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Effect of rolling force on tensile properties of additively manufactured Inconel 718 at ambient and elevated temperatures

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Abstract

Inferior mechanical properties and severe anisotropy behavior of wire + arc additive manufactured (WAAM) Inconel 718 due to the large epitaxial grown columnar dendrites restrict the industrial application of WAAM deposition. Cold rolling was integrated into the WAAM deposition process and the effect of rolling force on microstructure, precipitation distribution and tensile properties at ambient and elevated temperatures were investigated. The results show that the hardness of cold-rolled samples is much larger than that of the as-deposited and it increases with the increase in the rolling force. The columnar dendrites of the as-deposited sample changed to finer equiaxed grains of 26.5 and 14.7 μm after cold rolling with the force of 50 kN and 75 kN, respectively. Meanwhile, more uniformly distributed grains and less δ phase appear for 75 kN rolled sample. The stress-strain curves are smooth for the tensile tests at ambient temperature, while there are serrations at elevated temperature due to the dynamic strain aging behavior. The as-deposited sample shows inferior tensile properties to the wrought material at ambient and elevated temperatures. The cold-rolled samples both exceed the wrought material at ambient temperature; however, they show higher strength but lower elongation compared to the wrought material. The 75 kN cold rolled sample shows much higher strength and similar elongation to the wrought material for the test at elevated temperature. The grain morphology and recrystallization, as well as the strengthening mechanism of hybrid deposition and cold rolling process were discussed.

Keywords: Wire + arc additive manufacture; cold rolling force; microstructure; elevated tensile properties; dynamic strain aging; strengthening mechanism

1 Introduction

Inconel 718 (IN718) has been widely used in the crucial heated end components of gas turbine engine due to its weldability, excellent mechanical performances of high strength, fatigue life, and corrosion resistance and thermal stability at high temperatures [1-2]. In addition to the austenitic matrix, the brittle Laves phase, MX carbides, and the δ phase usually appear during the welding process of IN718; meanwhile, the main strengthening phases of γ'' and γ' phases are precipitated after heat treatment. The strength of IN718 is significantly enhanced through heat treatment due to the precipitation strengthening mechanism [3].

Additive Manufacturing (AM) has been considered as modern technology for the manufacturing of metallic components, the technique has the advantage of being flexible and rapid. The AM mainly consists of powder bed fusion (PBF) and direct energy deposition (DED) based on the different feeding methods of the feedstock [4]. Wire + Arc Additive Manufacture (WAAM), using an electric arc as the heat source and metal wire as the feedstock, has been regarded as a potential technology for the manufacturing of large-scale metal components. The process is cost-effective due to the low material and equipment cost, as well as high deposition rate and flexibility [5]. More importantly, the size of the WAAM deposited components can be significantly enlarged without the limitation of the vacuum chamber of the PBF process [6-7].

Many previous studies on the microstructure and mechanical properties of additive manufactured IN718 have been reported. Huang et al. [8] studied the microstructure and mechanical behavior of selective laser melting (SLM) processed IN718. Its mechanical properties were neither of the parts fabricated by casting and those by forging because the γ'' phase and γ' phase were distributed in the crystal, while the carbide phase and Laves phase were distributed in the inter-crystal region. Zhao et al. [9] comparatively studied the microstructure evolution of IN718 fabricated by SLM and wrought process during subsequent heat treatment process and their corresponding mechanical properties. The solution treatment did not change the grain size of SLM-IN718 specimen and retained its dislocation subgrain structure, and the subsequent aging process improved the precipitation of γ'' strengthening phase. As a result, the hardness and strength of SLM-IN718 were significantly enhanced, which far exceeded that of wrought IN718. Ni et al. [10] investigated the microstructure and anisotropic mechanical properties of SLM processed IN718 component and determined that the as-fabricated longitudinal samples showed lower ultimate tensile strength, while higher elongation was observed compared to the transverse samples. The anisotropy in strength and ductility was attributed to the {100} fiber texture and columnar grain morphology. Zhao et al. [11] found that the fine cellular subgrain structure was formed in SLM-IN718 material and it was reserved after holding at 1080 °C for 50 min. The reserved cellular subgrain structure promoted the precipitation of γ'' second phases, which hindered the motion of dislocation and contributed to the increase of the strength. Zhang et al. [12] compared the effect of standard solution+ aging (SA) heat treatment and homogenization +solution+ aging (HSA) heat treatment on the microstructure and mechanical properties of the SLM manufactured IN718 alloy. The HSA heat treatment showed a bit lower strength but higher elongation compared to the SA heat treatment. In addition to the PBF process of IN718, several studies of WAAM deposited IN718 alloy had also been conducted. Asala et al. [13] conducted a detailed study on the microstructure evolution of WAAM deposited 718Plus super-alloy by using a tungsten inert gas (TIG) welding process, which was vital to the comprehensive understanding of material properties produced by the WAAM technique. Furthermore, cold metal transfer (CMT) is a new welding technology without splash to produce components with high quality. Xu et al. [14] systematically evaluated the effect of oxide, wire source and heat treatment on the mechanical properties of WAAM deposited IN718. The as-deposited IN718 featured large columnar grains and numerous Laves phases, which caused lower strength than the wrought alloy. Oguntuase et al. [15] investigated the influence of post-deposition heat treatments on the microstructure and mechanical properties of WAAM deposited 718Plus, and developed a new heat treatment to significantly improve the mechanical properties and reduce the anisotropic effect. As IN718 was used as the main base material for the aerospace components operated at high temperatures, its mechanical properties at elevated temperatures (650°C) are essential. Zhang et al. [15] studied the effect of cold rolling on ductility, as well as the role of δ phase with regards to the differential morphologies in tensile behavior of IN718 alloy at 650 °C, and it was observed that the cold-rolling was beneficial in enhancing the resistance to crack propagation. The needlelike δ phase tightly hindered the migration of horizontal grain boundaries to enhance the strength, while promoting the intergranular brittle cracking, which results in the degradation of ductility. However, the modified granular δ phase in cold-rolled structures showed good deformation compatibility with horizontal grain boundaries, playing a positive role in delaying necking occurrence and impeding crack propagation. Trosch et al. [17] studied the microstructure and mechanical properties of SLM-processed IN718 compared to forging and casting processes, and performed the tensile tests at room and elevated temperatures (450 °C and 650 °C). The SLM-processed parts show superior mechanical properties than forged and cast material at room temperature, while similar properties were observed with the forged material at elevated temperatures with a high proportion of intragranular δ -phase in the SLM-processed material. Seow et al. [18] proposed a new modified heat treatment, which was found to be effective in dissolving Laves and δ phase, thus leading to low ductility. The modified heat treatment reduced the

anisotropy in grain structure, resulting in almost isotropic tensile properties at elevated temperature, which meet the minimum specifications for the conventional cast but not for wrought material.

Large columnar dendrites were the typical microstructure of WAAM deposited metal alloy, resulting in inferior mechanical properties and anisotropic behavior of the components. Therefore, inter-pass cold rolling was integrated into the WAAM deposition process to change and refine the microstructure, as well as to enhance the mechanical properties of the material. The inter-pass cold rolling changed the columnar dendrites of the WAAM deposited Ti-6Al-4V components to fine equiaxed grains with a rolling force of 75 kN, thus significantly enhancing the strength and improving the isotropic behavior [19]. Many WAAM deposited materials had inferior properties to their wrought counterparts, which impeded industrial implementation of the technology. The hybrid WAAM rolling process can provide significant benefits including reducing residual stresses and distortion, and grain refinement for titanium alloys, which results in isotropic mechanical properties that can be better than the wrought material [20]. This hybrid technique may become a promising method for the manufacturing of aerospace components. In addition, a skin part of the aircraft with no hot cracking and porosity inside the part was successfully achieved using this hybrid technique [21]. Porosity defect was a major problem for WAAM deposited aluminum alloy. Gu et al. [22] studied the spatial distribution, size, and shape of micropores by X-ray diffraction tomography, and revealed their formation and evolution mechanisms during the deposition and heat treatment of the WAAM deposited 2319 aluminum alloys. Inter-layer rolling [23] was introduced to eliminate micropores, the number, volume, size, and roundness of micropores in rolled alloys decreases similarly with increasing rolling loads. The evolution mechanisms include the reprecipitation of hydrogen pores, formation of vacant voids, and re-opening of unclosed pores, indicating that the hybrid technique of WAAM + cold rolling + heat treatment had great potential in promoting mechanical properties of WAAM deposited alloys [24]. Xu et al. [25] applied a cold rolling process to the plasma deposited IN718, concluding that the tensile strength of the inter-layer rolled samples exceed that of the wrought material.

However, many previously published research focused on the mechanical properties of the additive manufactured material at ambient temperature, or the tensile properties at elevated temperature. The effect of different rolling forces on the microstructure and tensile properties at ambient and elevated temperature has not been extensively investigated. In this study, the comparisons of hardness, microstructure, ambient, and elevated tensile properties under different cold rolling forces were analyzed. The reasons for the grain morphology and recrystallization mechanism induced by the cold rolling process were discussed. Finally, the strengthening mechanism of hybrid WAAM deposition and cold rolling process under different rolling forces was analyzed.

2 Material and methods

Fig.1 shows the WAAM deposition and hydraulic cold rolling system. A CMT power source (Fronius, CMT 4000 advanced) and a CMT torch are attached to the ABB robot to achieve the WAAM deposition process. After depositing one layer, the cold rolling process with different rolling forces controlled by the hydraulic system was applied on the surface of each deposited layer. The diameter of the IN718 wire used is 1.2 mm and its compositions (wt.%) are: 53.15 Ni - 19.42 Cr - 5.22 Nb + Ta - 2.95 Mo - 0.96 Ti - 0.47 Al - 0.41 Cu - 0.11 Mn - Bal. Fe. Its tensile strength is 1138 MPa with double aging treatment. A flat roller was adopted in this study. Table 1 shows the parameters for WAAM deposition and cold rolling process.

Table 1 Parameters for WAAM deposition and cold rolling

Parameter	Value
Contact tip-to-work distance	14 mm
Wire feed speed	7 m/min
Torch travel speed	6 mm/s
Rolling force	0, 50, 75 kN
Roller diameter	100 mm
Roller width	20 mm
Roller travel speed	5 mm/s
Inter-pass cooling time	3 mins
Shielding gas flow rate	15 L/min

Three comparative walls were built: as-deposited (AD), cold-rolled (CR) with a rolling force of 50 kN and 75 kN, respectively. Standard heat treatment was applied to the samples: they were heated to 970 °C and held for 1 h, followed by water quenching in the solution treatment, then heated to 720 °C and held for 8 h, and were cooled in the furnace to 620 °C, then held for 8 h followed by air cooling for the double aging treatment.

Both walls' end of about 10 mm was cut off. The specimens were ground, polished, and etched for microstructure analysis and hardness examination. The grain morphology and precipitation distribution were analyzed using optical microscopy (OM, OPTIPHOT, NIKON) and scanning electron microscopy (SEM, FEI XL30-SFEG) with an energy dispersive spectrometry (EDS, OXFORD INSTRUMENTS). HV hardness was measured under a load of 1 Kg and a holding time of 15 s using Zwick/Roell hardness tester (ZHV μ /ZHV30 Vickers Tester). Several tensile coupons were extracted in the horizontal direction (X) of the walls at all conditions. The size of tensile coupons for the ambient and elevated test is shown in Fig.2. The tensile tests at ambient and elevated temperature (650°C) with a crosshead speed of 1 mm/min were conducted by electro-mechanical machine (Instron 5500R). Three tensile coupons of each condition were tested and the average values of the tensile results were calculated.

3 Results

The effect of cold rolling force on average layer height and strain is shown in Table 2. The average layer height decreases from 2.82 mm in the as-deposited sample to 2.59 mm and 2.31 mm with the cold rolling force of 50 kN and 75 kN, respectively, which results in the strain of 8.51% and 18.1%. Based on Eq. (1), it can be observed that the strain increases significantly with an increase in the rolling force, however, the walls collapsed when the rolling force of 100 kN was applied.

$$\varepsilon = (h_{AD} - h_{CR}) / h_{AD} \times 100\% \quad (1)$$

Where h_{AD} is the layer height of WAAM deposition, and h_{CR} is the layer height after the cold rolling processes.

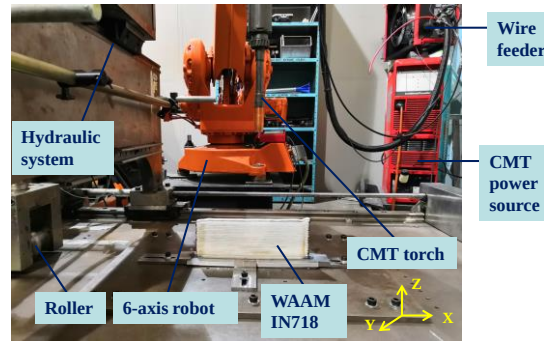


Fig. 1 WAAM deposition and hydraulic cold rolling system

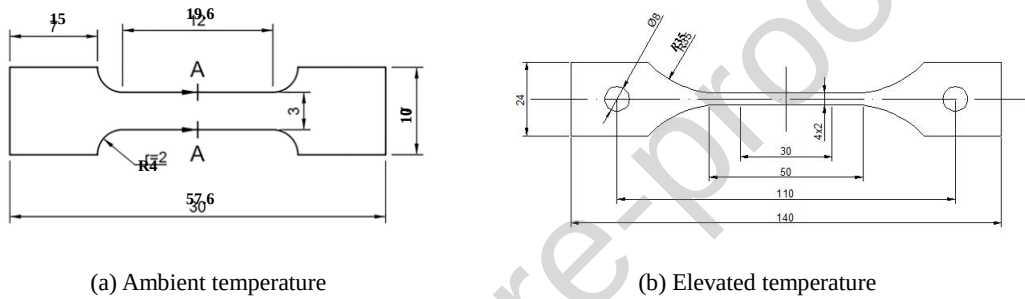


Fig.2 Dimensions of IN718 tensile coupon

Table 2 Size and strain of walls

	Layers	Total height(mm)	Average layer height (mm)	Strain (%)	Wall width (mm)
AD	32	90.24	2.82	/	10.65
CR (50 kN)	36	93.24	2.59	8.51	11.05
CR (75 kN)	40	92.4	2.31	18.1	11.93

AD: as deposited; CR: cold rolled

3.1 Hardness

Fig. 3 shows the effect of cold rolling force on the hardness distribution. Both the as-deposited and cold rolled samples show a homogeneous hardness distribution. The hardness increases from 250.6 HV in the as-deposited condition to 322.9 HV and 335.6 HV in the cold-rolled samples with the rolling force of 50 kN and 75 kN, respectively. The hardness increases significantly for both the as-deposited and cold-rolled samples after heat treatment. The hardness of all the heat-treated samples exceeds that of the wrought material (350 HV) based on Aerospace Material Specification (AMS 5662) [26]. In addition, the hardness fluctuation increases after heat treatment compared to that without heat treatment, in which the upper layer shows a lower hardness.

The cold rolling process increases the dislocation density inside the material, which increases with an increase in the rolling force. Nevertheless, the hardness observed for the applied rolling force of 75 kN is smaller than that of the wrought material. IN718 is a precipitation-hardened alloy and it requires a heat treatment for the precipitation of strengthening phases. The upper layer shows a lower hardness due to its different microstructure without rolling process and its response to the heat treatment.

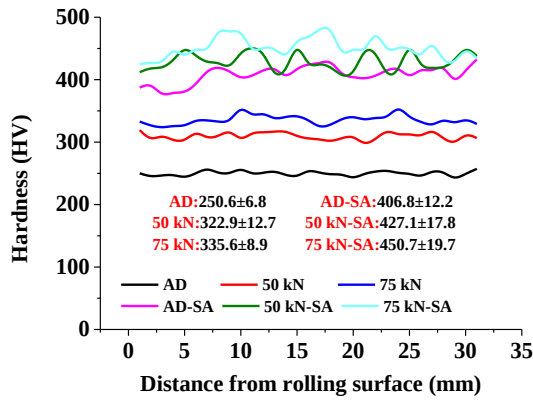


Fig.3 Effect of rolling force on hardness distribution

SA is standard heat treatment

3.2 Tensile properties

Fig. 4 shows the stress-strain curves at ambient and elevated temperatures. The cold-rolling increases the strength and reduces the elongation at both ambient and elevated temperatures before heat treatment. The increase in strength and the corresponding reduction in elongation both increase with increasing rolling force. The strain and dislocation density both increase with ascending rolling force, thus enhancing the yield strength (YS) and ultimate tensile stress (UTS), while decreasing the elongation. The double treatment significantly enhances the strength and reduces the elongation for both the as-deposited and cold-rolled samples at ambient and elevated temperatures. As presented in Table 3 and Table 4, in contrast to the results obtained before the heat treatment, the elongation at 75 kN is larger than that at 50 kN with heat treatment at ambient and elevated temperature.

The stress-strain curves are quite smooth at ambient temperature, while there are serrations at elevated temperature. The elastic modulus and strength both decrease significantly at elevated temperatures. In comparison with the wrought material for the test at ambient temperature, the as-deposited samples show lower strength and larger elongation after heat treatment, while the cold-rolled samples show superior strength and elongation. For the test performed at elevated temperature, the strength of cold-rolled samples exceeds that of the wrought material, while the elongation is lower. The elongation of the sample cold-rolled at 75 kN is quite close to that of the wrought material at elevated temperature.

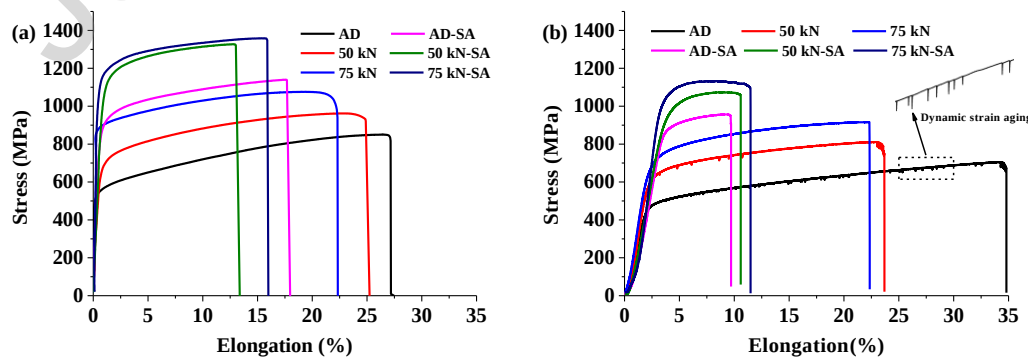


Fig.4 Tensile curves at (a): ambient temperature and (b): elevated temperature**Table 3** Tensile test results of IN718 at ambient temperature

	UTS/MPa	YS/MPa	Elongation (%)
AD	840.7 ± 13.3	472.5 ± 9.5	26.3 ± 1.1
AD-SA	1158 ± 18.2	836.3 ± 18.8	16.44 ± 1.24
50kN	971.7 ± 19	724.3 ± 18.2	24.2 ± 1.8
50kN -SA	1330.4 ± 4.3	1035 ± 9.9	13.75 ± 1.5
75kN	1075.2 ± 15.4	775.2 ± 14.2	22.3 ± 1.2
75kN -SA	1357.3 ± 12.1	1067.4 ± 12.4	15.8 ± 1.7
Wrought-20°C [26]	1276	1034	12

Table 4 Tensile test results of IN718 at elevated temperature of 650 °C

	UTS/MPa	YS/MPa	Elongation (%)
AD	703.3 ± 4.5	489.4 ± 51.9	34.77 ± 0.14
AD-SA	946.6 ± 43.2	865.7 ± 32	7.16 ± 0.05
50kN	811.3 ± 32.7	607.6 ± 45	23.46 ± 1.78
50kN -SA	1050.3 ± 25.9	923.1 ± 20	10.3 ± 0.19
75kN	962.1 ± 53.9	695.5 ± 65.3	19.3 ± 0.47
75kN -SA	1137 ± 18.2	1009.1 ± 3.2	11.48 ± 1.2
Wrought-650°C [26]	1000	862	12

3.3 Fracture morphology

The fracture mode mainly consists of dimple and cleavage fractures. The type of fracture (ductile or brittle) depends on the plastic strain of the material before fracture. In general, the dimple fracture always appears with a large strain, and the strength on the slip plane is larger than that of the grain interior. While there is quite a small strain at cleavage fracture and the grain interior shows a larger strength than the slip plane. The fractured surfaces in as-deposited and cold-rolled conditions with different rolling forces at ambient temperature are shown in Fig. 5. The as-deposited surface shows fine dimples with a dendritic pattern, indicating trans-granular ductile fracture and good ductility before heat treatment. The cold-rolled samples show uniformly distributed equiaxed dimples, while the size of dimples is smaller than that in as-deposited surface, which decreases with increasing rolling force. Some larger and deeper dimples appear at rolling force of 50 kN, showing better ductility compared to the surface at 75 kN, as shown in Fig. 5 (b).

The double aging treatment significantly reduces the elongation as it decreases the number and size of the dimples for both the as-deposited and cold-rolled samples. There are some cracks in the cold-rolled surfaces, which reduces the elongation during the tensile test by crack growth and propagation. The surface with 50 kN rolling force shows brittle characteristics without clear equiaxed dimples, while there are some shallow dimples for 75 kN. As a result, the surface of cold rolled condition with 75 kN shows a higher elongation than that of 50

kN.

Fig.6 shows the fractured surface at elevated temperature of 650 °C. The second particles of IN718 (including Laves phase, MC carbides, etc) provide the positions for the formation of microvoids during the tensile test at elevated temperature, which will be the region for the crack initiation. The movement and coalescence of microvoids may cause a fracture in the form of dimples, indicating microvoid coalescence fracture for the as-deposited and cold-rolled surfaces at elevated temperature. The as-deposited surface consists of equiaxed dimples at elevated temperature, which is quite different from the dendritic pattern at ambient temperature, as shown in Fig. 6(a). Large and deep dimples of the as-deposited sample indicate better ductility. There is a cleavage plane for the cold-rolled surface with shallower and smaller dimples compared to the as-deposited sample, indicating poor ductility of the cold-rolled surfaces. Nevertheless, the cold-rolled samples show ductile fracture due to the high elongation before fracture.

The smaller and shallower dimples after the double aging treatment significantly decrease the elongation, as shown in Fig. 6(c). However, the number and size of dimples both increase for the cold-rolled surfaces after double aging treatment, which is consistent with the higher elongation for the rolled samples at elevated temperatures. Meanwhile, there is no clear crack for the rolled surface, which is different from that in Fig. 5.

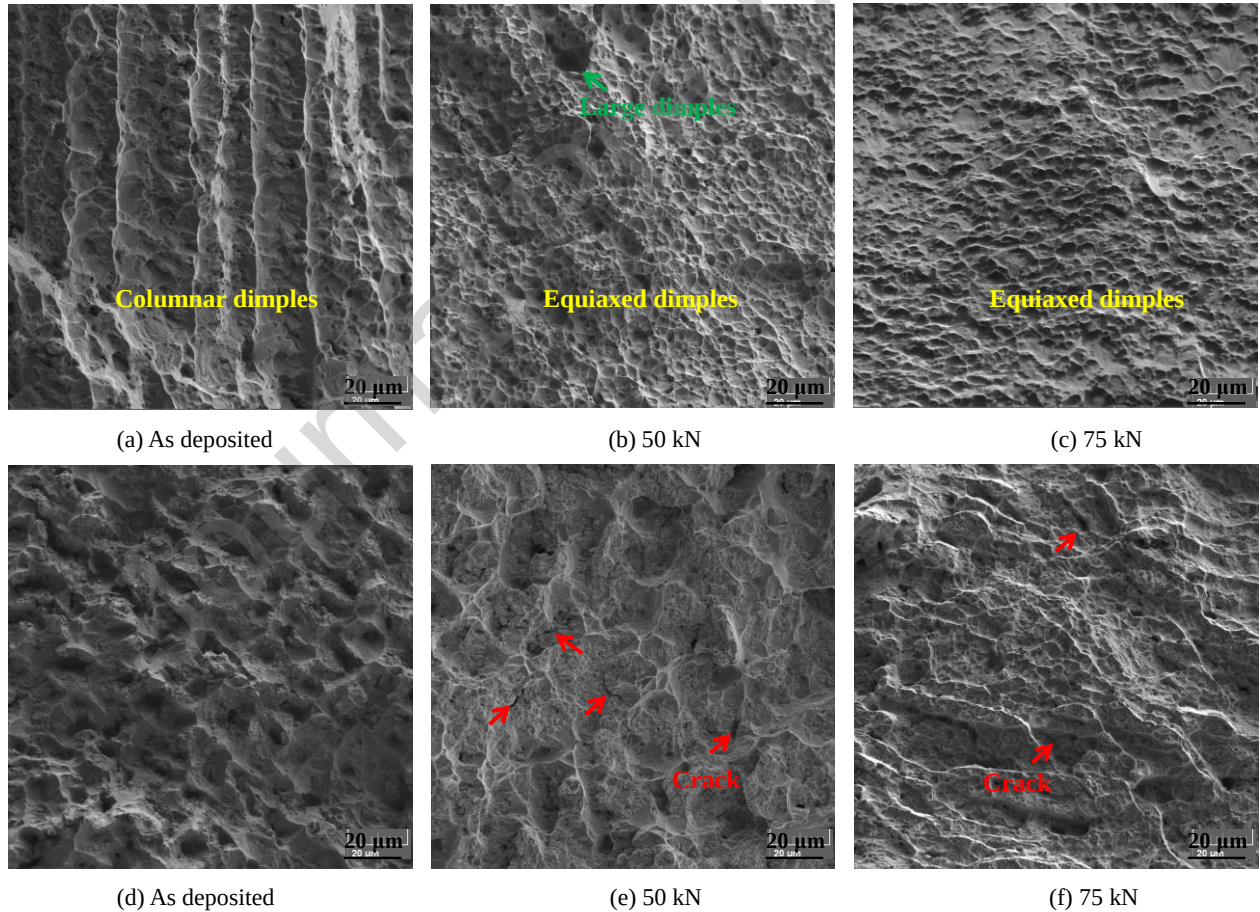


Fig.5 Fracture surfaces of the tensile test at ambient temperature: (a)-(c) before heat treatment; (d)-(e) after solution plus aging treatment

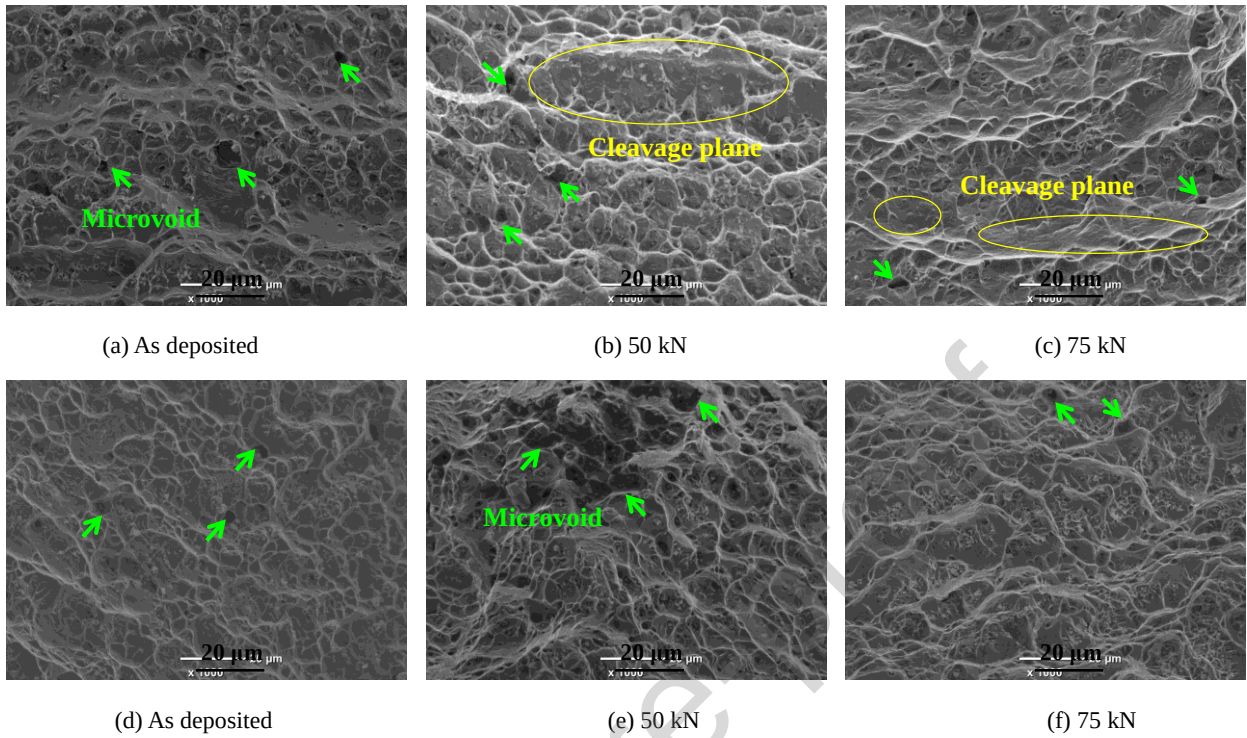


Fig.6 Fracture surfaces of the tensile test at elevated temperature: (a)-(c) before heat treatment; (d)-(e) after solution plus aging treatment

3.4 Microstructure

Fig. 7 shows the microstructure of the as-deposited and cold-rolled samples. The as-deposited samples show extensively grown columnar dendrites with the average dendrite arm spacing of 85 μm . The grain boundaries of the columnar dendrites are plotted with blue arrow in the images with higher magnification times, as shown in Fig.7 (a). Discrete islands of Laves phase (marked with green arrow) are precipitated at inter-dendritic boundaries. The Laves phase is rich in elements of Nb, Mo, and Ti, showing the micro-segregation behavior in the WAAM deposition process. After the cold rolling process, the Laves phase with the shape of discrete islands changes to separate circular Laves phase with small size, indicating that the cold rolling process is beneficial for the fragmentation of Laves phase. Meanwhile, the columnar dendrites in as-deposited condition change to equiaxed grains after cold rolling with sizes 26.5 and 14.7 μm for the force of 50 kN and 75 kN, respectively. The grain boundaries of the equiaxed grains are quite obvious, as marked with blue arrows in Fig.7 (b)-(c). As shown in Fig. 7(c), the microstructure uniformity further increases for the cold rolling force of 75 kN. Meanwhile, the vertical and dendritic distributed Laves phases in the as-deposited sample change to inclined and dendritic distributed for the rolling force of 50 kN, and then change to randomly distributed ones for 75 kN, indicating that the rolling force changes the microstructure and particles distribution at the boundaries.

The columnar dendrites still exist in the as-deposited sample after heat treatment as the solution temperature is lower than the recrystallization temperature of IN718 [27]. The average grain size increases to 29.2 μm and 18.8 μm after heat treatment for 50 kN and 75 kN, respectively, indicating a slight grain growth. The volume fraction of Laves phase decreases for the as-deposited and cold-rolled samples compared to the microstructure without heat treatment. The grain boundaries become fuzzy after heat treatment compared to that without heat treatment. In addition, a small quantity of twin crystals appears at the grain boundaries for both the as-deposited and cold rolled samples; meanwhile, most of the equiaxed grains gradually change to polygonal grains.

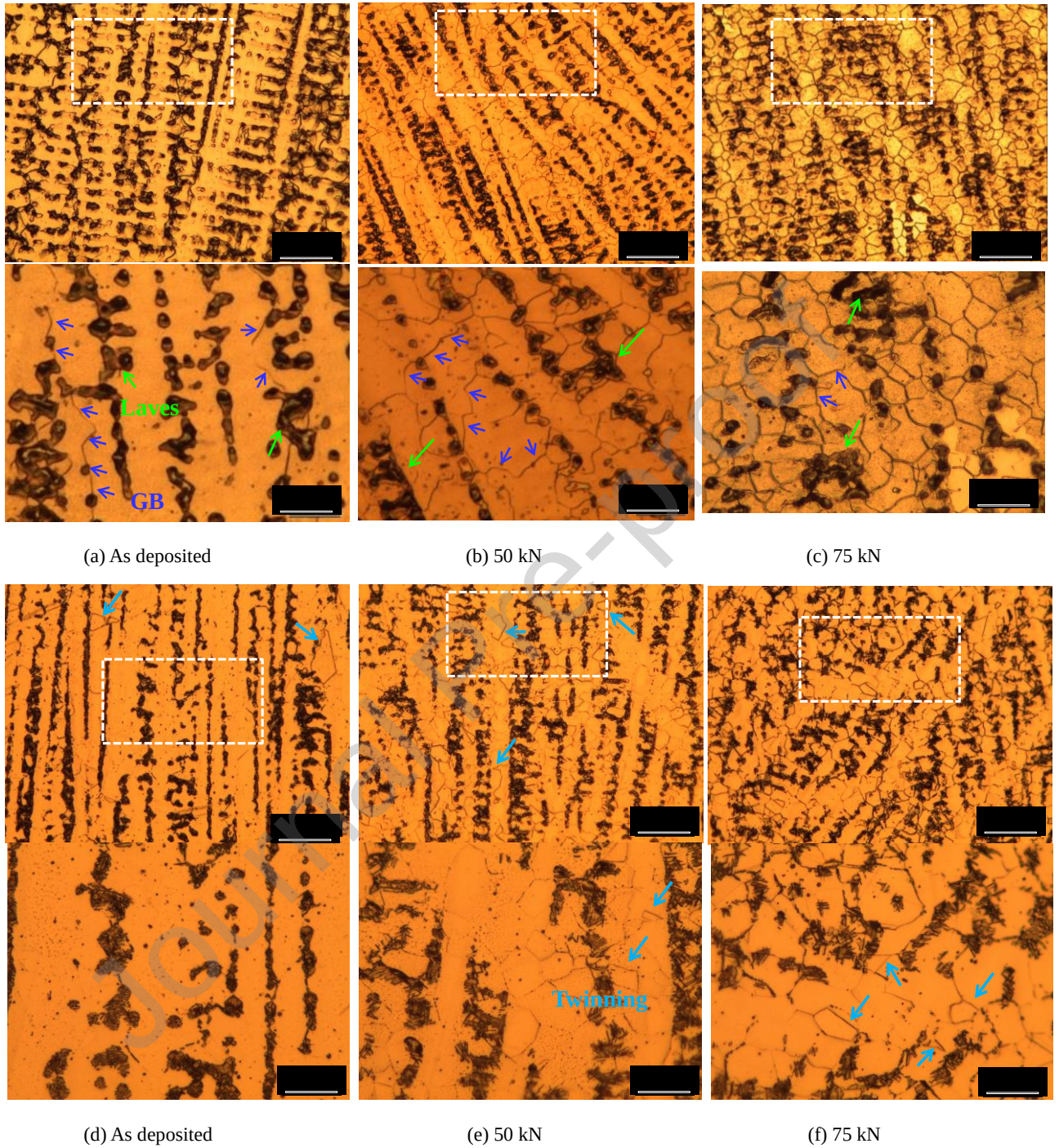


Fig.7 Microstructure of IN718: (a)-(c) without heat treatment; (d)-(f) after heat treatment

3.5 Precipitation

Fig. 8 shows the effect of rolling force on precipitation distribution after heat treatment and Table 5 shows the EDS analysis of the different second phases. Discrete islands of Laves phase with the dendritic pattern for the as-deposited sample and equiaxed pattern for the cold-rolled sample form at inter-dendritic boundaries. The cold rolling process contributes to the columnar dendrites for equiaxed grains transformation (CET). Thus, significantly changing the shape of the Laves phase due to the large rolling force. Meanwhile, large circular MC carbides appear at the as-deposited and cold-rolled samples as the solution temperature is much lower than its

dissolving temperature [18]. The acicular δ phase forms at the inter-dendritic boundaries of the as-deposited sample and grain boundaries of the cold-rolled sample, and some of the δ phases appear around the Laves phase due to the region rich in Nb element. The fraction of δ phase of 75 kN rolled sample is smaller than that of the 50 kN rolled one. As shown in Fig. 8(d), a large number of γ'' phase precipitates at both the grain boundaries (marked with yellow arrow) and the grain interiors (marked with a circle), which is the main strengthening phase of IN718. The dispersive distributed strengthening phases contribute to the strength increase of IN718 after heat treatment.

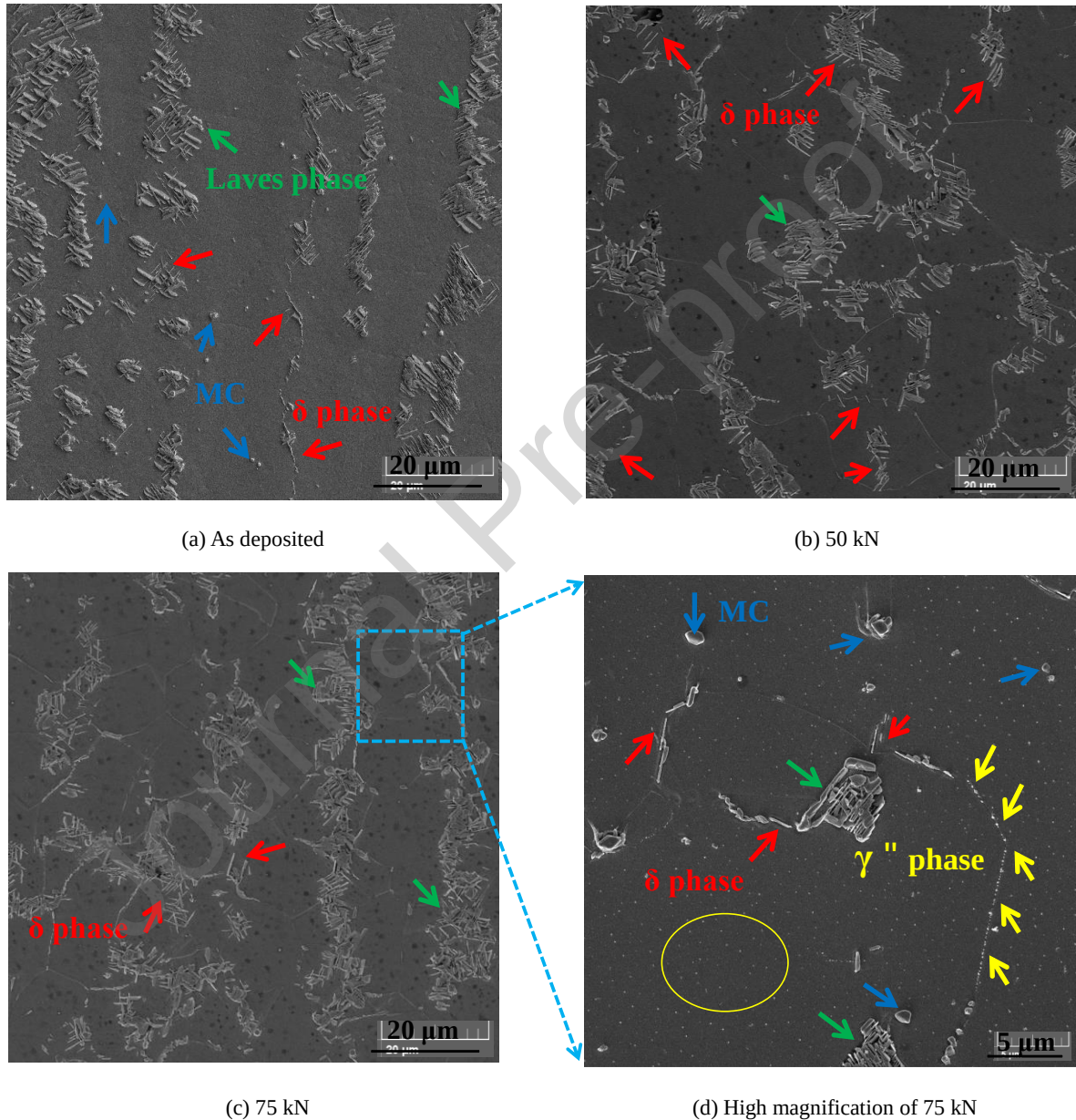


Fig.8 Effect of rolling force on the precipitation phases distribution after heat treatment

Table 5 EDS analysis of precipitated phases

Phases		Ni	Nb	Mo	Cr	Fe	C	N	Al	Si	Ti	Cl
Matrix	wt%	46.63	3.11	2.72	18.1	17.17	9.37	1.53	0.58	0.08	0.71	/
	Atomic%	32.55	1.37	1.16	14.26	12.6	31.98	4.48	0.88	0.11	0.61	/

Laves	wt%	55.57	23.92	2.65	7.77	7.49	/	/	0.33	/	1.95	0.3
	Atomic%	60.02	16.33	1.75	9.48	8.51	/	/	0.78	/	2.59	0.54
δ	wt%	53.17	14.59	/	13.88	12.87	3.91	/	0.47	0.03	1.07	/
	Atomic%	47.01	8.15	/	13.86	11.96	16.89	/	0.9	0.06	1.16	/
γ''	wt%	52.68	8.76	2.4	12.44	12.21	9.81		0.46	0.25	0.99	/
	Atomic%	38.38	4.03	1.07	10.23	9.35	34.95		0.73	0.38	0.88	/
MC	wt%	14.93	37.71	/	6.5	5.36	28.04	/	0.21	/	7.25	/
	Atomic%	7.54	12.03	/	3.7	2.85	69.17	/	0.23	/	4.48	/

4 Discussions

4.1 Grain morphology and recrystallization

The columnar dendrites are the typical microstructure of the additive manufactured IN718. The grain morphology depends on the temperature gradient and cooling speed. Ultrahigh temperature gradient and ultrafast cooling speed can be generated in the molten pool during WAAM deposition. Therefore, a large number of crystal nucleus appear during the solidification process. The growth direction of the crystal nucleus is vertical to the solid-liquid interface of the molten pool and opposite to the direction of the temperature gradient. Meanwhile, the substrate and previously solidified material both contribute to the epitaxial growth of the columnar dendrites.

The recrystallization process plays a vital role in the CET, as shown in Fig.7. The deposited material is subject to non-uniform rapid heating and cooling during the new layer WAAM deposition. The heat input from the new layer deposition will increase the temperature of the previously deposited material, which exceeds its recrystallization temperature. As a result, the deformation energy accumulated in the rolling process will trigger the recrystallization process, thus changing the columnar dendrites to the equiaxed grains through the formation of recrystallized nuclei and growth of recrystallized grains. Meanwhile, the residual stress and increasing dislocation density during the WAAM deposition and rolling process both contribute to the recrystallization process. Although, the heat input from the new layer deposition will melt part of the previously deposited material, and decrease the deformation energy. The melted area is much smaller than the rolled-hardened area. The hardened depth of cold-rolled sample for 50 kN or 75 kN rolling force is over 5 mm from the rolled surface, while the melted depth is smaller than the layer height [25]. The accumulated deformation energy at the heat-affected zone can still provide the driving force for the recrystallization process. In addition, new thermal stress will be generated during the rapid heating and cooling process, which also contributes to recrystallization and grain refinement.

The annealing twins play a vital role in the grain refinement during the static recrystallization process after heat treatment. The migration of grain boundaries and formation of cube texture can promote the generation of annealing twins [28]. The grain boundary bulging followed by twinning behind the bulged areas was the dominant nucleation mechanism of the recrystallization nucleation [29]. High grain boundary mobility promoted the nucleation of the twins by dividing a migrated original grain boundary into an immovable twin, a movable twin, and a low angle grain boundary [30]. The size of secondary particles decreases after heat treatment and the dislocations can bypass these particles by the Orowan looping mechanism, thus promoting the movement of the grain boundaries. High grain boundary mobility will in turn accelerates the nucleation of the twins both at the

grain boundaries and grain interiors, which further refines the grains and enhances the strength of the material [31]. Randle [32] revealed the mechanism of twinning-induced grain boundary engineering of nickel-based alloy, finding that the annealing twins with certain volume fraction can make much contribution to hardening and become the effective barriers to slip, which provided an extra driving force for the bulging of local grain boundary. According to Chen's experimental results [33], the solution treated microstructure of IN718 showed a high percentage of random high angle grain boundary with a great amount of $\Sigma 3$ boundaries and extremely limited quantities of $\Sigma 9$ and $\Sigma 27$ boundaries. The densities (length of annealing twin boundaries/length of all grain boundaries) of $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ boundaries in the solution treated microstructure can be estimated as 51.29%, 2.80%, and 1.54%, respectively. However, the densities of the as-deposited and cold rolled IN718 in this study is quite small, indicating that the twinning strengthening is not the main strengthening mechanism for the as-deposited and cold rolled IN718.

4.2 Mechanical properties at elevated temperature

As shown in Fig. 4, the tensile stress-strain curves show serrations at elevated temperatures, while smooth stress-strain curves are observed at ambient temperature. The serrated plastic flow induced by the dynamic strain aging (DSA) had been observed in nickel-based super-alloy deformed at elevated temperature [34]. The DSA is induced by the interaction between the diffusing solute atoms built up around mobile dislocations and secondary phases (carbides, Nb-rich phases, and Laves phase) within the matrix. The diffusing solute atoms piled up around the dislocations and contributed to the pinning effect on the dislocation movement, thus, increasing the strength. The strength may decrease when the mobile dislocations cut through the secondary phases or bypass the strengthening phases by the Orowan looping mechanism [35]. As a result, the tensile stress-strain curves show serrations due to the repeated pinning effect and the mechanism of cutting or looping. The deformed temperature and strain rate both affect the atomic diffusion behavior of the solute atoms and then change the DSA behavior. In addition, the unique additive manufactured microstructure with dispersed secondary phases and textured grains plays a significant role in the appearance of serrated tensile stress-strain curves [36]. Lin [37] also developed a dislocation-solute atom dynamic constitutive model and analyzed the evolution of moving dislocations and solute atoms, concluding that the moving dislocations were hindered by solute atoms at lower strain rates, while the pinning effects decreased at higher strain rates. Different kinds of serrations appeared at different deformation parameters, and the serration in this study (below the flow stress curves) appeared at high deformation temperatures (500–700 °C) [38].

Compared to the tensile test at ambient temperature, lower strength and higher elongation appear for the tensile test at elevated temperature due to the decreasing strength at the grain boundaries and grain interiors. The dislocation slipping induces the plastic deformation of the material during the tensile test. During the tensile test at ambient temperature, the dislocation may be tangled and piled up at some positions (grain boundaries, etc.), thus hindering the dislocation motion. To activate the dislocation multiplication and motion, a large load needs to be applied to increase the shear stress of the dislocation slipping, thus increasing the strength during the tensile test at ambient temperature. The strength at grain boundaries is higher than that in the grain interiors at ambient temperature. However, the atoms and vacancy inside the material will be thermally activated from the heat input during the tensile test at elevated temperature, and the dislocation motion can be achieved by overcoming the obstacles of vacancy or dislocation pile-up. The strength at grain boundaries is much lower than that in the grain interiors at elevated temperatures [39], which is quite different from that at ambient temperature. As a result, the elongation observed for the test at elevated temperature is much higher than that at ambient temperature. In addition, the strengthening phase is difficult to be precipitated during the elevated tensile test due to the

insufficient time, thus inducing the prominent softening behavior of the material.

4.3 Strengthening mechanism

As discussed above, the strengthening mechanism of IN718 is complex, which mainly consists of strengthening through work hardening, grain boundary strengthening, twinning strengthening, and precipitation strengthening [40]. The cold rolling process increases the dislocation density inside the material [41], which increases with the increase in rolling force. The tangled dislocation and piled-up dislocation cell will impede the dislocation motion, thus, increasing the strength of the material. In addition, the rolling process accelerates the accumulation of deformation energy inside the material and provides the driving force for recrystallization and grain refinement. The strength of the material is inverse to the average grain size based on the Hall-Petch relationship. A larger rolling force results in smaller equiaxed grains, which in turn increases the strength. Meanwhile, the twins also contribute to the grain refinement and increase in strength. However, a certain amount of twins is essential for the enhancement of the strength, as discussed in Section 4.1. Plenty of strengthening phases are precipitated both at the grain boundaries and grain interiors, which significantly increases the strength of IN718.

The hybrid WAAM deposited and cold rolled sample using the rolling force of 75 kN almost reaches or exceeds the wrought material at both the ambient and elevated temperature. The wrought material is subject to several cycles of hot deformation and heating-cooling processes; thus, large grains can be effectively refined and undesired detrimental phases (such as Laves phase, etc.) can be significantly eliminated. The fine equiaxed grains provide a large number of grain boundaries for the precipitation of strengthening phases after the double aging treatment, which significantly increases the strength and improves the isotropic properties. In this study, the inter-layer cold rolling process provides the effect of ‘micro-forging’ to the as-deposited material, and the columnar dendrites transform to uniformly distributed equiaxed grains in the cold-rolled sample with the rolling force of 75 kN.

As shown in Fig. 8, less δ phase is precipitated in the cold-rolled with rolling force of 75 kN rolled in comparison with that of 50 kN. As a result, the 75 kN rolled sample shows superior strength than the 50 kN sample, this variation can be explained as follows: Nb is the most important element at strengthening phase as its atomic fraction is around 6-8% for the δ phase, 4% for the γ'' phase and 10-12% for the Laves phase [12]. The Laves phase cannot be completely dissolved into the matrix as the solution temperature is lower than its dissolving temperature. Therefore, a large amount of δ phase may deplete the fraction of Nb in the matrix, thus decreasing the amount of γ' and γ'' strengthening phases after heat treatment and then decreasing the strength of the material.

Laves phase and MC carbides are hard brittle phases of IN718, which are difficult to move during the tensile test. The cracks induced from the inconsistent deformation will reduce the elongation due to the different speeds between the matrix and brittle phases. Meanwhile, these brittle phases are difficult to be dissolved into the matrix during the solution and aging treatment as the treated temperature is much lower than their dissolving temperatures. The double aging treatment significantly decreases the elongation for both the as-deposited and cold-rolled samples. The precipitated strengthening phases and undissolved brittle phases provide the positions for the formation of the initial crack and its propagation, thus significantly reducing the elongation. The 75 kN rolled sample shows larger elongation than the 50 kN rolled sample after double aging treatment both at ambient and elevated temperatures, which can be explained as follows: first, the δ phase is incoherent with the matrix, and crack will first initiate at these positions due to the inconsistent movement between the dislocation and the δ phase. Less fraction of δ phase for the 75 kN rolled sample is beneficial for higher elongation. Secondly, the formation of the δ phase will deplete the strengthening γ'' phase [18], thus

decreasing the strength of the material. In addition, a moderate fraction of δ phase plays a positive role in eliminating notch sensitivity and stress concentration [42]. The excessive δ phase is more likely to promote crack initiation and propagation [43-44]. Thirdly, the morphology of the δ phase is also related to the ductility, and the short rod-like δ phase with dispersed distribution contributes to higher elongation [16]. Large rolling force is more likely to decrease the size of the δ phase and promote the formation of dispersed δ phase.

5 Conclusions

The cold rolling process was integrated into the WAAM deposition and the effect of rolling force on the microstructure and tensile properties at ambient and elevated temperatures were analyzed. The results are summarized as below:

1. The columnar dendrites of the as-deposited sample changed to finer equiaxed grains and its size decreased with the increase in the rolling force. The columnar dendrites with an average dendrite arm spacing of 85 μm are refined into equiaxed grains with size of 26.5 and 14.7 μm using the cold rolling force with 50 kN and 75 kN, respectively.
2. The cold-rolled samples show superior tensile properties than the wrought material at ambient temperature. The increase in strength and reduction in elongation both increase with the increase in rolling force. The less δ phase obtained for 75 kN rolled sample contributes to the precipitation of more strengthening phases and the consistent movement between the dislocation and matrix, thus increasing the strength and elongation.
3. There are serrations for the stress-strain curves at elevated temperature due to the dynamic strain aging behavior. The 75 kN rolled sample shows equal tensile properties at elevated temperature to the wrought material. The YS and UTS increase remarkably from 862 and 1000 MPa in the wrought material to ~1009 and ~1137 MPa for the 75 kN rolled, accompanied by a slight decreased elongation from 12% to ~11.5 %.
4. The strengthening mechanism of hybrid WAAM deposition and cold rolling process mainly consists of strengthening through work hardening, grain boundary strengthening and precipitation strengthening.
5. The hybrid WAAM deposition and cold rolling process was proved to be potential for the manufacturing of large metal components with equal properties to the wrought components. How to further increase the elongation of the hybrid manufactured components without decreasing its strength needs to be further studied.

CRedit authorship contribution statement

Tao Zhang: Conceptualization, Writing – original draft. **Huigui Li:** Experiment and analysis. **Hai Gong:** Supervision, Writing – review & editing. **Abdulrahman Shuaibu Ahmad:** Proof reading and revision. **Yunxin Wu:** Methodology, Writing –review & editing. **Xin Chen:** Investigation, Resources.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Highlights:

1. Cold rolling process is integrated into the WAAM to refine the grains.
2. Effect of rolling force on tensile properties at elevated temperature is analyzed.
3. Large rolling force results in finer grains and uniform strengthening phases.
4. 75 kN rolled sample shows equal properties to wrought material at 650°C.
5. The grain morphology, recrystallization and strengthening mechanism are revealed.



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Effect of rolling force on tensile properties of additively manufactured Inconel 718 at ambient and elevated temperatures

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