High temperature tensile properties and microstructure of Ni20Cr alloy fabricated by laser powder bed fusion

Shubham Sanjay Joshi[1, a*], Clément Keller[2, b], Williams Lefebvre[1, c], Eric Hug[3, d]

[1] Groupe de Physique des Matériaux, Normandie Université, INSA de Rouen Normandie, Université de Rouen, UMRCNRS 6634, Saint-Etienne du Rouvray, France
[2] Laboratoire Génie de Production, INP-ENIT, Tarbes, France
[3] Laboratoire CRISMAT, Normandie Université, Université de Caen, UMRCNRS 6508, Caen, France
[a]shubham.joshi@insa-rouen.fr, [b]ckeller@enit.fr, [c]williams.lefebvre@univ-rouen.fr, [d]eric.hug@ensicaen.fr

Keywords: Additive manufacturing, LPBF, Ni-alloys, Ni20Cr, high-temperature testing, dynamic strain ageing, Portevin–Le Chatelier effect, microstructure.

Additive Manufacturing (AM) brings about an array of modifications in microstructure with respect to conventional routes transforming mechanical performances. These new microstructure features depend on process parameters and especially on volume energy-density delivered by the laser on powder layer. Among the different alloys manufactured by AM, Ni-alloys exhibit high-strength at elevated temperature opening the way of fabrication of gas turbines and jet-engine parts. Ni-superalloys experience precipitation hardening due to the formation of γ' and γ'' phases leading to complex microstructures. To better study the influence of the AM microstructure on Ni-alloys mechanical properties, in particular at elevated temperatures, a theoretically monophasic and binary Ni20Cr-alloy manufactured by laser powder-bed fusion was studied in this work. Remarkable Yield Strength (400 MPa) and Ultimate Tensile Strength (UTS) (600 MPa) were observed at 500°C with hardly any loss of properties from room temperature, owing to the thermal stability of cellular dendrites till 700°C. Ductility drop was reported at 700°C due to anomalous brittle behaviour of Ni-alloys. Hardening behaviour vanished at 900°C signifying the deletion of dendrites, disappearance of dislocations, diffusion of Cr from dendritic walls and growth of oxides.

This non edited article has been accepted for publication in Key Engineering Materials: https://doi.org/10.4028/p-0Wza3S. The authors acknowledge this journal for the publication.

Introduction

Ni-alloys exhibit excellent high temperature mechanical performance and corrosion resistance in variety of alloy systems like Inconel 718 [1], IN 625 [2], IN 792 [3], etc. Similarly Ni20Cr, a binary alloy is employed in industrial applications where high creep resistance in the temperature range of 550°C to 950°C is required [4]. Ni20Cr also forms a model material for superalloys due to similar Cr content [5], [6]. The process of laser powder bed fusion (LPBF) has been on the forefront of manufacturing technologies in recent years to fabricate near-net-shaped parts bringing about a tremendous flexibility in the processing sector. Since more than a decade now, Ni-alloys have been successfully printed using various LPBF systems and parameters [1][2][3]. However, LPBF modifies the microstructure of material in as-built state inducing porosities, oxides, lack of fusions, metastable precipitates, residual stresses, etc [7], and further this modified microstructure unquestionably impacts the mechanical performance. Hence it becomes important to characterise the mechanical properties of LPBFed materials in extreme conditions like high temperature to avoid catastrophic failures in industry. Li et al [8] pointed comparable creep strength of LPBFed IN 625 between 650°C and 800°C than its wrought counterpart. IN792 exhibits little or no modification in Yield Strength (YS) and Ultimate Tensile Strength (UTS) till 760°C but decreases significantly above that temperature [3]. Whereas, Liu et al. [9] focussed on the supposed negative influence of incompatibly between columnar grains formed parallel to the building direction in Hastelloy X on their high temperature tensile properties [10]. As a result, the goal of this work is to widen our understanding of how the typical LPBFed microstructure changes with temperature, as well as to comprehend the raised temperature tensile performance of LPBFed Ni20Cr alloy.

Experimental procedure

The SLM125HL machine was used to print the Ni20Cr samples via Laser Powder Bed Fusion (LPBF) process. Cylindrical samples with dimensions of 8 mm in diameter and 90 mm in length were printed and then machined to create ‘screw-headed’ dog-bone specimens of 4 mm diameter and gauge length of 22 mm according to ISO 6892-1 (ASTM E8). For LPBF fabrication, laser power of 200 W, scanning speed of 900 mm/s, hatch distance of 0.12 mm, powder layer thickness of 30 µm were considered to input volumetric energy density of 62 J/mm^3 in the system. Further, the scanning strategy was selected to be stripes with rotation angle of 67°. A Scanning Electron Microscope (JEOL 7900F with SE detector and Electron Back-Scattered Detector (EBSD) along with Electron Channelling Contrast Imaging (ECCI) method was employed for microstructural characterisation. The samples underwent mechanical grinding with SiC papers followed by electropolishing using Struers A2 electrolyte. For EBSD data acquisition, a step size of 1.5 µm and a 5° misorientation criterion for grain boundary characterisation were designated. Monotonic tensile tests were carried out under strain-controlled circumstances using an MTS servo-hydraulic machine with a load capacity of 100 kN and a "clip-on" extensometer. The strain rate employed was 10^{-3} s^{-1} and testing temperatures were 200°C, 500°C, 700°C and 900°C. The machine is equipped with furnace of maximum capacity of 1000°C. About 30 minutes are for the system (furnace and sample) to reach a constant temperature for accurate test (no thermal dilatation).

Results and Discussion

Initial microstructural investigation: Fig. 1(a)-(c) depicts SEM micrographs of as-built LPBF Ni20Cr samples. Fig. 1(a) heterogenous grain structure with lasing planes of 67° same as lasing rotation angle. Fig. 2(c) indicates a typical cellular dendritic structure (average size = 0.47 µm) which is formed due to rapid solidification behaviour of LPBF process. Cr-rich precipitates or oxides are formed in inter-dendritic zones as seen in fig. 1(b)-(c) as mentioned in literature [6]. Heterogeneous dislocations can be observed near cellular structures due to continuous heating and cooling cycles of LPBF process as seen in all LPBFed materials [7].

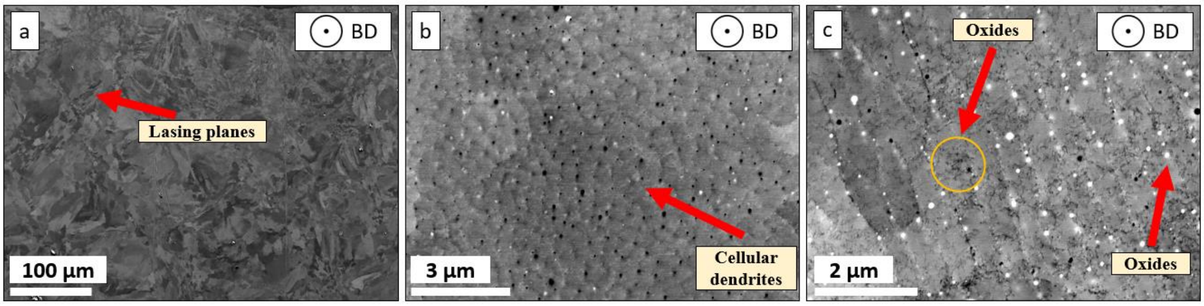


Figure 1: (a) General initial grain structure in XY plane, (b) Cellular dendritic structures and (c) Initial dislocation structures in cells of LPBFed Ni20Cr alloy using SEM (ECCI).

EBSD analysis yields grain orientation maps, kernel average misorientation maps, pole figures (PF) and geometrically necessary dislocation (GND) density maps for samples in YZ direction (fig. 2(a)-(d)) and for XY direction (fig. 2(e)-(h)). Maximum texture intensity is 2.387 in YZ direction is comparable to that of 2.375 in XY direction, indicating lack of crystallographic texture for this printing parameters set. KAM maps indicate presence of misorientation close to grain boundaries for LPBF Ni20Cr specimen in both directions. GND density maps also reveal the presence of GNDs close to grain boundaries as shown by Wang et al. [11].

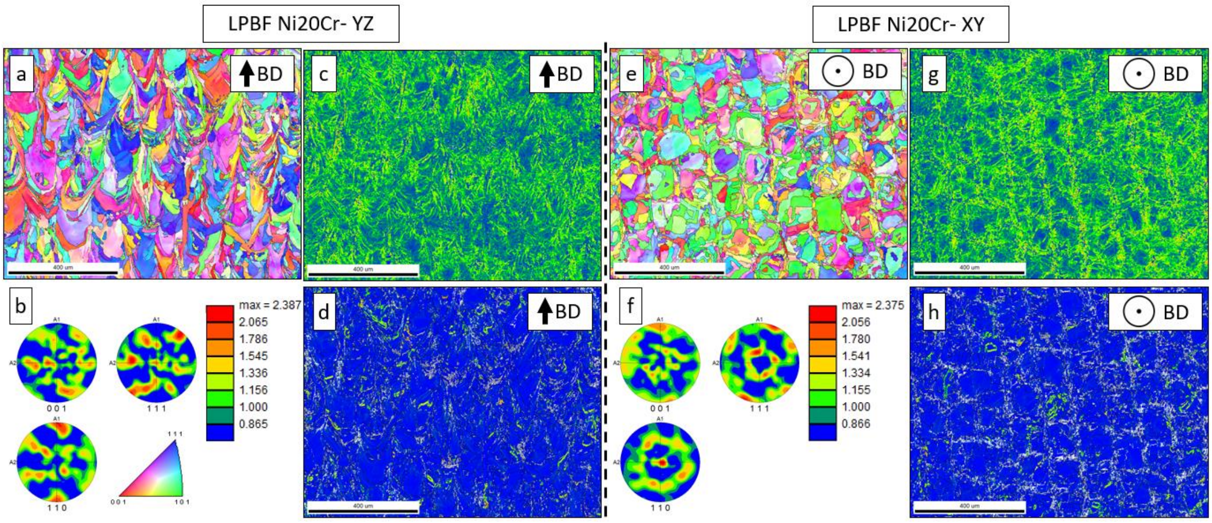
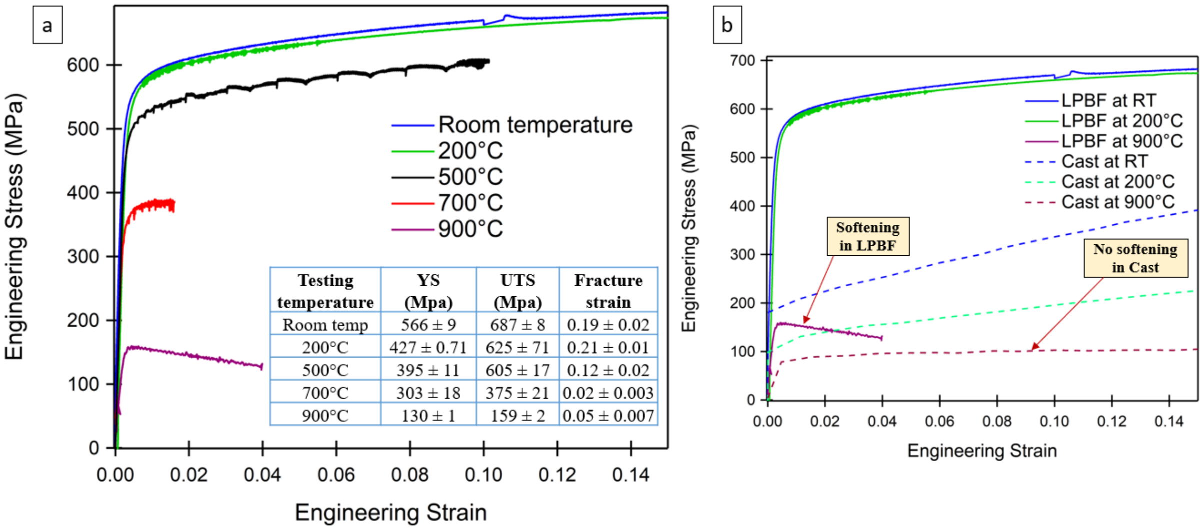


Figure 2: EBSD Inverse pole figure maps (a-e), PF maps with maximum texture intensities (b-f) with KAM (c-g) and GND maps (d-h) of the LPBF Ni20Cr in YZ and XY planes.

Monotonic tensile testing: Fig. 3 portrays engineering stress-strain curves for LPBF samples tested at room temperature, 200°C, 500°C, 700°C and 900°C, fig. 3(a) also contains YS, UTS and fracture strain values with standard deviation values. YS drops by close to 140 MPa from room temperature (566 MPa) to 427 MPa for 200°C, but UTS values are comparable till 200°C, indicating the presence of similar strain hardening behaviour unaffected by temperature. Elevated YS and UTS values are linked with presence of cellular dendrites [6]. Cr is segregated at the dendrites walls which hinders dislocation movement and also sustains stability of these dendrites. YS further lowers by 30 MPa for 500°C with comparable UTS values. Slight decrement in YS (of 95 MPa) with a significant decrement by 275 MPa in UTS values are experienced by samples at 700°C, also observed for IN 792 alloy beyond 760°C [3]. Noteworthy modification is the enormously early fracture at about of 2% elongation, classified to be an anomalous brittle behaviour experienced by several Ni-alloys [3]. Low YS (130 MPa) and UTS (160 MPa) values are detected at 900°C with LPBF samples undergoing softening. It is noteworthy to observe in fig. 3(b), that LPBF samples undergo softening at 900°C unlike cast samples which neither indicate softening not hardening. Tensile curves of Cast samples (same strain rate employed in literature [4]) are also given at room temperature and 200°C in fig. 3(b).

In order to understand the influence of temperature on the tensile behaviour, a pre-testing heat treatment was performed for 30 minutes (same time which is required for system to attain the testing temperature) at all testing temperatures, followed by water quenching to characterize the microstructure at the time of high temperature testing. Fig. 4(a)-(d) depicts expected microstructures at the beginning of the high temperature tensile tests. Fig. 4(a)-(b) indicates cellular dendritic structures are unchanged from those at as-built state, indicating thermal stability of dendritic structures. However, these dendrites seem to have partially deleted at 700°C as seen in fig. 4(c) as well as the inter-dendritic Cr-rich oxides appear to have grown in size, indicated by bright coloured particles. The trend continuous till 900°C with virtually complete deletion of dendrites with increased size of oxides.



Testing temperature	YS (Mpa)	UTS (Mpa)	Fracture strain
Room temp	566 ± 9	687 ± 8	0.19 ± 0.02
200°C	427 ± 0.71	625 ± 71	0.21 ± 0.01
500°C	395 ± 11	605 ± 17	0.12 ± 0.02
700°C	303 ± 18	375 ± 21	0.02 ± 0.003
900°C	130 ± 1	159 ± 2	0.05 ± 0.007

Figure 3: (a) Stress-Strain curves of LPBF Ni20Cr at room temperature, 200°C, 500°C, 700°C and 900°C with YS, UTS and fracture strain values; (b) Comparison of tensile curves of LPBF and Cast (from literature [4]) Ni20Cr at room temperature, 200°C and 900°C.

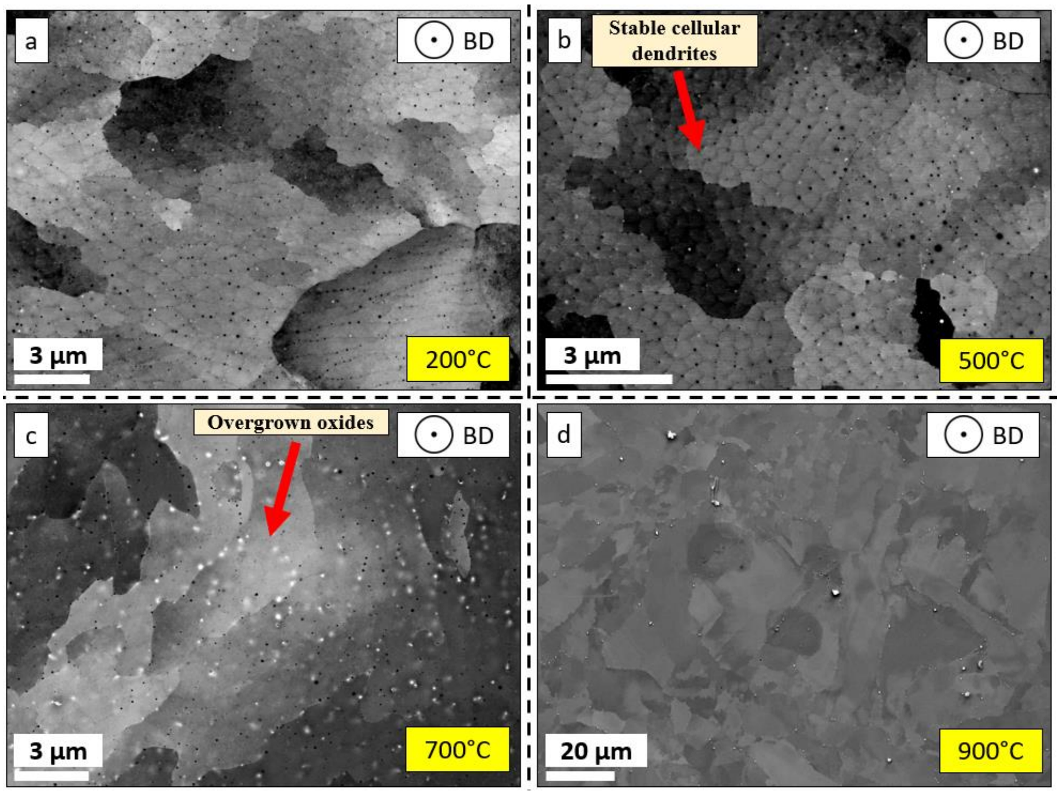


Figure 4: Microstructure of LPBF Ni20Cr alloy at pre-testing state after heat treatment for 30 minutes at (a) 200°C, (b) 500°C, (b) 700°C and (b) 900°C respectively using SEM (ECCI).

This could be associated with complete deletion of dendrites and overgrown oxides. Cr is believed to diffuse out of dendrite walls and dislocation deletion paves the way for vanishing of dendrites. It's crucial to point out that ductility rose from 700°C to 900°C. The curves indicate serrations at 500°C to 900°C demonstrating the presence of Dynamic Strain Ageing (DSA) indicating Portevin–Le Chatelier effect, also discussed by Kim et al for LPBFed IN 625 [12]. Such DSA behaviour is observed in several Ni-alloys between 450-700°C [3][12]. It is interesting to note that cast Ni20Cr samples do not experience such serrations at 200°C and 900°C (as seen in fig. 3(b)). The 500°C curve shows maximum serration amplitudes possibly linking to Short Range Order found in Ni-Cr system at 500°C.

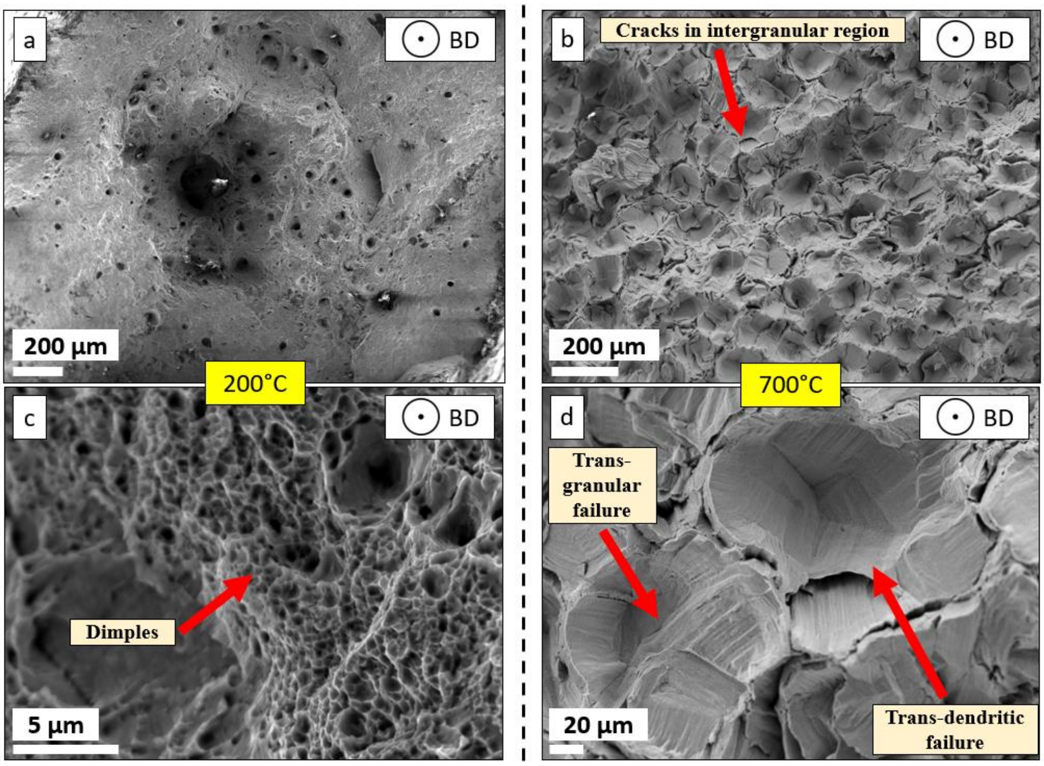


Figure 5: Fractography of LPBF Ni20Cr samples tested (a)-(b) 200°C, and (c)-(d) 700°C.

Fig. 5(a)-(d) shows the fracture surfaces for LPBF samples tested at 200°C and 700°C using the SEM. The sample tested at 200°C shows several voids in fig. 5(a) and ductile dimples whose sizes are comparable to the dendrite size (close to 0.5 µm). However, the fracture surface of sample tested at 700°C shows cracks in intergranular region with trans-dendritic and trans-granular failure seen in fig. 5(d). Fracture mechanisms seem to have modified at 700°C, in connection with changed microstructure especially deletion of dendritic colonies. Such a shift in fracture mechanisms can also been seen in literature, at 760°C testing of IN 792 alloy [3].

Conclusions

Following are the key conclusions of high-temperature tensile testing of LPBF-fabricated Ni20Cr:

- High Yield Strength and Ultimate Tensile Strength for LPBF Ni20Cr till 500°C.
- LPBF Ni20Cr shows thermal stability of cellular dendrites till 700°C.
- Softening behaviour of LPBF Ni20Cr at 900°C is associated to deletion of cellular dendrites, diffusion of Cr and growth of Cr-rich oxides, no such softening experienced by Cast Ni20Cr samples.

- Onset of the modification in fracture mechanisms observed at 700°C.
- Presence of Dynamic Strain Ageing from 500°C to 900°C indicating Portevin–Le Chatelier effect in LPBF Ni20Cr.
- Decreased ductility at 700°C due to ductility hole is present for LPBF Ni20Cr.

Acknowledgement: The Labex EMC3 for the financial support of the Nichrofab project.

References

[1] E. Paccou *et al.*, "Investigations of powder reusing on microstructure and mechanical properties of Inconel 718 obtained by additive manufacturing," *Materials Science and Engineering: A*, vol. 828, p. 142113, Nov. 2021, doi: 10.1016/j.msea.2021.142113.
[2] A. Banerjee *et al.*, "The mechanical response of additively manufactured IN625 thin-walled structures," *Scripta Materialia*, vol. 205, p. 114188, Dec. 2021, doi: 10.1016/j.scriptamat.2021.114188.
[3] B. Du, L. Sheng, Z. Hu, C. Cui, J. Yang, and X. Sun, "Investigation on the microstructure and tensile behavior of a Ni-based IN792 superalloy," *Advances in Mechanical Engineering*, vol. 10, no. 2, p. 1687814017752167, Feb. 2018, doi: 10.1177/1687814017752167.
[4] M. Rudloff, "Etude des mécanismes de transition volume/surface du comportement mécanique d'un alliage Ni20Cr," PhD dissertation, Université Caen Normandie, 2010.
[5] G. Calvarin-Amiri, A. F. Gourgues, A. M. Huntz, and R. Molins, "Effect of environment on the mechanical behavior of Ni–20Cr thin strip," *Materials Science and Engineering: A*, vol. 298, no. 1, pp. 200–208, Jan. 2001, doi: 10.1016/S0921-5093(00)01319-8.
[6] S. S. Joshi, C. Keller, L. Mas, W. Lefebvre, E. Hug, and J.-P. Couzinie, "On the origin of the strain hardening mechanisms of Ni20Cr alloy manufactured by laser powder bed fusion," *International Journal of Plasticity*, vol. 165, p. 103610, Jun. 2023, doi: 10.1016/j.ijplas.2023.103610.
[7] S. Gorsse, C. Hutchinson, M. Gouné, and R. Banerjee, "Additive manufacturing of metals: a brief review of the characteristic microstructures and properties of steels, Ti-6Al-4V and high-entropy alloys," *Science and Technology of Advanced Materials*, vol. 18, no. 1, pp. 584–610, Dec. 2017, doi: 10.1080/14686996.2017.1361305.
[8] S. Li, Q. Wei, Y. Shi, Z. Zhu, and D. Zhang, "Microstructure Characteristics of Inconel 625 Superalloy Manufactured by Selective Laser Melting," *Journal of Materials Science & Technology*, vol. 31, no. 9, pp. 946–952, Sep. 2015, doi: 10.1016/j.jmst.2014.09.020.
[9] M. Liu *et al.*, "High-Temperature Tensile Properties of Hastelloy X Produced by Laser Powder Bed Fusion with Different Heat Treatments," *Metals*, vol. 12, no. 9, Art. no. 9, Sep. 2022, doi: 10.3390/met12091435.
[10] M. Montero Sistiaga, S. Nardone, C. Hautfenne, and J. Van Humbeeck, "Effect of heat treatment of 316L stainless steel produced by selective laser melting (SLM)," in *Proceedings of the 27th Annual International Solid Freeform Fabrication Symposium - An Additive Manufacturing Conference,* Solid Freeform Fabrication, Aug. 2016, pp. 558–565. Accessed: Jun. 14, 2022. [Online]. Available: https://lirias.kuleuven.be/1942281
[11] Y. M. Wang *et al.*, "Additively manufactured hierarchical stainless steels with high strength and ductility," *Nature Mater*, vol. 17, no. 1, Art. no. 1, Jan. 2018, doi: 10.1038/nmat5021.
[12] K.-S. Kim, T.-H. Kang, M. E. Kassner, K.-T. Son, and K.-A. Lee, "High-temperature tensile and high cycle fatigue properties of inconel 625 alloy manufactured by laser powder bed fusion," *Additive Manufacturing*, vol. 35, p. 101377, Oct. 2020, doi: 10.1016/j.addma.2020.101377.