ESRF	**Experiment title:** Microstructure changes in Additively Manufactured IN718 during stress relief heat treatment	**Experiment number**: MA-5212
Beamline: ID22	**Date of experiment**: from: 20.09.2022 to: 23.09.2022	**Date of report**: 19.12.2022
Shifts: 9	**Local contact(s)**: Andrew FITCH	*Received at ESRF:*

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Report:

Inconel 718 must undergo heat treatments in order to achieve optimal microstructure and properties through precipitation hardening. When produced by additive manufacturing (AM), this alloy does not respond to the established thermal routines in the same way that the conventional material does, and there is still no consensus on which routines yield the best results [1]. This experiment aimed at providing a fundamental understanding of the heat treatment response by monitoring the microstructural evolution during all stages of the routine, including heating and cooling, by means of in-situ heat treatments at the beamline. The results emerging from this experiment build upon and establish a direct conversation with the findings by Gallmeyer et al. (recommendation of a short solution treatment at 1020 °C [1]) and those by Kermanpur et al. (suggesting a shorter aging for the AM material [2]).

Samples were produced with two different scanning strategies (67°-rotational scan and 0/67°-alternate scan) leading to different initial textures, and were brought to the facility in the as-built condition, after being machined into cylinders with a diameter of 1 mm and a length of 5 mm to fit inside the ceramic capillary (sample holder) provided at the beamline. On site, the heat treatments consisted of a solution treatment for 1 hour plus a single aging step for 5 hours. Two different temperatures were used for the solution treatment: 1020 °C and 1080 °C, and all aging treatments were performed at 760 °C. A third solution temperature – 1050 °C – was tested also for 1 hour, but without following the aging step. The heat treatments were carried out with an induction cylindrical furnace supplied with an argon flow. The furnace could be controlled from the cabin via command, and it was calibrated before the experiment using test samples of the same material and geometry. The temperature at the sample is 20 °C lower than the temperature set on the furnace control. The samples were attached to the end of a ceramic capillary, which was placed horizontally, with the end containing the sample inside the furnace. The beam hit the samples at about ¾ of the sample length (about ½ of the sample length was placed inside the ceramic capillary). The experimental setup is shown in Figure 1.

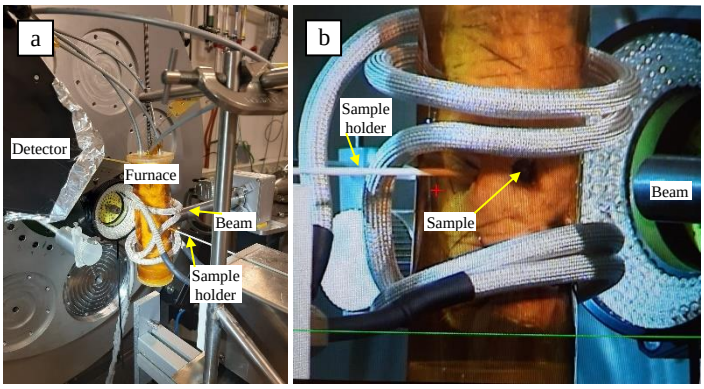

Figure 1: a. *Experimental setup inside the hutch.* ***b.*** *Detail of the insertion of the sample in the furnace.*

Originally, the Perkin Elmer area detector was planned to be used throughout the experiment, with some measurements doubled using the high-resolution detector for the most relevant moments of the schedules. However, there appeared to be an interference effect on the area detector caused by the magnetic field formed around the coil of the furnace. Hence, the utilization of the Perkin Elmer detector needed to be ruled out, leading to the use of the high-resolution detector throughout the whole experiment instead. Data was acquired continuously throughout every heating or cooling stage, as well as throughout the isothermal stage of the solution treatment, and every 5 minutes throughout the isothermal hold in the aging treatment. The energy of the beam was set to 50 keV, the wavelength was 0.24802 Å, the beam had a square cross-section of 1 mm x 1 mm, and the effective 2θ interval of acquisition was 0.0° to 26.0°.

The acquired data is still being processed, but a preview of some preliminary results using the software PDIndexer [3] is shown in Figure 2. In Figure 2a, it is observed that the diffraction profile changes as a function of increasing temperature for the solution treatment up to 1020 °C. As the temperature increases (from the black curve towards the green curve), the peaks shift to the left, which is a consequence of thermal expansion of the lattice. Most importantly, the asymmetry in the peaks gradually vanishes, which indicates the dissolution of Nb/Mo segregation. The segregation of these heavy atoms at interdendritic zones during processing causes a local increase in the lattice parameter of the matrix, and thus a corresponding contribution to an accessory peak slightly shifted towards smaller scattering angles, adjacent to the main γ peaks, resulting in this asymmetry effect [4]. The disappearance of the asymmetry indicates that chemical homogenization is already largely achieved even before the dwelling temperature is reached. Figure 2b shows the diffractograms during the dwelling temperature in the aging step at 760 °C after the solution treatment at 1020 °C for the same sample. As time progresses, the curve goes from black towards blue, in approximately uniform intervals of about 30 minutes. New shoulders are developed adjacently to the main γ peaks (around 6.8° and 7.8°), which indicates the precipitation of the key strengthening phase γ" (Ni_3Nb, BCT, space group: I4/mmm), as γ" diffraction peaks are located in the vicinity of the γ peaks. From this plot, one can conclude that after about 3.5 hours – corresponding to the light pink curve–, the heat treatment enters a regime of diminishing aging rates, since the difference between one curve and the next becomes increasingly smaller. Additionally, δ (Ni_3Nb, orthorhombic, space group: Pmmn) precipitation occurred in the samples solutionized at 1020 °C, which is evidenced by the peaks at about 70.5° and 71.5°. In these samples, these δ peaks appeared during the solution treatment itself. δ formation did not happen in the samples solutionized at 1080 °C.

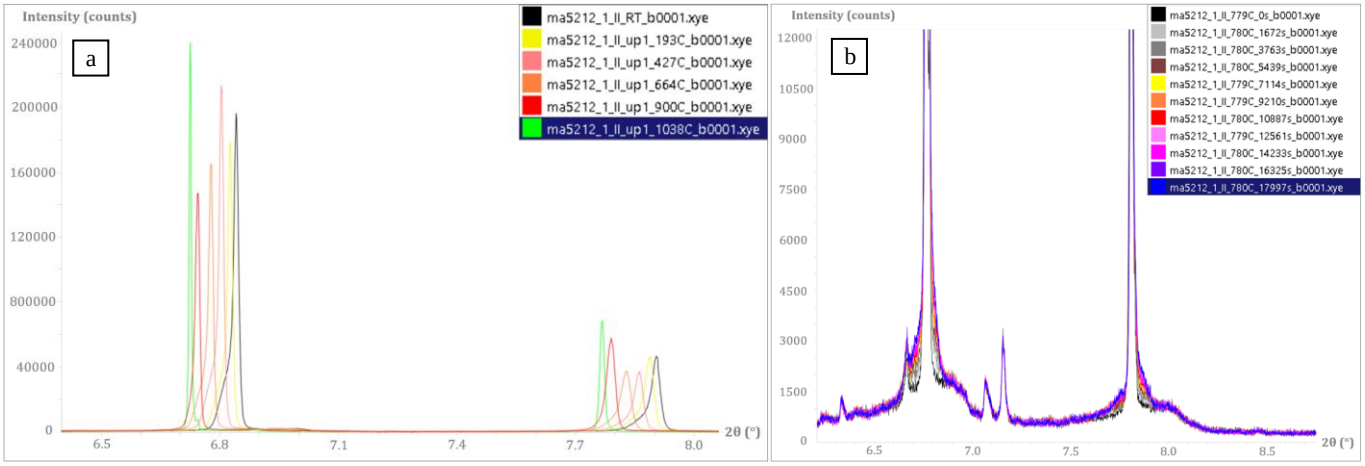

Figure 2: a. *Evolution of the diffraction profile during heating up to 1020 °C using the sample produced by 67°-rotational scanning strategy. The black curve represents the room temperature material, and the temperature increases towards the green curve (the last curve before the isothermal hold at 1020 °C).* ***b.*** *Evolution of the diffraction profile during the aging isothermal hold at 760 °C after solutionizing at 1020 °C (67°-rotational scanning strategy). The black curve corresponds to the material at the start of the isothermal hold, and the spectra progress toward the blue curve with increasing holding times.*

Besides the interference of the furnace on the 2D detector, another difficulty to be aware of is mild oxidation of the sample (despite the Argon flow). This experiment was designed to be included in Bruno Ferrari's PhD thesis, and its outcome is being incorporated into a manuscript (to be complemented with post-mortem electron microscopy on the same samples used in this experiment).

References:

[1] Gallmeyer, T. G. et al., Additive Manufacturing, Volume 31, 2020.
[2] Kermanpur, A. et al., Materials Characterization, Volume 191, 2022.
[3] Seto, Y. et al., The Review of High Pressure Science and Technology, Volume 20, 2010.
[4] Schröder, J. et al., Journal of Materials Science, Volume 57, 2022.