are uniformly distributed in the material. But no Laves phase is observed anymore.

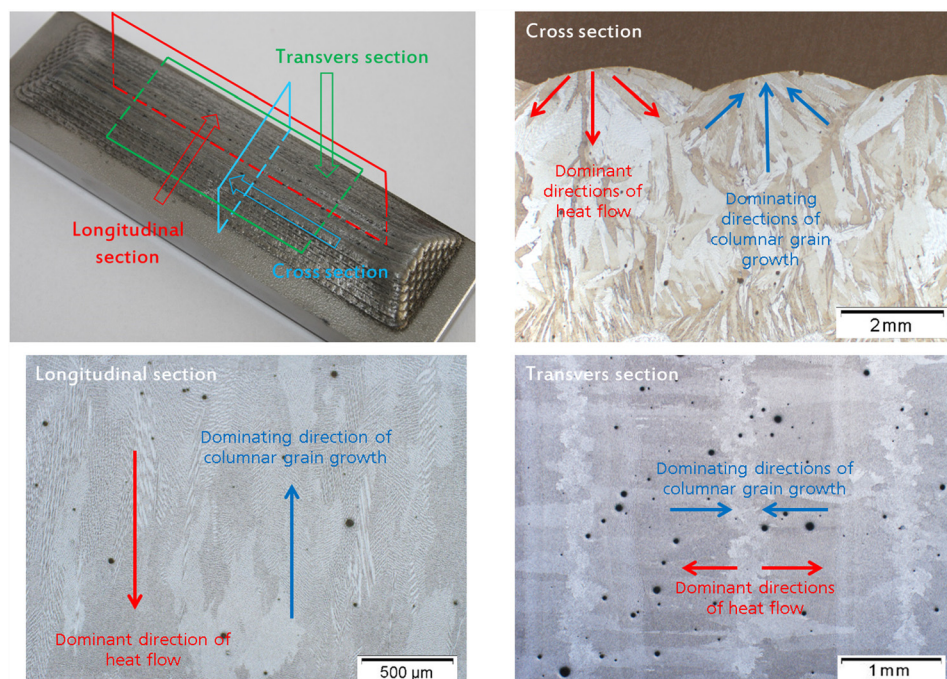


FIG. 4. Microstructure of IN718 formed by high deposition-rate LMD.

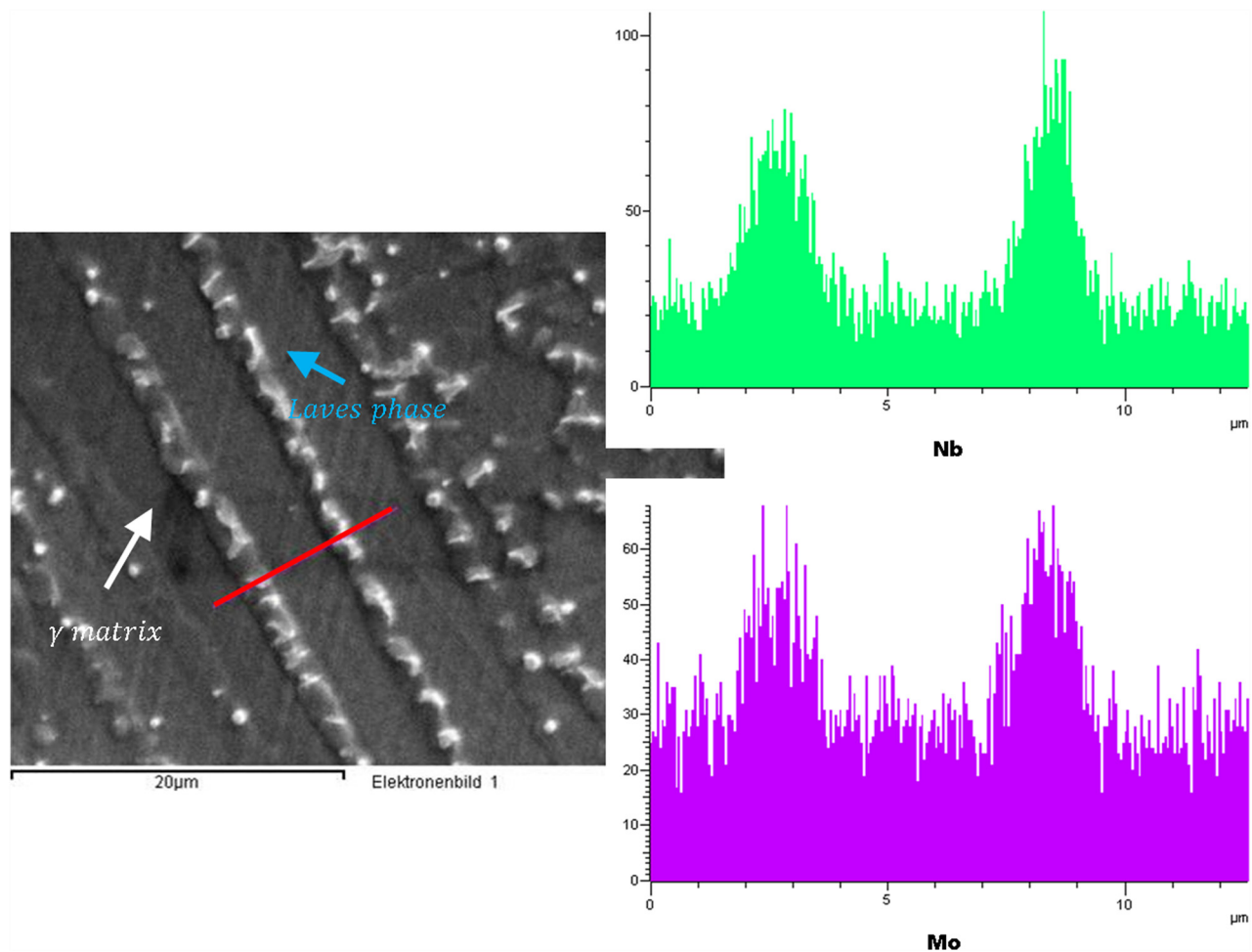


FIG. 5. SEM micrographs showing the intermetallic precipitates of the deposited material and EDX analysis showing interdendritic segregation.

The plate- or needlelike precipitates are presumed to be δ phase. δ phase, which has a composition of Ni_3Nb and an orthorhombic (D0_a) crystal structure, precipitates in between 700°C and 1000°C ,²³ and it is mostly found as plates growing on the (1 1 1) planes or found on the grain boundaries.^{24–26} The temperature of solution heat treatment is just the temperature for δ phase precipitation, and the morphology of these white precipitates satisfies the morphology of δ phase. Qi *et al.*¹⁶ and Chang and Nahm²⁷ also observed precipitates with similar morphology, and it was confirmed as δ phase in their work. Therefore, it is believed that during solution heat treatment, most Laves phase have been transformed to plate- or needlelike δ phase at the interdendritic regions.

It is assumed that the extra precipitated granular phases are strengthening phases γ' and γ'' . γ' and γ'' phases, which precipitate between 600°C and 900°C , are uniformly distributed small particles and form a basis for the precipitation hardening of the alloy.^{23,28–33} They have distinctly different morphologies which help in their identification; γ'' phases are round shaped and about 20 nm in size while γ' precipitates are disklike with an aspect ratio of 5–6.^{24–26} First, double aging is the last step of the applied heat treatment of our work, and these aging temperatures are just temperatures for γ' and γ'' phases precipitation. Second, the morphologies of

the observed precipitates are in accordance with the morphology of γ' and γ'' phases. Finally, IN718 is strengthened by precipitating γ' (Ni_3Nb) and γ'' ($\text{Ni}_3(\text{Al,Ti})$) phases in the γ matrix after a full heat treatment,^{16,34} and the tensile test results which are discussed in Sec. III B show that the material is dramatically strengthened by heat treatment. Therefore, it is believed that after double aging, γ' and γ'' phases were precipitated. SEM analysis has been done, but they could not be analyzed by EDX due to their nanometric size. In order to confirm this conclusion, further analysis such as transmission electronic microscopy analysis and X-Ray Diffraction analysis are necessary.

In summary, during heat treatment, Laves phases which are the typical detrimental phase of IN718 were dissolved to γ matrix or transformed to δ phase, and strength phases γ' and γ'' were precipitated. Therefore, the mechanical properties should be enhanced by heat treatment.

B. Mechanical properties

Figure 7 presents the average ultimate stress, 0.2% yield strength, and plastic elongation of the tensile test results of IN718 formed by high deposition-rate LMD with and without heat treatment. These results were compared with AMS specifications for casting and wrought IN718, respectively.

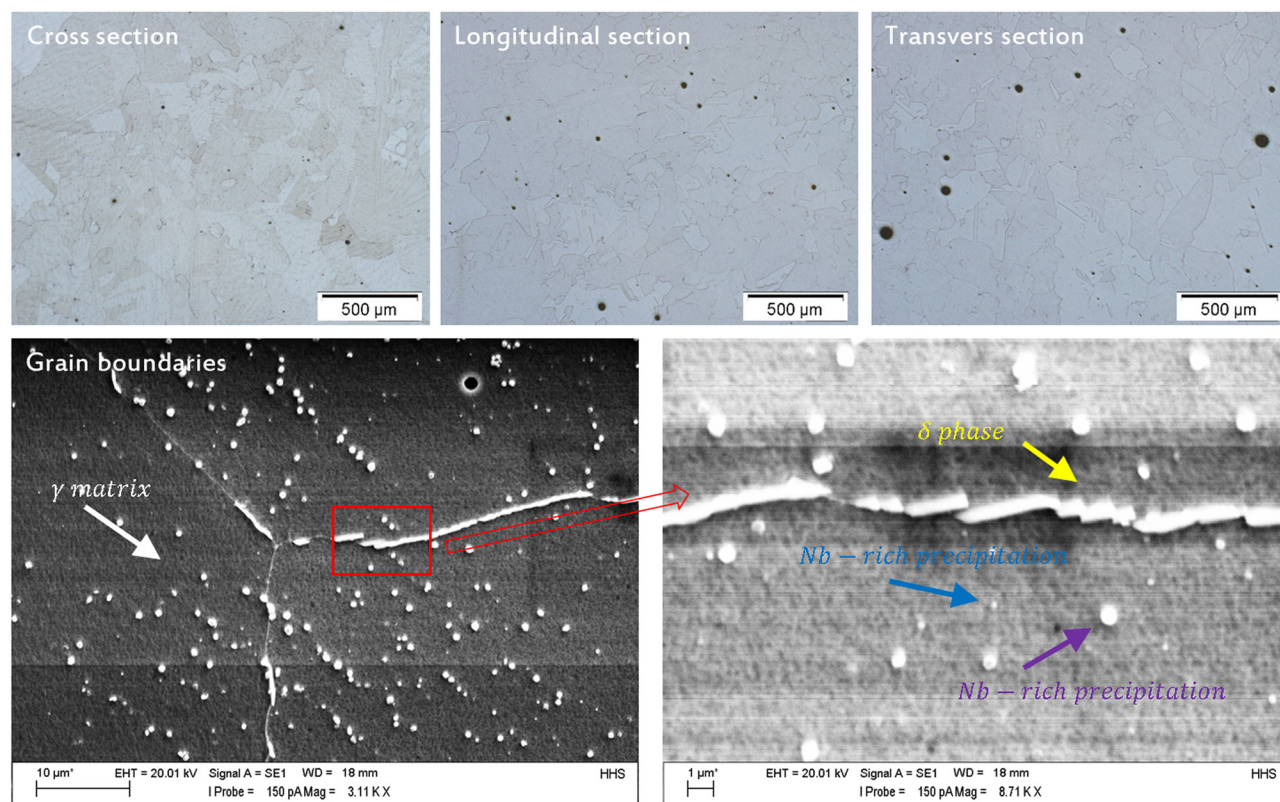


FIG. 6. Microstructures of the heat-treated IN718 formed by high deposition-rate LMD.

As can be seen in Fig. 7, AMS specifications in terms of ultimate strength, 0.2% yield strength, and plastic elongation for casting IN718 are lower than those specifications for wrought IN718, and the ultimate strength and 0.2% yield strength of direct-deposited material are lower than the specification for casting IN718, but its plastic elongation is higher than both of these specifications for casting and wrought material. After heat treatment, the average ultimate stress increased by approximately 54% to 1303 MPa, while the average yield stress increased by approximately 115% and reached 1127 MPa. By heat treatment, both ultimate strength and 0.2%

yield strength increased significantly, and these values are above AMS casting and wrought properties. Although the plastic elongation of the heat-treated material compared with the direct-deposited material dropped significantly from 29 to 16, the final value is still higher than AMS specifications for both casting and wrought material. Compared to the heat-treated materials, the directly deposited material was soft and produced low yield and ultimate stresses, while demonstrating great ductility. It is clear that by heat treatment, the precipitation of strength phase and the dissolution of detrimental phase have improved the microstructures of the material, resulting in

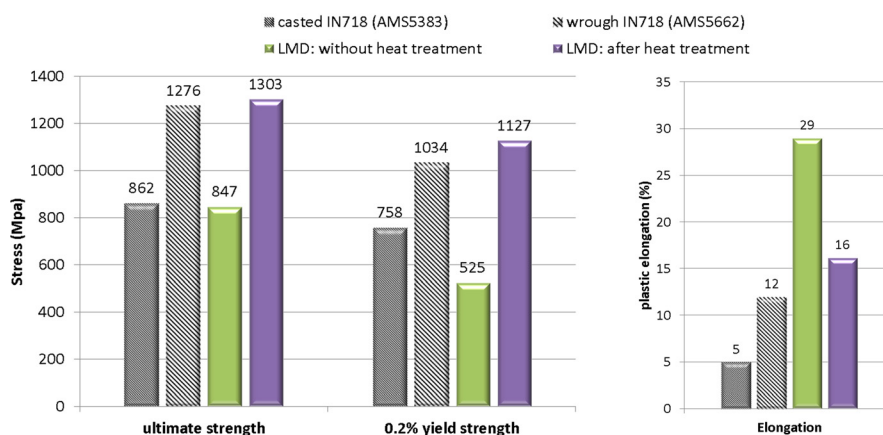


FIG. 7. Tensile test results showing the averaged ultimate stress, 0.2% yield strength, and plastic elongation compared with AMS specifications for cast and wrought IN718, respectively.

the enhancement of its tensile strength. In summary, the tensile test results indicate that by heat treatment, tensile strength and 0.2% yield strength of IN718 formed by high deposition-rate LMD were improved, while the ductility was reduced, but all of these indexes exceeded AMS properties of casting and wrought IN718.

IV. CONCLUSIONS

Microstructure and tensile properties of IN718 formed by high deposition-rate LMD have been studied in the current paper. Based on the high deposition-rate LMD process with a deposition rate of approximately 2 kg/h, blocks for producing tensile specimens were deposited. Afterward, refer to the heat treatment method for casted IN718, a three-step heat treatment process was carried out for some of the deposits. Finally, microstructures and tensile properties of directly deposited and heat-treated IN718 were analyzed. In comparison to conventional manufacturing technology, the heat-treated IN718 presented superior tensile properties in terms of tensile strength, 0.2% yield strength, and plastic elongation in reference to AMS specifications for casted and wrought IN718.

The directly deposited material exhibited two kinds of regularly orientated columnar grains, namely, columnar grains oriented along deposition direction and columnar grains along track edges to track top, which indicates the solidification directions of the process. SEM and EDX analysis showed Nb and Mo segregation at interdendritic regions, and precipitation of irregular shaped phase, which is believed to be Laves phase and is detrimental to material mechanical properties, was observed at interdendritic regions. In addition, no strength phase was found in the directly deposited material. Therefore, the tensile strength and 0.2% yield strength of the directly deposited material were relative low. Considering the high value of plastic elongation, the directly deposited material is quite soft.

Equiaxed grains were observed in the heat-treated material, which was due to recrystallization occurred during the heat treatment. After heat treatment, Laves phase was dissolved, three extra phases, namely, plate- or needlelike δ phase, strength phase γ' and γ'' were precipitated. Microstructure was improved by heat treatment, therefore, the tensile strength and 0.2% yield strength were significantly enhanced, and both of them are superior to AMS specifications for casted and wrought IN718. Although the plastic elongation of heat-treated material decreased, which means the ductility of the material was reduced, the value is still higher than AMS properties for casted and wrought IN718.

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