

Microstructure and mechanical behavior of the 3D printed Inconel 718: In-situ TEM study

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Development of additive manufacturing (AM) process such as selective laser melting (SLM) enables direct 3D printing of metal alloys. SLM method uses a scanning laser to melt and fuse powder bed layer-by-layer to create bulk materials with complicated three-dimensional structures. This laser processing accompanies rapid heating and cooling, leading to the formation of non-equilibrium phases with fine-scale microstructure. The mechanical properties of the materials produced by SLM can be different from that by conventional fabrication routes such as casting and forging, which can be advantage or disadvantage depending on the applications.[1]

Among various 3D-printable alloys, Inconel 718 (In718), a nickel-based superalloy, shows excellent high temperature mechanical properties along with the commendable corrosion resistance. However, the mechanical property of the 3D-printed In718 may not satisfy the requirement of the highly demanding applications because of its microstructure characteristic associated with SLM process. Here, post-production heat-treatment can be a key to improve the mechanical properties. In718 with Ni, Cr, and Fe as the major composition, can achieve very high strength with the face-centered γ' ($\text{Ni}_3(\text{Al}, \text{Ti})$) and body-centered tetragonal γ'' (Ni_3Nb) that precipitate around 650°C. Although heat treatment processes of the alloys produced by conventional techniques have been widely studied, the SLM-produced alloys may undergo different structural evolution because of the difference in the initial structure. In this study, we conducted in-situ transmission electron microscopy (TEM) experiments to directly observe the microstructure evolution of In718 during heat treatment. Additionally, mechanical tests were conducted in-situ inside TEM to quantify the mechanical property and correlate it with the microstructure. [1-3]

The in-situ tensile test was carried out with Nanofactory scanning tunneling microscopy (STM)-TEM holder with a 3D-piezo manipulator. An atomic force microscopy (AFM) cantilever with the tip modified to hook the dog-bone-shaped specimen was used. This set up enables us to image the specimen in the real time during the tension and to the fracture as shown in Fig 1. Focused ion beam (FIB) was used to prepare dog-bone shape sample with the trapezoidal cross section for the mechanical testing. The small micron-sized specimen was lifted out from the bulk alloy. Thus, the measured mechanical property represents the specific area of the sample which provides the dependency on the local microstructure. This information, which is averaged out in the mechanical test using a bulk sample, provides rich information that can be used to optimize the SLM-produced alloy by controlling the processing parameter to create favorable microstructures.

The in-situ heating experiment was conducted using Gatan heating holder (Model 652). The thin TEM samples were prepared by FIB and annealed inside the TEM at three different temperatures (600, 700 and 800°C) with 10 minutes of holding time to analyze the nucleation and the growth of precipitates (γ' , γ'') as shown in Fig 2. Significant stress contrast was observed at the initial stage

of the heat treatment (a,b), and the stress was slowly relaxed as the precipitates began to form (c). Once the precipitates were developed to a diameter of ~20 nm, they seized to grow further (d,e). At this point the stress was fully relaxed. The needle-like δ -phase precipitates were also observed. Our observation from the in-situ heating experiments of the 3D-printed In718 gives insights to predicting the microstructure evolution of the alloy during the heat treatment and contribute to designing the process to improve the mechanical property.

Our in-situ TEM observation quantifies the mechanical properties of local region of the sample and its correlation with the microstructure becomes possible, which can play a pivotal role in designing SLM process to manufacture in bulk with desired characteristics.

References:

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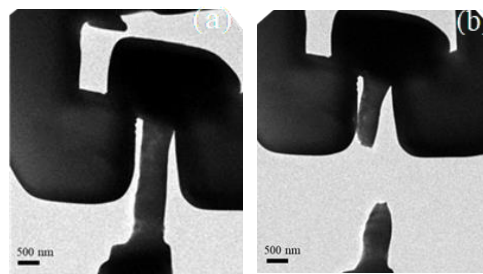


Figure 1. TEM micrograph of IN 718 mechanical testing (a) before testing (b) after the testing.

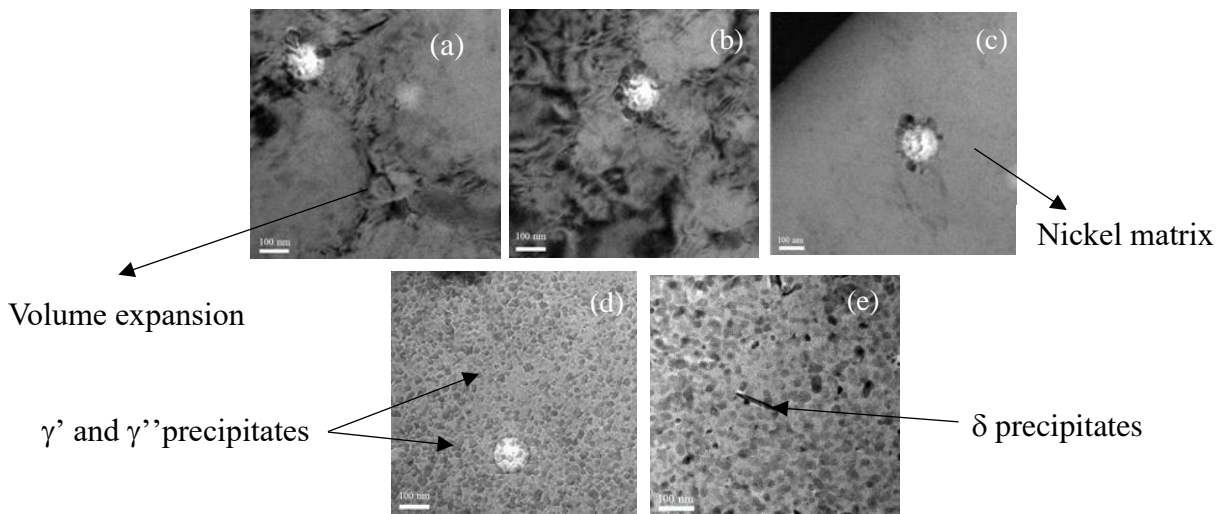


Figure 2. Sequence of precipitate formation at 700 °C with a holding time of 10 minutes.