

Heat treatment behaviour of IN718 superalloy fabricated by laser-powder bed fusion

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ABSTRACT

IN718 Ni-superalloy samples were produced by Laser Powder Bed Fusion (LPBF). The phase transformations that occur during different heat treatments, the microstructure evolution and static mechanical properties were investigated. Two-steps artificial ageing (AA), solution annealing followed by artificial ageing (SA + AA) and homogenization annealing followed by solution annealing and artificial ageing (HA + SA + AA) were performed and the relevant phase transformations were investigated through dilatometric analysis. The precipitation temperature of γ' (525 °C), γ'' (725 °C) and δ (935 °C) were determined. The activation energy of γ' precipitation was estimated by means of Kissinger-like method to be about 248 ± 21 kJ/mol. It was demonstrated that the strengthening effect after AA is attributed to γ'' precipitation. Besides, SA and HA are responsible of the chemical homogenization and anisotropy removal. SA itself is not able to completely recover the solidification structure, so HA is necessary for a full recrystallization.

1. Introduction

Additive Manufacturing offers a wide number of production techniques [1,2]. Among these, LPBF provides outstanding results [3]. This technique is based on the selective laser fusion of already deposited powder bed regions. High resolution and complex parts can be obtained at the expense of part size, which is limited compared to other additive manufacturing process. LPBF is particularly suitable for demanding applications as aerospace or automotive ones, where materials such as titanium alloys, nanostructured alloys and nickel-based alloys are of growing interest [4–13]. Nickel-based superalloys are increasingly used thanks to their excellent mechanical properties at both low and high temperatures, especially after proper solution annealing and double ageing treatments [14,15]. They show also good corrosion and oxidation resistance thanks to the alloying by Cr and Al, which form very stable and protective oxide layers. Good fatigue behaviour and creep resistance are also provided [16–27]. Ni Superalloys are essentially based on the Ni-Cr-Fe ternary system and develop outstanding mechanical properties thanks to the precipitation of coherent intermetallic compounds and their strain hardening response. Intermetallic compounds and carbides (both MC and M₂₃C₆) can precipitate during the solidification, but the highest strengthening is provided by those precipitating during heat

treatment [27]. Among the nickel-based superalloy the Inconel 718 (IN718) superalloy is one of the most studied and used [5,6,17–48]. In the last years, the high dimension precision required by the aerospace and automotive components suggests a shift from traditional cast and wrought product to novel non-traditional processing technology as additive manufacturing.

Nie et al. studied in detail the microstructure evolution of IN718 during laser additive manufacturing [31]. Higher cooling rate and smaller equiaxed dendrite arm spacing give rise to the formation of the eutectic microconstituents in Nb and Mo rich interdendritic areas, such as Laves phases and NbC carbide [32]. One of the most diffused techniques for additive manufacturing Inconel 718 is the selective laser melting [33,34]. Tao et al. studied the solidification microstructure of selective laser melted IN718 focusing on both macroscopic and microscopic perspectives, highlighting the role of rapid solidification on intercolumnar segregation and intercellular spacing [35]. J.H. Yi et al. proved how the porosity is inversely proportional to the laser energy density, therefore a higher source power is suitable for obtaining higher density and so mechanical properties [36]. Ferrar et al. studied the influence of the protective gas flow on density and compression strength, highlighting its important role on the removal of detrimental by-products formed around the fusion region [37]. Y. Tian et al.

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schematized the microstructural evolution of IN718 made by the direct laser additive manufacturing process [38]. In details, they highlighted that the interdendritic eutectic products formed after the first laser scan, namely MC-type carbides and a Nb,Mo-rich Laves, were remelted after the second laser layer and the realised Nb diffuses into dendrite core promoting the precipitation of γ'' nearby the eutectic products. An important feature of additive manufacturing is microstructural anisotropy leading to anisotropy in mechanical properties [39]. The as-built solidification structure is similar to a welded component, with easily detectable melting pools. The use of a highly focused laser beam introduces large thermal gradients, while the rapid solidification of thin layers results in columnar growth directed towards the thermal centre of the laser spot and formation of strongly segregated interdendritic zone. All this introduces undesired thermal stresses that must be removed or at least reduced before cutting the supports from the baseplate, so to avoid distortions [40–42]. Also factors as scanning speed and hatch distance must be considered. As well powder characteristics are paramount. Particle size distribution plays an important role in final product quality and fatigue behaviour, dealing with surface finishing and density [41–43]. Indeed, presence of Laves phase and/or lath δ phase leads to poor performance of the material [44]. Heat treatments are therefore necessary to recover residual stresses and also to tune the desired microstructure and mechanical properties. Standard heat treatments of IN718 include homogenization annealing (HA), solution annealing (SA) and double artificial ageing (AA) [45,46]. In details, SA (980 °C, 1 h/air cooling) and AA (720 °C, 8 h/furnace cooling at 55 °C/ 620 °C, 8 h/air cooling) is the industrial standard heat treatment for wrought IN718. Differently, HA (1080 °C, 1.5 h/air cooling) follows by SA and AA is the one used in case of casting IN718 [42]. As reported in previous works, HA at a temperature between 1050 and 1150 °C is performed to recover interdendritic Nb microsegregation which can lead to undesirable phases, including NbC and the Laves phases [47,48]. Solution annealing (SA), carried out at lower temperature (925–1010 °C) cannot redissolve the non-equilibrium phases that may precipitate during rapid solidification hindering the homogenization of the chemical composition [42]. Typically, it is carried out to obtain the supersaturated solid solution needed for precipitation hardening. The final double ageing (AA) at about 720 °C and 620 °C promotes the precipitation of the strengthening phases, namely fcc- γ' ($\text{Ni}_3(\text{Al,Ti,Nb})$) and bct- γ'' (Ni_3Nb), which is instead suppressed during the additive manufacturing process [49–58]. Y. Tian et al. analysed the microstructural evolution after standard solution heat treatment (SA up to 980 °C for 2 h followed by AA) of a IN718 obtained by pulsed laser powder bed fusion highlighting the dissolution of the separated melting pool boundaries, a partial recrystallization with the formation of small equiaxed grains and presence of needle-like δ phase and nanoscale precipitates [32]. Only the addition of HA before SA and AA permits to obtain a complete recrystallization and a uniform element distribution [59]. TTT curves of IN718 reported similar temperature range of γ' and γ'' precipitations [11,45,51–62,66] even if very few authors reported that γ' precipitates occurs at around 500 °C and γ'' at around 700 °C [67,69]. Usually γ' precipitates present a cuboidal or spherical shape of dimension around 50 nm; differently from γ'' that looks like lenticular, disc shaped particle with a length range from 50 to 400 nm and a thickness of about 30 nm [38,70–72]. Only very few author distinguish the real contribute of γ' and γ'' on the strengthening effect [68,70,72]. Mignanelli et al. [68] found that only γ' precipitates in Ni-15Cr-4Al-6Nb superalloy leads to a hardness of around 300 HV; differently, a regular network of acicular γ'' precipitates bringing about to a peak strength of 500 HV. The precipitation of strengthening phases seems to be dominated by the diffusion of solute atoms, i.e. Al, Ti and Nb. In details, the activation energy of diffusion-controlled growth of γ'' is around 272 KJ/mol equal to the one of Nb diffusion into Ni [73–76]. Very few authors [77] evaluated the activation energy of γ' precipitation around 245 KJ/mol nearly similar to those of diffusion of Al into Ni alloy [78]. In literature, different HA temperatures are proposed for additive manufacturing parts ranging however from 1050 °C to 1150 °C

[42,46,51–54]. Zhou et al. showed that a temperature of 1065 °C is enough to completely remove microsegregation [49], while Deng et al. suggest a HA temperature of 1080 °C [52]. These temperatures are slightly beyond the δ phase (orthorhombic Ni_3Nb) solvus temperature that is 1020 °C. This allows its dissolution and the Nb redistribution into the matrix [5]. Subsequent solution treatment (SA) can precipitate δ phase at the grain boundaries, reducing grain growth at high temperatures [60]. The precipitation hardening process results in improved mechanical strength compared to the as-built alloy, at the expense of elongation, which is dramatically decreased [45]. This is clear especially comparing the selected laser melted mechanical properties with the traditional cast and forged ones. AMS specification defines yield strength values of around 750 MPa for casted products [64] and around 1000 MPa for wrought ones [79]. With AM instead, it is possible to reach yield strength up to 1400 MPa [43,45,52], proving the potentiality of this technology applied to this material.

The aim of this work was to highlight the precipitation sequence during HA, SA and AA thermal treatments on IN718 obtained by LPBF by thermal analysis techniques as dilatometric and Differential Scanning Calorimetry (DSC). In details, a distinguish of the precipitation temperature range of γ' and γ'' was detected and the different precipitation temperatures were evaluated. Since very few data, evaluation of activation energy of γ' precipitation was also performed. Metallographic characterization and XRD analysis were performed to supported thermal analysis results. At the end, mechanical properties after different treated conditions were evaluated to highlight the effect on the different precipitation phases on the final performances of the material.

2. Materials and methods

IN718 gas atomized powders with an average particle size ranging from 10 to 45 μm were used to produce standard dilatometry (10 mm $\times$ $\varnothing$ 4 mm) and tensile test samples by LPBF. All the samples were built with a SISMA MySint 100 machine using an alternate scan strategy.

Main features and processing parameters are summarized in Table 1.

The particle size distribution was measured by means of quantitative optical image analysis on a polished metallographic cross section of powder sample. Ten different micrographs at 100 $\times$ were analysed using the ImageJ software. Dilatometric tests were carried out using a Bahr DIL 805 dilatometer. Heating was conducted under dynamic vacuum conditions (5×10^{-4} mbar) and cooling was performed under argon flow. The different heat treatments studied in this work are summarized in Table 2.

Isochronal annealing tests up to 1100 °C were carried out to highlight the phase transformations and any possible precipitation events in the as-built alloy. The dilatometric strain (Δ) was differentiated with respect to time ($d\Delta/dt$) to magnify the contribution associated to phase transformations [79]. The phase transformation that occurs at low temperature was studied by differential scanning calorimetry (DSC) using Perkin Elmer DSC7 under argon atmosphere to avoid oxidation. All the DSC experiments were carried out with empty reference pan. The mass of the specimen was around 60 mg. Samples were heated from room temperature (RT) to 720 °C with different heating rates of 5 (0.08), 10 (0.17), 20 (0.33), 40 (0.67) °C/min (K/s) and cooled down to RT with a cooling rate of 40 °C/min (0.67 K/s). The baselines were obtained from

Table 1
Main features and processing parameters.

Parameters	Value
Operating laser power	200 W
Scanning speed	900 mm/s
Hatch spacing	55 μm
Layer thickness	40 μm
Rotation between consecutive layers	90°
Atmosphere	Ar ($\text{O}_2 < 0.3 \text{ vol}\%$)

Table 2
Schematization of the different thermal treatments.

Sample	Homogenization annealing (HA)	Solution annealing (SA)	Artificial ageing (aa)
	10 °C/min (0.17 K/s) - 1100 °C 1100 °C x 1 h 35 °C/min (0.58 K/s) - RT	10 °C/min (0.17 K/s) - 980 °C 980 °C x 1 h 35 °C/min (0.58 K/s) - RT	10 °C/min (0.17 K/s) - 720 °C 720 °C x 8 h 0.9 °C/min (0.02 K/s) - 620 °C 620 °C - 8 h 5 °C/min (0.08 K/s) - RT
AA	–	–	✓
SA + AA	–	✓	✓
HA + SA	✓	✓	✓
+ AA	✓	✓	✓

a second scan using equal experimental conditions, supposing that all transformations are completed and irreversible after the first run. The activation energy has been obtained by linear interpolation of experimental points on the basis of the Kissinger-like methods [67,80–82] (Eq. (1)).

$$\ln \left(\frac{T_f^2}{\varphi} \right) = \frac{E}{RT_f} + \text{constant} \quad (1)$$

where R is the gas constant, T_f is the transformation temperature in K corresponding to a certain fraction (f) of phase transformation and φ is the heating rate in K/s. The peak maximum has been chosen as T_f . An analogue procedure has been employed also using the dilatometric test in order to compare the activation energies obtained. A complete microstructural characterization was carried out after proper metallographic preparation. Kalling's II (2 g of CuCl_2 , 40 ml of HCl and 60 ml of ethanol 95%) [83]. The microstructure was characterized by Light Optical- (LOM) and Scanning Electron Microscopy (SEM). A JEOL IT 300 SEM apparatus equipped with EDX Bruker XFlash detector 630 M with an active detector area of 30 mm² was used to characterized the micrometric precipitates presence in the as-built and thermal treated samples. Thermodynamic simulations were carried out using ThermoCalc software [84] (2022a, TCNI8 database). To highlight the nanometric phases present inside the as built and thermal treated samples not detectable through JEOL IT 300 SEM, a field emission scanning electron microscopy (FE-SEM) equipped with symmetry EBSD detector was used.

Density measurements were carried out according to ASTM B 962–13 following the Archimedes' principle. Residual porosity was calculated considering the theoretical density of 8.22 g/cm³. The phase constitution of heat treated alloy was determined by X-Ray Diffraction (XRD) analysis using an Italstructures (IPD3000/CPS120) instrument equipped with Co K α source of 2000 W ($\lambda = 0.17889$ nm). The tilting angle of the specimen was set as $\Omega = 6^\circ$. The diffraction spectra were elaborated by the Rietveld method by MAUD software (Materials Analysis Using Diffraction) [85]. Vickers microhardness with a load of 500 gf (HV0.5) on metallographic cross sections of the samples was measured. Tensile tests with a strain rate of 1 mm/ were carried out at room temperature (25 °C, 60% R.H.), and by means of Instron® 8516 (Instron, Nordwood, MA, USA), equipped with hydraulic grips, a load cell of 100kN (non-linearity $\pm 0.1\%$ of R.O.) and an axial extensometer (12.5 mm gage length, nonlinearity $\pm 0.15\%$ of R.O.) 12 Dogbone samples were printed parallel to the building direction and with the geometry shown in Fig. 1 (ASTM E8M-21). 3 samples for each conditions were used to evaluate the tensile mechanical properties.

The heat treatments indicated in Table 2 were carried out using a TAV Minijet industrial furnace under a partial pressure of 10^{-4} bar.

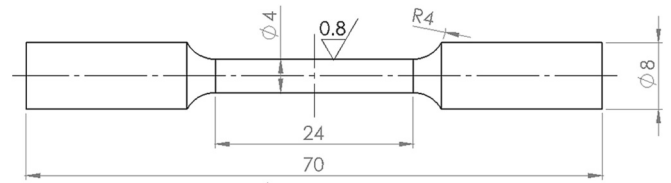


Fig. 1. Dogbone specimen used in tensile test.

3. Results

3.1. Powder and AB material

EDXS analyses highlight very similar chemical composition for both IN718 powder and AB sample, (Table 3).

Fig. 2 displays the SEM micrographs of the powder. Particles are spherical and show a limited number of satellites smaller than 5 μm (Fig. 2a).

The nominal and cumulative particle size distribution determine a mean particle size of 20.5 μm (Fig. 3), while the circularity factor ($4\pi \times \text{area}/\text{perimeter}^2$) determined by image analysis was 0.78 ± 0.12 .

The powder alloy microstructure evidences a dendritic structure as a result of the rapid solidification during LPBF (Fig. 2b). The light grey interdendritic regions in BED micrograph refer to higher amount of heavy elements, as indicated by EDXS analysis (Table 4).

Dendritic area was characterized by higher amount of Al, Fe and Cr, while interdendritic zones showed increased Nb and Mo content due to the preferential partitioning of these elements to the liquid phase during solidification. This microsegregation was also confirmed by considering the full frame analysis of the powder that was characterized by a average composition respect to the primary crystal and the interdendritic areas.

Considering the AB condition, the epitaxial growth of crystals along the building direction overcame the boundaries of melting pools (Fig. 4a). Differently, the building technique and scanning direction ($0-90^\circ$) were underlined in transversal direction (Fig. 4b). The melt pool width was around 100 μm and the average layer thickness was observed to be around 45 μm .

Chemical composition of the Laves phase (spot 1), primary crystal (spot 2) and interdendritic regions (spot 3) are shown in Table 5.

Microsegregation phenomenon is observed by comparing the chemical composition of primary crystal (matrix) (spot 2) and interdendritic areas (spot 3). Higher amounts of Nb and Mo were detected in the interdendritic areas, differently from the primary crystals that are richer in Ni, Cr and Fe. Chainlike Laves phase precipitates (white areas, spot 1) were observed in AB sample (Fig. 4c) in agreement with the higher concentration of Nb and Mo in these regions (Table 5) [47,86].

XRD spectra of the AB sample have been collected in two directions, parallel and perpendicular to the building direction in order to highlight the texture introduced by LPBF (Fig. 5a).

An fcc- γ nickel matrix (cell parameter $a = 0.358$ nm) was detected for both, powder and AB samples. Furthermore, the shoulders of (220) and (331) γ peaks evidence the presence of a small fraction of Laves phase (3% in the powder, 6% in AB). No other phases were detected even if some nitrides (TiN) are clearly visible in LOM and SEM micrographs (orange particles in Fig. 3b). It is plausible that the amount of this phase is lower than the detection limit of XRD (2 vol%). In case of powder, a prominent (111) texture of γ was observed. Differently, AB

Table 3
EDXS chemical composition of powder and AB sample.

EDXS analyses (wt%)							
	Ni	Cr	Fe	Nb	Mo	Ti	Al
Powder	Bal.	19.6	18.3	5.5	1.4	1.0	0.3
AB	Bal.	19.0	18.0	5.5	2.0	1.2	0.3

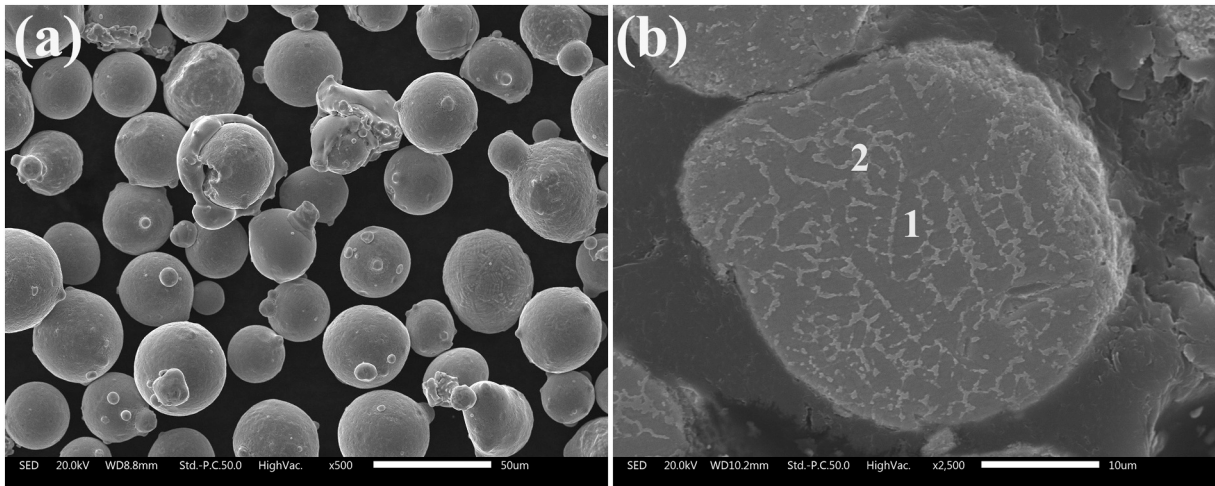


Fig. 2. SEM micrographs of powder.

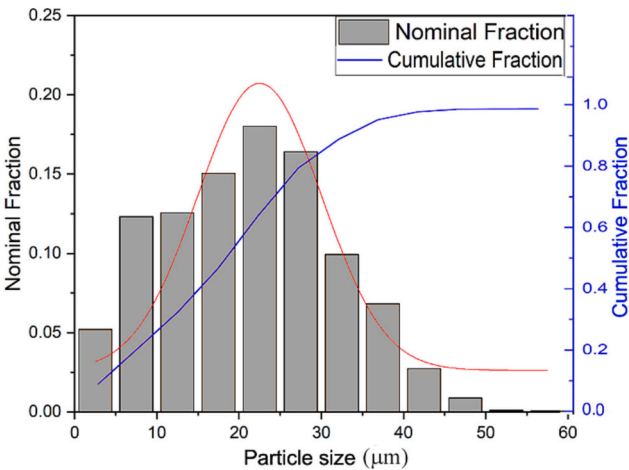


Fig. 3. Cumulative and Nominal fraction of particle size.

Table 4
EDXS analyses in primary crystals (1), interdendritic region (2) and full frame of the powder.

EDXS analyses (wt%)							
	Ni	Cr	Fe	Nb	Mo	Ti	Al
1	Bal.	20.0	19.2	5.1	1.1	0.8	0.5
2	Bal.	18.3	17.2	9.8	1.7	1.3	0.4
Powder	Bal.	19.6	18.3	5.5	1.4	1.0	0.3

samples shows mixed (111) and (200) texture peaks depending on the direction respect to the building one. The stronger (200) texture visible in the longitudinal sample is due to the preferential $\langle 111 \rangle$ growth direction of γ -fcc grains, parallel to the build one. The out-of-equilibrium solidification during L-PBF could be modelled by means of the Scheil module of Thermo-Calc [87–89] (Fig. 5b). The sequential formation of MC, Laves phase and d phase from the heavily enriched liquid at the end of the solidification was predicted. MC and d phases were not detected by XRD plausibly due to a volume concentration lower than the detectable limit of XRD analysis (2%vol).

The relatively fine dimension of powders allowed to get compact products by LPBF [40]. The density ($8.21 \pm 0.02 \text{ g/cm}^3$ with a relative density of 99.9%) and porosity of the as-built alloy evidence the choice of suitable LPBF parameters.

3.2. Heat treated alloy

Fig. 6 shows LOM microstructure of heat treated samples in the longitudinal and transversal directions.

Table 5
EDXS analyses in Laves phase (spot 1), primary crystals (spot 2) and interdendritic areas (spot 3) of the AB sample.

EDXS analyses (wt%)							
Spot	Ni	Cr	Fe	Nb	Mo	Ti	Al
1	Bal.	15.4	12.7	16.5	9.6	2.0	0
2	Bal.	20.6	18.2	4.2	3.3	0.8	0.7
3	Bal.	18.9	14.8	12.0	4.2	1.5	0.6

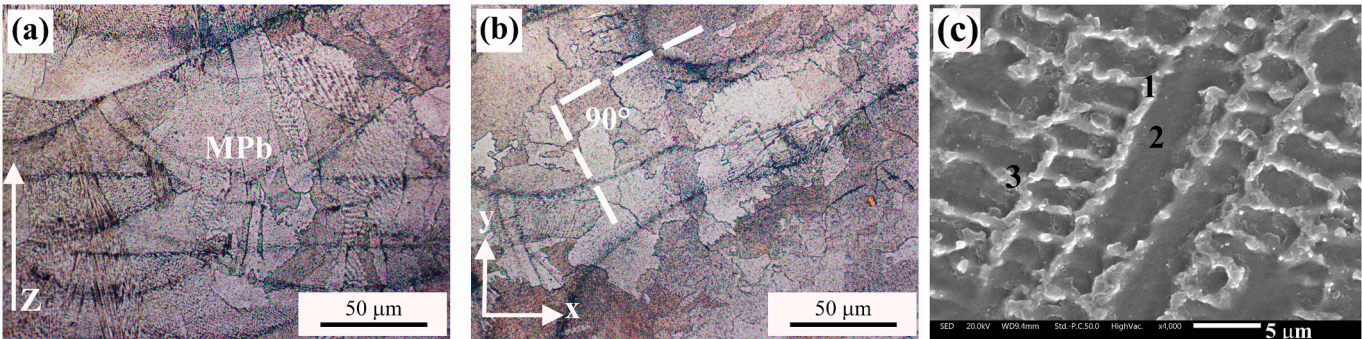


Fig. 4. Microstructure of AB sample a) parallel and b) perpendicular to the building direction (z). c) SEM micrograph showing microsegregation.

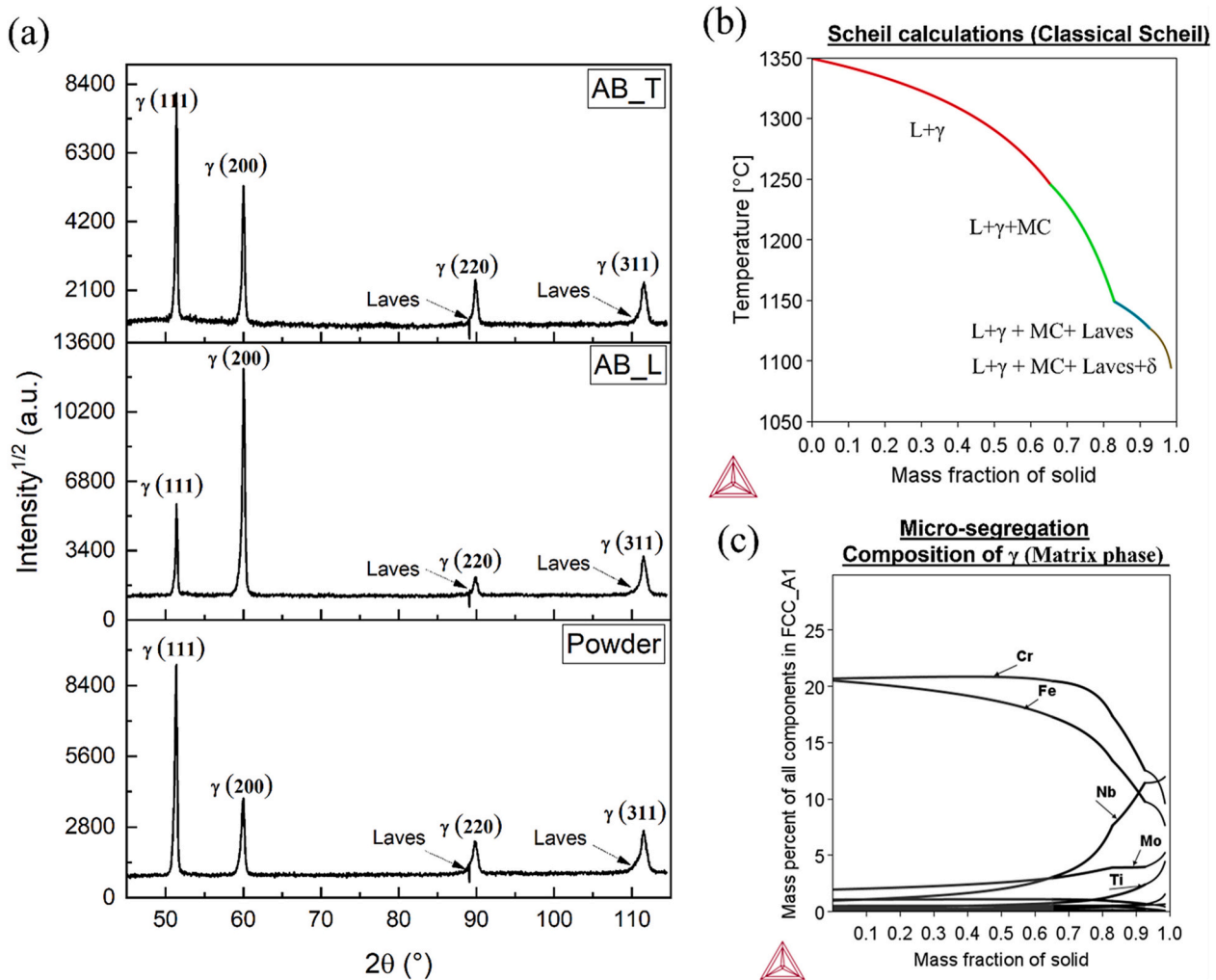


Fig. 5. a) XRD patterns for the base powder and AB samples along L and T directions, b) Scheil solidification simulation by means of Thermo-Calc of Inconel 718 with chemical composition detected with EDXS (Table 3), c) evolution of the chemical composition of the matrix during the solidification process.

The directly aged sample AA (Fig. 6 a,b), retains most of the features evidenced by the AB one, with melt pool boundaries and epitaxial growth of gamma crystals still well evident. As expected, the traces of prior solidification structure cannot be recovered by ageing, due to the too low heat treatment temperature.

Considering the SA + AA sample (Fig. 6 e,f), the melting pool boundaries are no longer evident thanks to the recrystallization taking place during solution annealing at high temperature. A residual preferential orientation of grains along the building direction and some traces of the laser spot paths at 90° in the transversal one confirms that solution annealing provides only a partial recrystallization.

On the other end, after HA + SA + AA heat treatment (Fig. 6g,h), the melting pool boundaries were no longer visible thanks to the higher temperature of homogenizing. The prolonged heat cycles caused the full recrystallization, which were however still partially oriented along the building direction.

These observations were confirmed by SEM analysis (Fig. 7), where various precipitates were observed after the different thermal treatments.

XRD spectra of heat treated samples in the parallel direction respect to the building one (z) were evaluated to define the different phases observed with SEM analysis (Fig. 8). Also, quantitative analysis through Maud software was performed (Table 6).

Peaks of fcc- γ^I ($\text{Ni}_3(\text{Al,Ti,Nb})$) and bct- γ^{II} (Ni_3Nb) were observed in all samples. Some peaks of these strengthening phases were observed

only in AA sample since it was still influenced by the preferential orientation of grains due to the rapid cooling that occurs in the production process. For this reason, γ^I and γ^{II} that precipitate after AA in AB sample take the preferential orientation of γ matrix. Differently, orthorhombic- δ phase are visible only in HA + SA + AA highlighting the complete dissolution of Laves phases during HA and the release of Nb promoting the precipitation of more δ particles. Laves phase in AA and SA + AA and δ phase in SA + AA were not detected, even if present in the SEM micrographs shown in Fig. 7, probably since concentration lower than 2 vol%. As shown in Fig. 6c, Laves phase gradually dissolve or fracture and turn into particles after SA. It does not disappear completely since higher temperature and/or time are necessary to completely dissolve into matrix. As results, after SA + AA treatment there were still residual Laves phase particles and also small amount of δ phase was observed. This is attributed to the solution treatment that is in the temperature range of δ precipitation. Nevertheless, since presence of residual Laves phase that traps some Nb and solution annealing temperature higher than the temperature of maximum kinetic of δ precipitation (954 °C [67]), a small amount of δ phase was present respect to what it is expected to be after SA at 980 °C for 1 h. Homogenization (HA) before SA + AA leads to the complete dissolution of Laves phase, the partial redistribution of alloying elements and full recrystallization of the matrix. In this condition all Nb is released and can promote the precipitation of δ during solution annealing (SA).

Considering the precipitation of strengthening phases, namely γ^I and

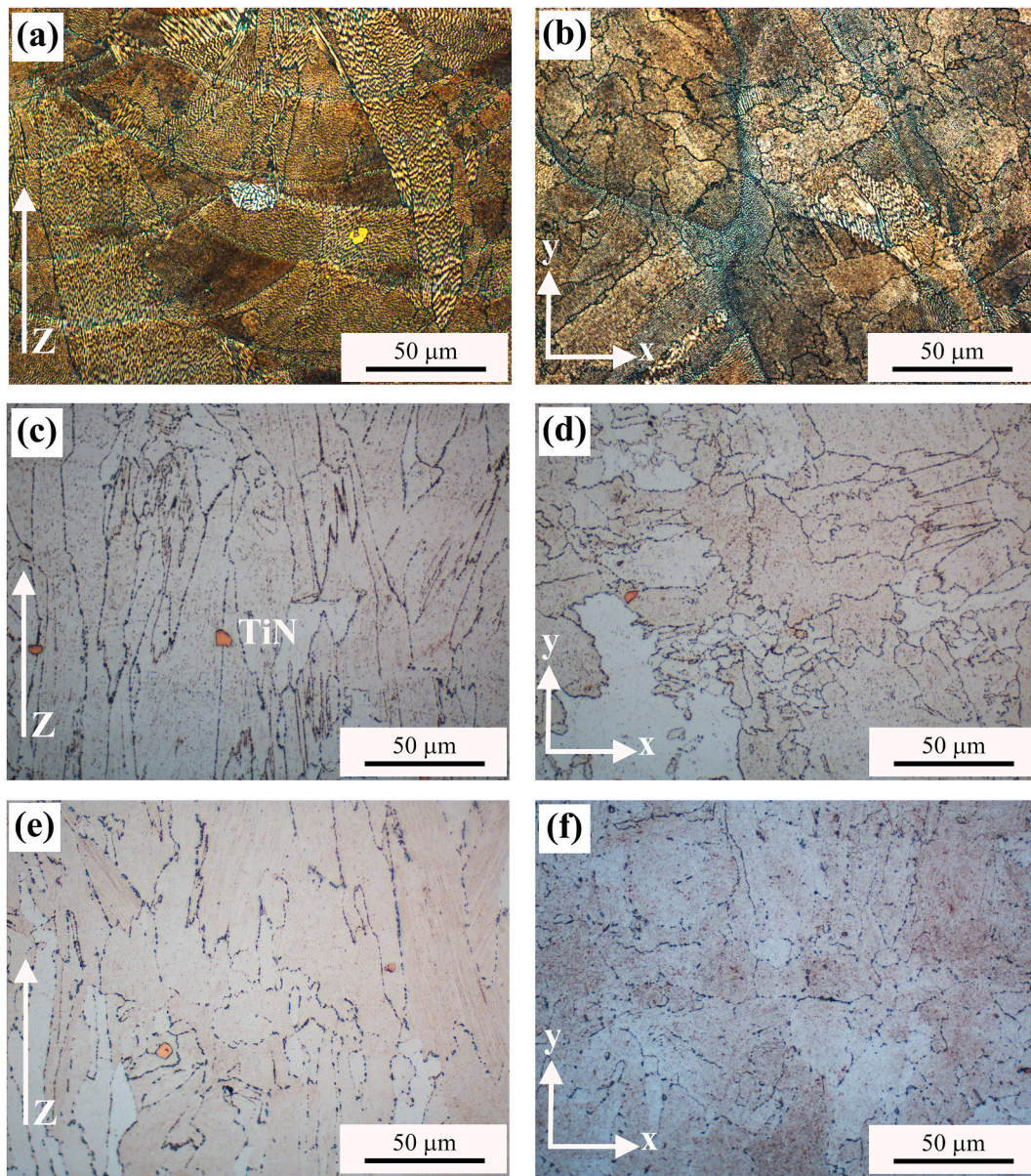


Fig. 6. LOM micrographs of (a,b) AA, (c,d) SA + AA, (e,f) HA + SA + AA parallel and perpendicular to the building direction (z).

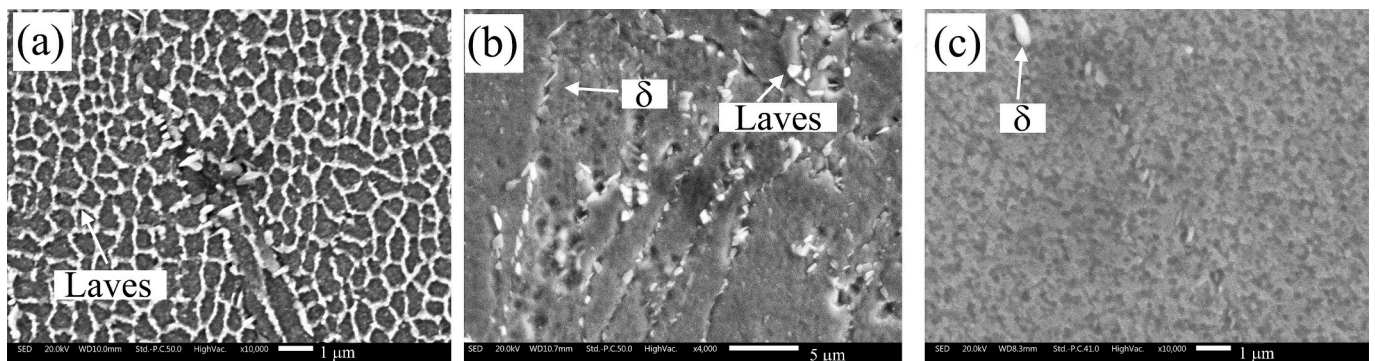


Fig. 7. SEM micrographs of (a) AA, (b) SA + AA, (c) HA + SA + AA.

γ^{II} phases, it is evident that the columnar structure of AB alloy, showing strong Nb, Mo microsegregation in the intercolumnar areas, gives reason of principal precipitation of γ^{I} in dendritic areas. Small amount of γ^{II}

precipitates in the intercolumnar areas, since presence of embrittled phases rich of Nb such as Laves phase, δ phase or/and NbC. Differently, after HA treatment the dissolution of Nb-rich phases and the

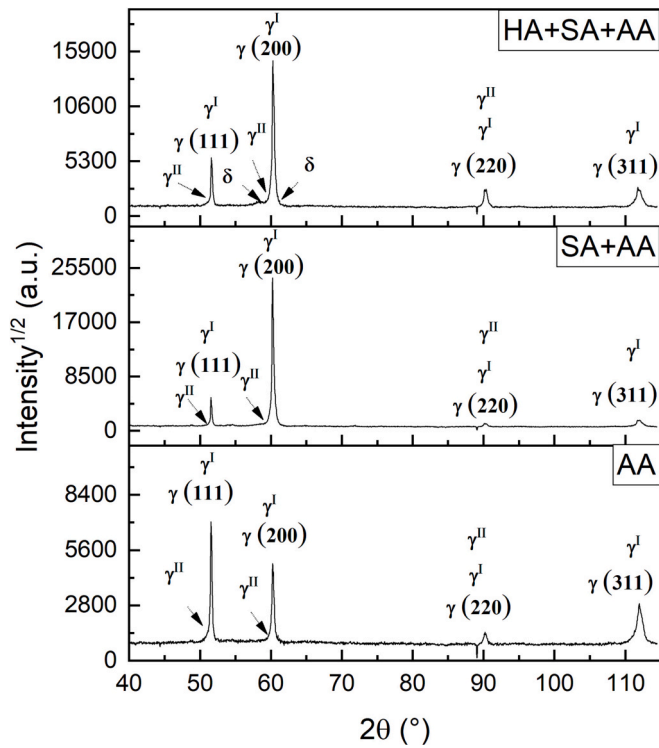


Fig. 8. XRD spectra for AA, SA + AA and HA + SA + AA samples in the parallel direction respect to the building one (z).

Table 6
Quantitative phase analysis through Maud software.

Quantitative analysis (% vol.)				
	γ	γ^I	γ^{II}	δ
AA	83	9	8	—
SA + AA	84	9	7	—
HA + SA + AA	76	11	4	9

homogenization of the elements could promote the precipitation of γ^{II} rather than γ^I .

3.2.1. Ageing behaviour

Dilatometric curves were obtained to evaluate the different phase transformations that occur during the heat treatment of as-built alloy (Fig. 9). In general, it is left to be noticed that in the differential dilatometric curves ($d\Delta/dt$) contraction peaks (downward oriented) are related to precipitation phenomena, while expansion peaks (upward oriented) refer to dissolution ones.

The first curve up to 1100 °C displays the transformations occurring in the AB alloy during heating up to 1100 °C, i.e. during the heating stage of HA heat treatment: three contraction peaks, namely I, II and V at 525, 725 and 935 °C, and three expansion peaks, III, IV and VI at 803, 900 and 990 °C, could be individuated. On the base of microstructural characterization and XRD analysis (Table 6), γ^I and γ^{II} precipitate after AA that occurs at a maximum temperature of 720 °C [49,67–70]. This is further confirmed by the isochronal ageing curve (red curve) showing a first slight hardening after the peak I and a much stronger one after peak II, in agreement with the strengthening effect of these phases. It is possible to confirm that peak I and II refer to γ^I , γ^{II} precipitations. Since peak III refers to a dissolution phenomenon and the amplitude is similar to the addition of peak I and II, it is possible to associate it to the dissolution of γ^I and γ^{II} . Considering XRD patterns (Fig. 7) only after HA + SA + AA treatment the formation of δ phase was observed. Based on this result, contraction peak around 935 °C and the dissolution peak at around 990 °C are associated to the precipitation and dissolution of the δ phase, respectively. The transformation overlap, particularly in the temperature range between 800 and 930 °C, where the large contraction associated to peak V (with the onset at 803 °C, and the peak minimum at 935 °C) is superimposed to the expansion related to peak IV. Hence, the precipitation of δ occurs simultaneously to the dissolution of Laves phase, as described in previous works [49–54]. To clarify the phase that precipitate at around 500 °C, isothermal treatment at 500 °C for 2 h on AB, HA and HA + SA samples was carried out and the dilatometric curves are shown in Fig. 10.

The dilatometric curves underline that the precipitation proceeds at the start of the isothermal stage and only in case of AB sample. On the other hand, the lack of a clear contraction for HA and HA + SA during isothermal treatment at 500 °C is evened.

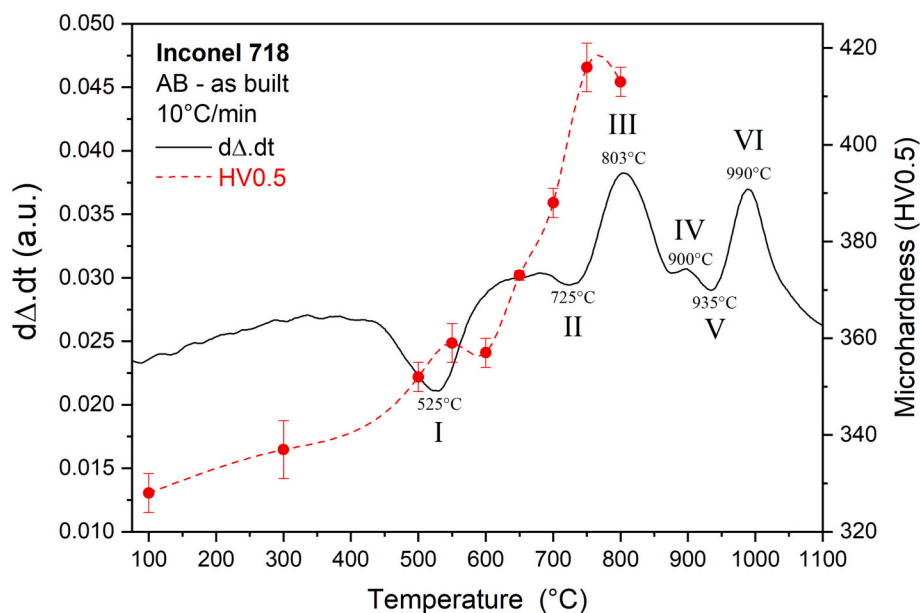


Fig. 9. Dilatometric curve of IN718 (black) and isochronal ageing curve (red). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

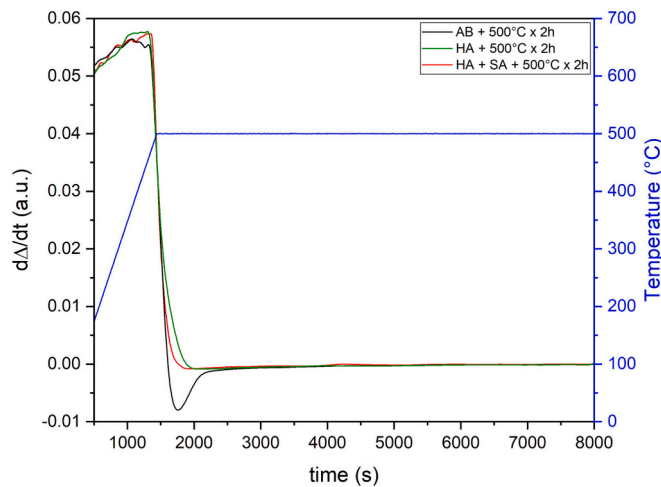


Fig. 10. Dilatometric curves of the isothermal stage at 500 °C for sample AB+500 °C, HA + 500 °C and HA + SA + 500 °C.

XRD patterns of AB, HA and HA + SA samples aged at 500 °C for 2 h confirm these statements (Fig. 11).

Quantitative analysis of XRD patterns using Maud software is reported in Table 7.

The quantitative analysis of phase constitution carried out on AB+500 °C x 2 h using the Maud software revealed about 16%vol. γ^I , and

Table 7

Quantitative XRD phase analysis using Maud software for AB, HA and HA + SA samples, preaged a 500 °C x 2 h.

Quantitative analysis (vol%)				
	γ	γ^I	Laves phase	δ
AB + 500 °C x 2 h	78	16	6	—
HA + 500 °C x 2 h	92	3	—	5
HA + SA + 500 °C x 2 h	92	3	—	5

6%vol. Laves phase, i.e. the same fraction of this last phase measured in the AB sample. Accordingly, peak I could be ascribed to the precipitation of γ^I and consequently peak II to the precipitation of γ^{II} that is the real strengthening phase as shown in isochronal ageing curve (Fig. 9). Considering HA + 500 °C x 2 h and HA + SA + 500 °C x 2 h samples, 3% vol. γ^I , and 5%vol. δ were detected confirming a less intense γ^I precipitation than in AB + 500 °C x 2 h.

Fig. 12 highlights the influence of 500 °C x 2 h preageing on the dilatometric behaviour of AB, HA and HA + SA.

The extensive precipitation of γ^I , confirmed by XRD, suppressed peak I and remarked the presence of an expansion just before peak II for AB, like for HA and HA + SA with pre-ageing at 500 °C for 2 h (Fig. 7). The above hypothesis about the origin of such expansion, i.e., the dissolution of γ^I , seems to be verified.

Considering the annealing curves of HA and HA + SA, the peak I is almost undetectable (Fig. 13).

The disappear of peak IV in HA sample is justified considering that HA treatment leads to the complete dissolution of Laves phase [47]. Tracing the tangent to the low T part of the curves (dashed line), a contraction is still visible, but definitely much less intense than in AB. Differently, peak II becomes more intense and shifted to lower temperature. In both cases, the contraction peak is anticipated by an expansion that is related to the anticipated dissolution of the small amount of γ^I [69]. Considering AB and SA samples, element microsegregation and presence of Laves phase promote the precipitation of γ^I to the detriment of γ^{II} . Differently, after HA thermal treatment, complete dissolution of Laves phase and homogenization occur. As a consequence, during the subsequently heating up to 1000 °C just a small amount of γ^I precipitate and due to the high amount of Nb released by Laves phase dissolution, a huge γ^{II} precipitation and an early γ^I dissolution takes place. The curve of SA is very similar to AB, in particular peak I remains well evident, confirming that his disappearance is related to some effect occurring during HA, i.e. at temperature higher than SA.

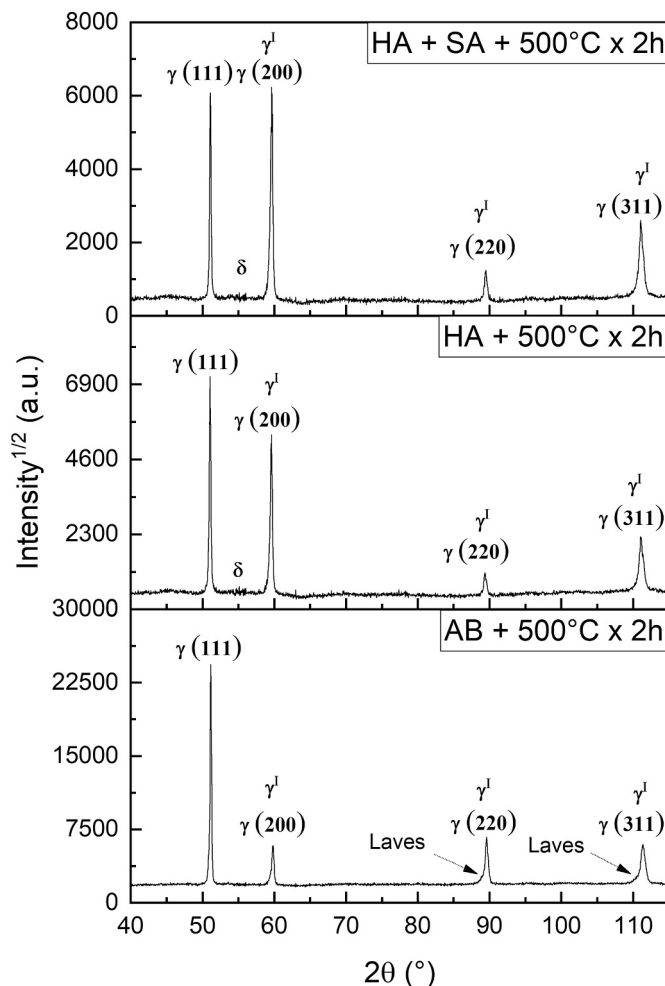


Fig. 11. XRD patterns for AB, HA and HA + SA samples aged at 500 °C x 2 h.

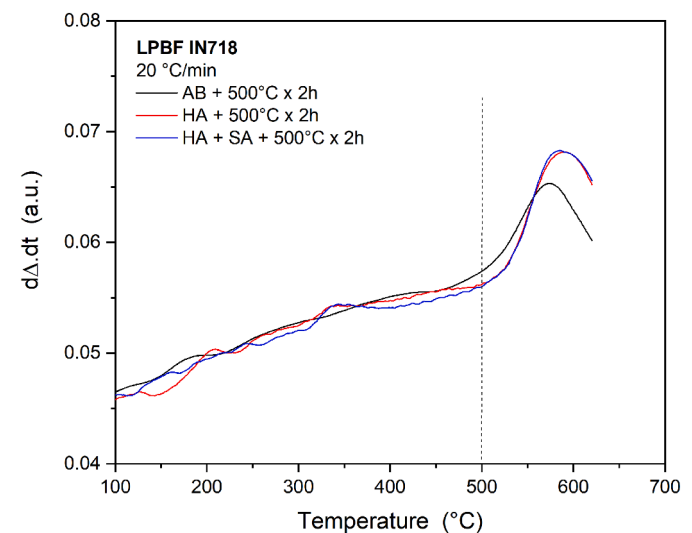


Fig. 12. Dilatometric curves of AB, HA and HA + SA samples aged at 500 °C x 2 h.

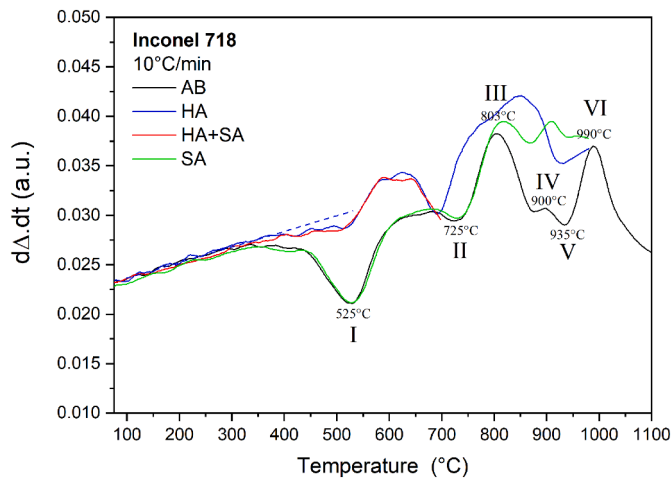


Fig. 13. Dilatometric curves after different thermal treatments: HA, SA after HA and AA after HA + SA.

FE-SEM investigation was performed on AB, AB +500, AA, HA and HA + SA + AA to confirm the nanometric detected phases through XRD after the different thermal treatments (Fig. 14).

EDXS analysis of the different detected phases are summarized in Table 8.

C amount is not detectable through EDXS analysis but a high peak of C was observed in case of carbides. Presence of MC (NbC) was also highlighted through Scheil simulation (Fig. 5b) confirming that this carbide was formed during the production process [32,38]. NbC was not evaluated through XRD analysis since it is in amount lower than 2 vol%, the detectable limit of the XRD analysis. Phases which could be formed during the different thermal treatment may be predicted through equilibrium (Fig. 15a) or metastable (Fig. 15b) thermodynamic calculations depending on the temperature of heat treatment and on the cooling rate.

In both diagrams, presence of $M_{23}C_6$ carbide at around 650 °C is evident, confirming the EDXS analysis on AA samples. The volume

Table 8

EDXS analyses of the different phases detected in the AB, AB +500, AA, HA and SA + HA + SA samples.

EDXS analyses (wt%)							
	Ni	Cr	Fe	Nb	Mo	Ti	C*
$M_{23}C_6$	Bal.	67.0	4.7	–	4.7	–	High
MC	Bal.	–	–	63.2	–	6.8	High
Laves	Bal.	12.3	10.2	13.2	7.7	1.6	–
d	Bal.	10.1	8.7	14.9	4.6	8.2	–

fraction of MC and $M_{23}C_6$ results lower than 2 vol% confirming the absence of these carbides in XRD spectra. In contrast with microstructural analysis results for AA (Fig. 14c), d phase precipitation is predicted under equilibrium conditions below 1040 °C. In view of the non-equilibrium solidification microstructure produced by L-PBF process, characterized by micro-segregation of elements, the possible inhibition of d phase precipitation must be taken into account and a metastable diagram has to be considered. The metastable property diagram calculated suppressing the precipitation of d phase (Fig. 15b), exhibits the formation of g^I and g^{II} , in line with XRD results (Fig. 8). Considering HA treatment, presence of d phase was observed since the homogenization of the microstructure with total removal of the solidification structure of L-PBF. The nanometric particles highlighted inside the matrix in high magnification micrographs of AB+500 (Fig. 14f), AA (Fig. 14g), HA (Fig. 14h) and HA-SA-AA (Fig. 14i), were not possible to be characterized through EDXS analysis since they are too fine and a 3 kV acceleration voltage with low working distance was used to resolve them, this leads to highly incorrect EDXS analysis. From high magnification micrographs it is possible to highlight a smoother matrix in case of AB sample (Fig. 14e) confirming the absence of g^I and g^{II} precipitates as shown by XRD analysis (Fig. 8). Small amount of round particles seems to be present around the NbC in case of HA sample (Fig. 14h). Nanoparticles with a round shape were observed after isothermal treatment up to 500 °C (Fig. 14f). Differently, after direct AA (Fig. 14g) and HA-SA-AA (Fig. 14f), high amounts of nanoparticles with a spherical and lenticular-like shape were observed. To further investigate the

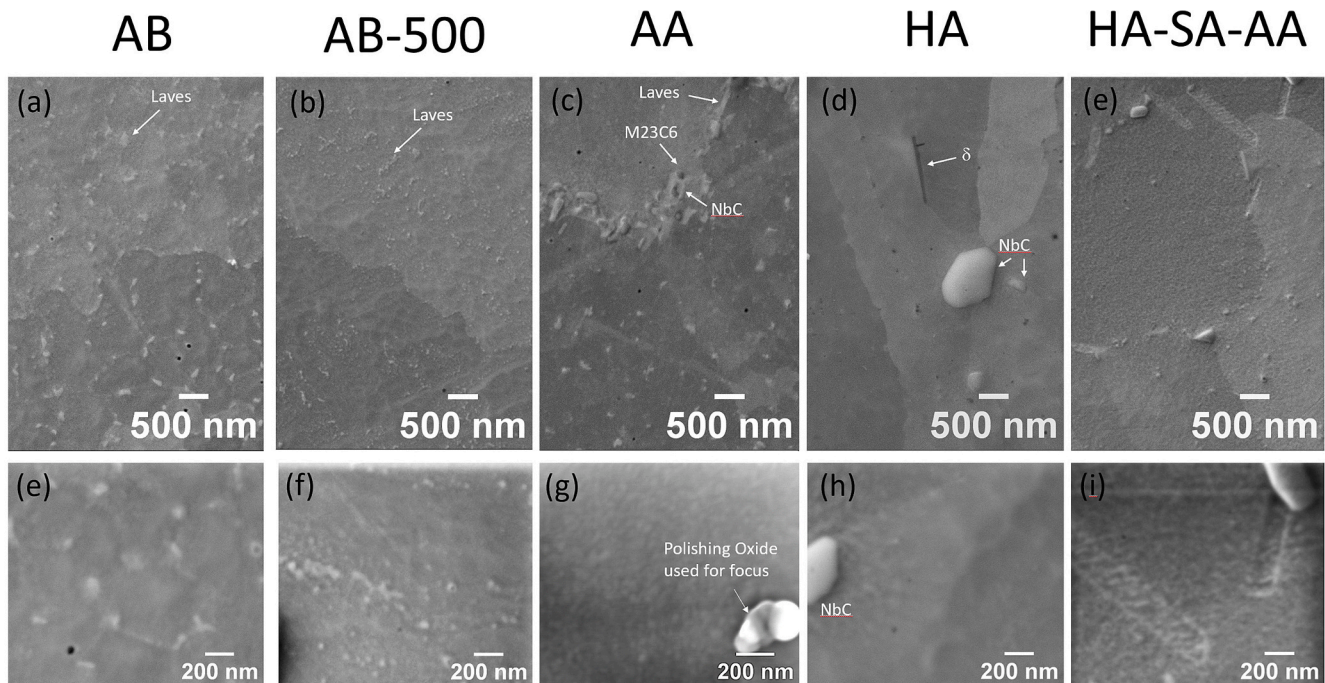


Fig. 14. FE-SEM micrographs of AB, AB +500 °C, AA, HA and HA-SA-AA samples at a magnification of (a,b,c,d,e) 30kX and (e,f,g,h,i) 120kX to highlight nanometric phases, respectively.

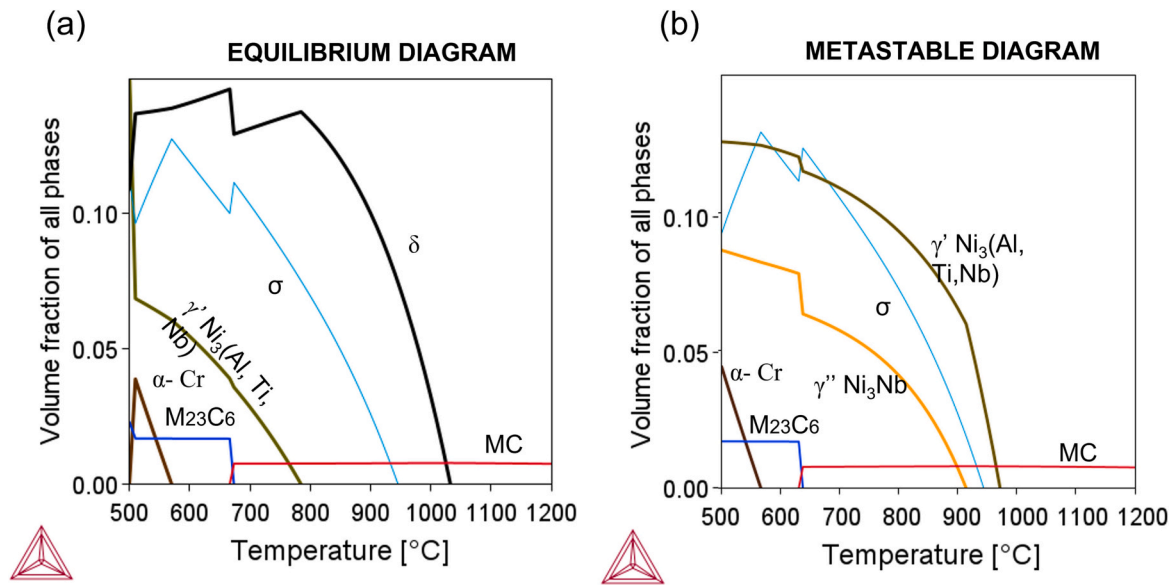


Fig. 15. a) Equilibrium diagram and b) metastable diagram obtained by suppressing the formation of d phase by means of Thermo-Calc.

precipitation sequence of g^I and g^{II} highlighted by dilatometry (Fig. 9), one AB sample was aged at 500 °C for 30 h (AB-500_30h) while a second AB sample was firstly homogenized (HA) to inhibit the phase precipitation that occurs at 500 °C (Fig. 10) and subsequently aged at 720 °C for 24 h (AB-HA-500_24h). In both cases, the very long dwell time were aimed at coarsening the nanometric precipitates so to better characterize them by EDXS. The results of FE-SEM and EDXS analysis performed on these two samples are shown in Fig. 16 and Table 9, respectively.

Spherical particles with a diameter of around 50 nm containing Nb, Cr, Al and Ti (spot 1) were observed in AB-500_30h. While lenticular particles with a length of around 100 nm rich of Nb without Ti and Al (spot 2) were observed in AB-HA_720_24h. According to their morphology and composition these precipitates could be associated to g^I and g^{II} , respectively [38,70–72]. This microstructural evidences corroborate the results of dilatometric and XRD analysis, where dilatometric peaks observed at 525 °C and at 725 °C were correlated with g^I and g^{II} precipitation, respectively.

A complete list of phase transformations involved is summarized in Table 10.

Since very few authors previously investigated γ^I precipitation, dilatometric and DSC analysis were performed by varying the heating rate to determine the activation energy associated to this transformation.

Table 9

EDXS analysis of spherical particles (spot 1) observed in AB-500_30h and of lenticular-lake particles (spot 2) observe in AB-HA-720_24h.

EDXS analyses (wt%)							
Spot	Ni	Cr	Fe	Nb	Mo	Ti	Al
1	Bal.	16.8	13.6	11.8	–	3.5	0.9
2	Bal.	7.1	8.8	17.5	3.3	–	–

Table 10

The list of phase transformations evidenced by dilatometric curves in Fig. 9.

	T peak	Strain	Transformation [49,67–70]
I	525 °C	–	$\gamma \rightarrow \gamma^I$
II	725 °C	–	$\gamma \rightarrow \gamma^{II}$
III	803 °C	+	γ^I - γ^{II} dissolutions
IV	900 °C	+	Laves phase dissolution
V	935 °C	–	$\gamma \rightarrow \delta$
VI	990 °C	+	δ dissolution

3.2.2. γ^I precipitation

The derivative curves of AB measured at different heating rates evidence that increasing the heating rate is responsible for a rightwards shift of the kinetic maximum of the transformation occurring in the 500–600 °C range. The same measurements carried out by DSC led to the same result (Fig. 17).

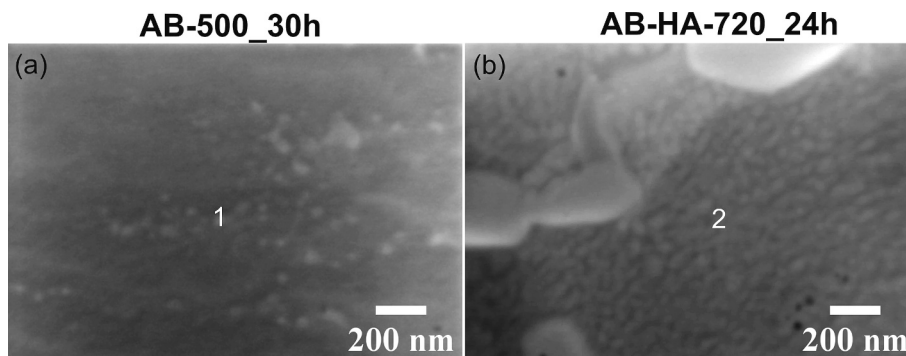


Fig. 16. FE-SEM micrographs of a) AB-500_30h and b) AB-HA-720_24h.

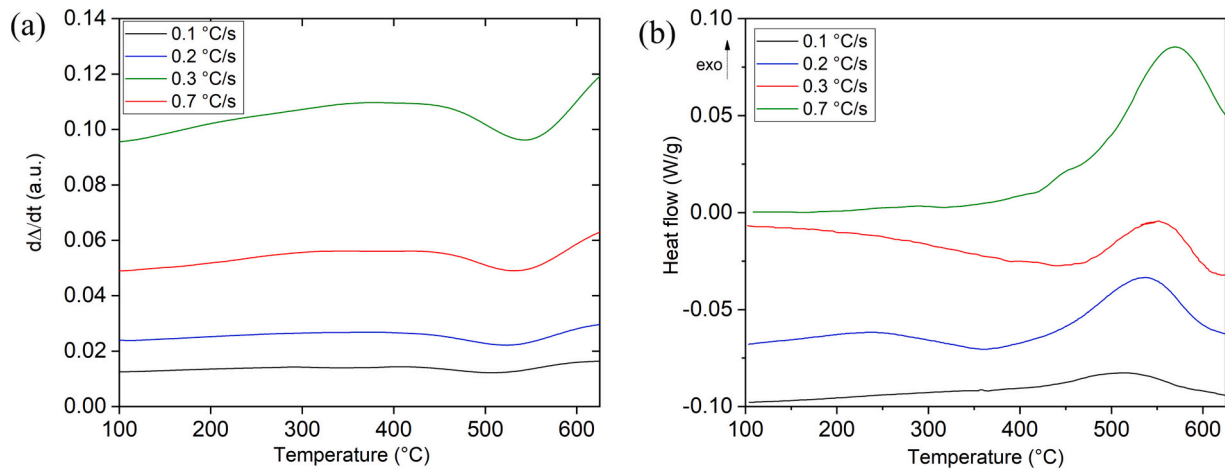


Fig. 17. a) Dilatometric and b) DSC curves obtained at different heating rates.

The kinetics maxima for all the heating rates are reported in Table 11.

The DSC peak temperatures are higher than those recorded by dilatometry, fact that has been of concern for other authors in the past [82].

Measured kinetic maxima can be related by Eq. (1) [80,81], for which the ratio between peak temperatures and heating rate is linearly dependent with the reciprocal of temperature, in an Arrhenius-like relationship known as Kissinger equation. From a linear interpolation of the points a value of activation energy can be found. Comparing the activation energies obtained using dilatometry and DSC (Table 11), a slightly lower value was obtained by DSC, although the R^2 and the correlation was quite good in both cases. Average value of γ^I activation energy was of 248 ± 21 kJ/mol that is similar to those of diffusion of Al into Ni alloy (230 kJ/mol) confirming that γ^I precipitation is governed by this phenomenon.

3.3. Mechanical properties

To evaluate the effect of the standard thermal treatments on the IN718 performances, tensile tests on AB, AA, SA + AA and HA + SA + AA samples were conducted. The relevant engineering stress-strain curves at room temperature are displayed in Fig. 18.

Mechanical properties (yield strength σ_y , ultimate tensile strength UTS, rupture elongation ϵ_R) microhardness values, and the microstructure/phase constitution according to the results reported in the previous chapters are summarized in Table 12, to emphasize the structure-property relationship.

The results show a remarkable difference between the AB and the heat treated alloy. As expected, the as built sample exhibits the lowest tensile strength and the highest elongation to fracture, due to the lack of strengthening phases precipitation during ageing. After heat treatment all samples show increased strength and a decreased ductility, due to the precipitation of γ^{II} during double ageing (AA). In particular, the sample double aged directly after printing (AA) shows the highest strength and

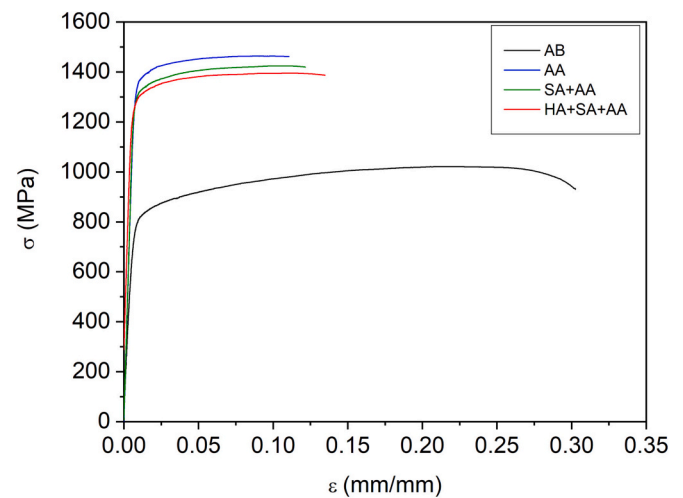


Fig. 18. Tensile engineering stress-strain curves of the AB, AA, SA + AA, HA + SA + AA samples at room temperature.

the lowest rupture elongation. Addition of SA and HA + SA leads to a progressively lower strength and increased ductility. This is justified considering the partial (SA) or complete (HA) recrystallization that deletes the as-built microstructure and the high amount of structural defects (dislocations) introduced by LPBF during rapid solidification.

The lowest strength and highest ductility of HA + SA + AA respect to SA + AA is justified by the coarser grain size due to the higher temperature reached in HA in agreement with the Hall-Petch relationship. Considering the microhardness values (Table 12), direct ageing AA considerably enhanced microhardness of as built IN718 from 327 ± 2 HV0.5 up to 479 ± 4 HV0.5 due to precipitation of γ^{II} . SA + AA and HA + SA + AA samples present slightly higher microhardness than directly aged sample and this is thought to be related to the dissolution of Nb-rich phases and the homogenization of the elements due to SA and or HA that promote the precipitation of γ^{II} rather than γ^I as shown from dilatometric analysis (Fig. 13).

These mechanical properties obtained in present work are very good, if compared with data reported in literature [43,45,70,90,91] (Fig. 19).

In particular, the mechanical properties measured in AB condition are slightly improved compared to those observed in literature. In details, higher ductility with slightly lower strength was observed compared with Zhang et al. [40]. Differently, comparing with Li et al. [53] similar ductility but higher strength was obtained in this work. Considering the AA, higher ductility and UTS but lower σ_y , were

Table 11

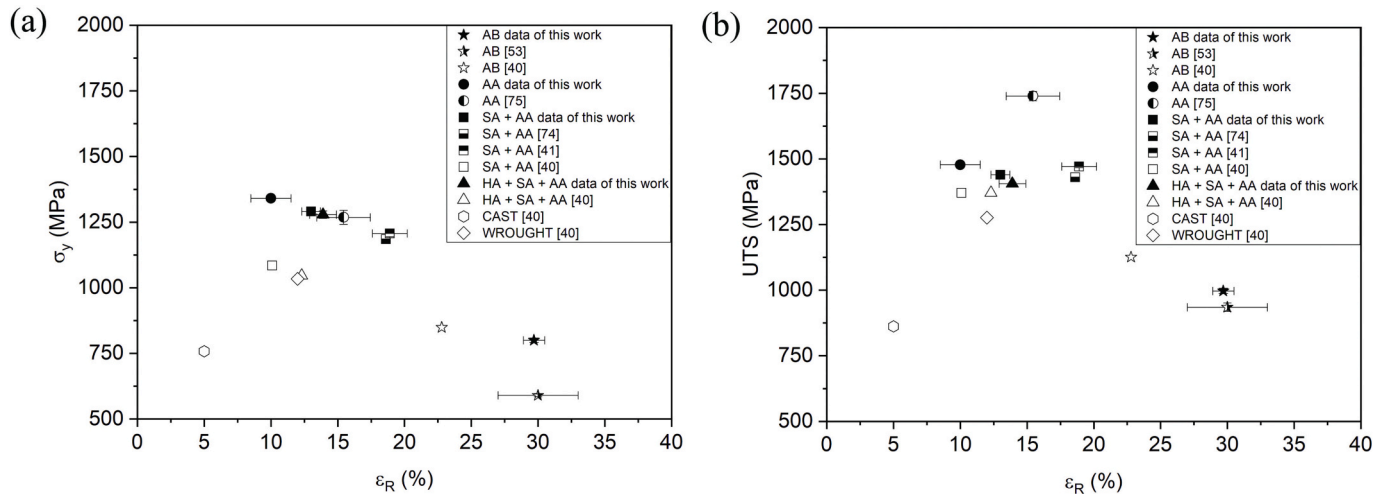
Peak temperatures determined by dilatometry and DSC at different heating rates, and values of the activation energy E_a with the two techniques.

	T_{peak} (°C)				E_a (KJ/Mol)	R^2
	5 °C/min (0.08 K/s)	10 °C/min (0.17 K/s)	20 °C/min (0.33 K/s)	40 °C/min (0.67 K/s)		
DIL.	507	525	536	546	263	0.9844
DSC	505	540	550	571	233	0.9557

Table 12

Mechanical properties of AB, AA, SA + AA, HA + SA + AA samples.

	σ_y (MPa)	UTS (MPa)	ϵ_R (%)	HV0.5	Microstructure	Phase
AB	800 $\pm$ 8	997 $\pm$ 10	29.7 $\pm$ 0.8	327 $\pm$ 4	Columnar	γ , laves phase
AA	1341 $\pm$ 2	1478 $\pm$ 4	10.0 $\pm$ 1.5	479 $\pm$ 2	Columnar	γ , laves phase, γ^I , γ^{II}
SA + AA	1291 $\pm$ 10	1440 $\pm$ 1	13.0 $\pm$ 0.7	502 $\pm$ 7	Microstructural homogenization, partial recrystallization	γ , laves phase, γ^I , γ^{II}
HA + SA + AA	1279 $\pm$ 14	1406 $\pm$ 4	13.9 $\pm$ 1.0	487 $\pm$ 1	Microstructural homogenization, recrystallization, grain growth	γ , δ , γ^I , γ^{II}

**Fig. 19.** Comparison of mechanical properties obtained by the different thermal treatment conditions with the data shown in literature [43,45,70,90,91].

observed by Schneider et al. [75]. Considering SA + AA and HA + SA + AA, higher strength and ductility was obtained comparing with Zhang et al. [40]. In case of HA + SA treatment, Trosch et al. [74] and Huang et al. [41] observed higher ductility, lower σ_y but similar UTS. Considering the mechanical properties of cast and wrought IN718 [74], it is event that the alloy produced by LPBF shows improved properties, confirming the good potential of this technology for industrial applications. The results of this investigation confirm that final properties are strongly correlated to the specific heat treatment strategy, i.e. to the specific microstructure. Nevertheless, present results, do not completely address the choice of the most proper heat treatment, which should be evaluated time by time for each specific application. Indeed, just only with AA it is possible to achieve high strength with a high reduction of process cost comparing with SA + AA and HA + SA + AA. If higher ductility is necessary, probably just SA + AA permits to obtain the required properties without the need for HA addition. Nevertheless, properties other than those presented in this paper should be also considered, like impact, fracture toughness and fatigue strength. Most of them are much more sensitive to the improved microstructural homogeneity induced by solution annealing and homogenizing.

At the end, fracture surfaces of the different thermal treated sample are reported in Fig. 20.

In all conditions the rupture of the samples occurred by ductile fracture, underlined by the presence of dimples. Dealing with the AB material (Fig. 20a, b), high melting pools cohesion was observed since it is not evident a stepwise geometry that follows the laser traces. The epitaxial columnar growth is visible in the AB and AA (arrow in Fig. 20b, d) samples and disappears in the SA + AA (Fig. 20e, f) and HA + SA + AA (Fig. 20g, h) samples since the partial or complete recrystallization.

4. Conclusions

In this work, the heat treatment behaviour of IN718 obtained by LPBF was studied. The different phase transformations occurring during the homogenization (HA), solution annealing (SA) and artificial ageing (AA) were investigated. The precipitation sequence of γ^I , γ^{II} and δ was

evaluated by dilatometry under different conditions, namely after direct ageing (AA), after solution annealing (SA) and after homogenizing followed by solution annealing (HA + SA). The mechanical properties after different treatments were compared and correlated with the microstructure to understand the effect of the strengthening phases on the final performances of the material. The main conclusions can be summarized as follows.

- Microstructural and structural anisotropy are one of the main issues that must be considered in additive manufacturing parts since they primarily affect the dynamic behaviour during working. The AB and AA are strongly anisotropic in terms of microstructure since the evident dominant columnar structure and also present a structural anisotropy due to the huge orientation of the grain. This is highlighted by the stronger (200) texture visible in the AB longitudinal sample due to the preferential $\langle 111 \rangle$ growth direction of γ -fcc grains, parallel to the build one. SA partially removes anisotropy but the orientation of grains and traces of laser pattern are still observable. HA + SA is therefore necessary to remove it.
- Dilatometric peaks related to the precipitation of γ^I , γ^{II} and δ phases were detected at 525, 725 and 935 °C, respectively. Dissolution peaks of the same three phases were observed at 803 °C, 900 °C and 990 °C, respectively. The dendritic microstructure of the AB sample was favourable for the precipitation of γ^I rather than γ^{II} . A similar behaviour was exhibited by SA sample. Differently, the dissolution of Nb-rich phases and the partial redistribution of alloying elements during HA promote the precipitation of γ^{II} rather than γ^I , controlled by the diffusion of Nb and Al into Ni, respectively.
- The average activation energy for γ^I precipitation determined by dilatometry and DSC was 248 $\pm$ 21 kJ, similar to that of Al diffusion into Ni.
- The strengthening effect of AA in IN718 is attributed to the precipitation of γ^{II} . The contribution associated to the to the precipitation of γ^I is considerably lower.
- Mechanical properties are strongly affected by the specific heat treatment. Direct ageing (AA), preserving most of the structure

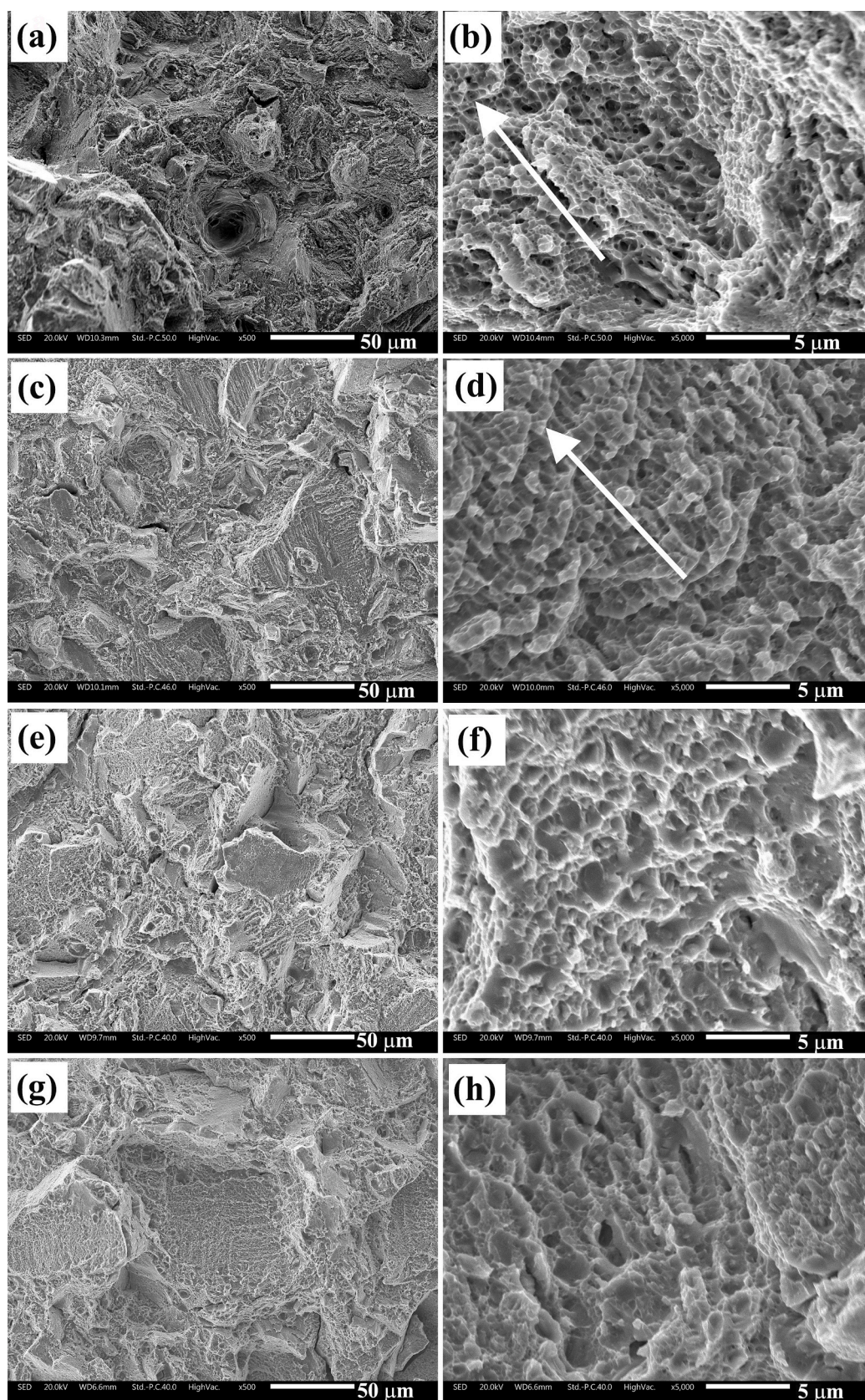


Fig. 20. Fracture surface of (a,b) AB, (c,d) AA, (e,f) SA + AA, (g,h) HA + SA + AA.

induced by LPBF process, led to the maximum tensile strength (1479 MPa) and limited fracture elongation (10%). Solution annealing followed by ageing (SA + AA) and homogenizing followed by solution annealing and ageing (HA + SA + AA) exhibited progressively lower strength (1440, 1406 MPa) and higher ductility (13.0, 13.9%) due to the partial or complete recrystallization.

Declaration of Competing Interest

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

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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