TEM investigation of Hastelloy X produced by additive manufacturing

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The powder bed based additive manufacturing technique of Selective Laser Melting (SLM) is used to produce a so-called As-Built (AB) sample of a Hastelloy X, which is a Nickel base superalloy with high oxidation resistance and high strength at elevated temperatures. This AB sample was then post processed at 1155°C for 3h yielding a Hot Isostatic Pressed (HIP) sample. The concentration used in this work is $Ni_{47}Cr_{22}Fe_{18}Mo_9X_4$ (at.%) with X some minor additional fractions.

The AB sample consists of large grains divided by dislocation walls into cells with disorientations of a few degrees, as determined by Automated Crystal Orientation Mapping (ACOM), and a lower density of dislocations inside. In the HIP sample the dislocation walls are transformed into low-angle boundaries and the cells into sub-grains. In both AB and HIP samples cells and sub-grains have elongated shapes with lengths of tens of microns and diameters of about 1 micron. The images in Figure 1 are taken along the long axis, while in Figure 2 the long axis is in the plane of the image (here for the AB sample). This texture results from the grain growth along the heat gradient existing during SLM production of the AB sample. Electron diffraction patterns of the matrix reveal a disordered FCC structure with lattice parameter of 3.591 Å, with energy dispersive X-ray (EDX) spectroscopy confirming the majority elements.

In both AB and HIP samples some elements are segregated into two types of round precipitates with size of about 30-70 nm formed during solidification and after several thermal cycles during production. In the AB case these precipitates are mostly located on the dislocation cell walls, while in the HIP sample some are also found inside the sub-grains, as seen in the High Angle Annular Dark Field Scanning Transmission Electron Microscopy (HAADF-STEM) image shown in Figure 3 (a). From EDX it is concluded that the bright precipitates show an enrichment in the lighter elements such as Al, Ti and O while the dark ones correspond with enrichment in Mo. In the HIP sample an additional phase compared to the AB sample is observed on some grain boundaries, as shown in Figure 3 (b). The grains of this additional phase are about 100-200 nm wide and are Cr and C enriched. Acquired electron diffraction patterns of the phase together with energy dispersive X-ray spectroscopy reveals a $Cr_{23}C_6$ structure.

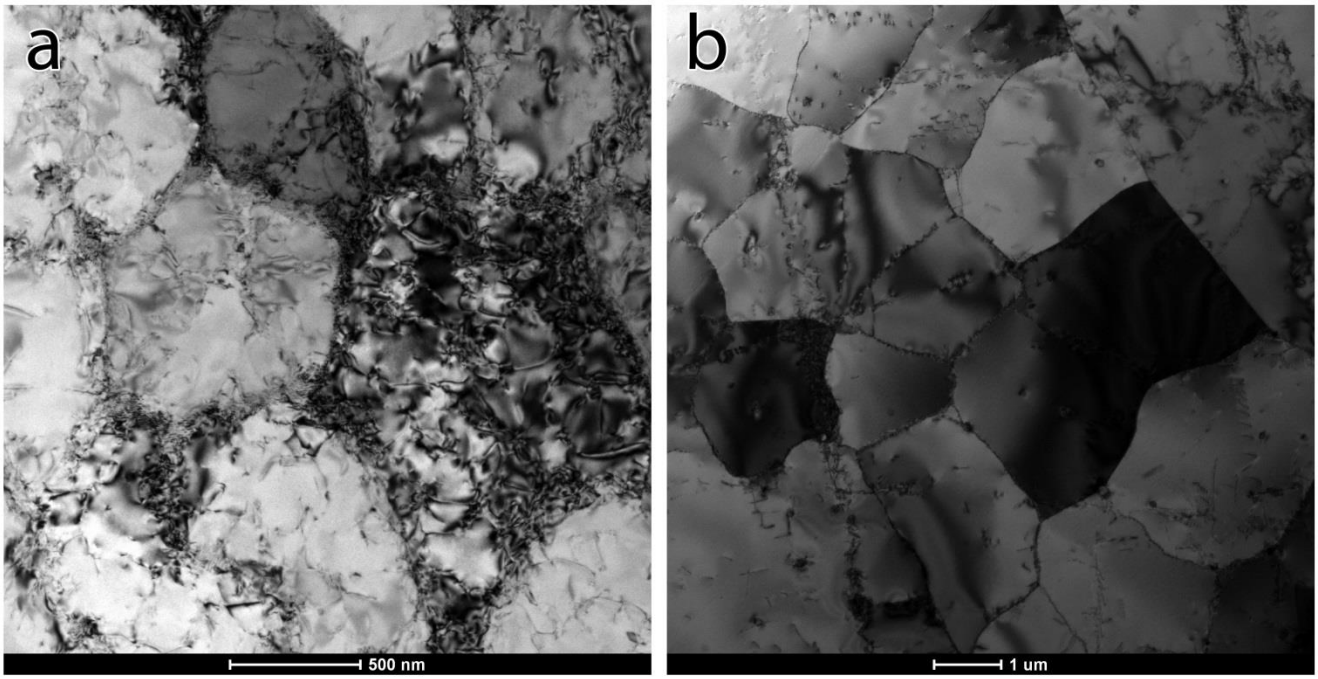

Figure 1 BF-TEM image of (a) dislocation cells in AB and (b) sub-grains in HIP sample.

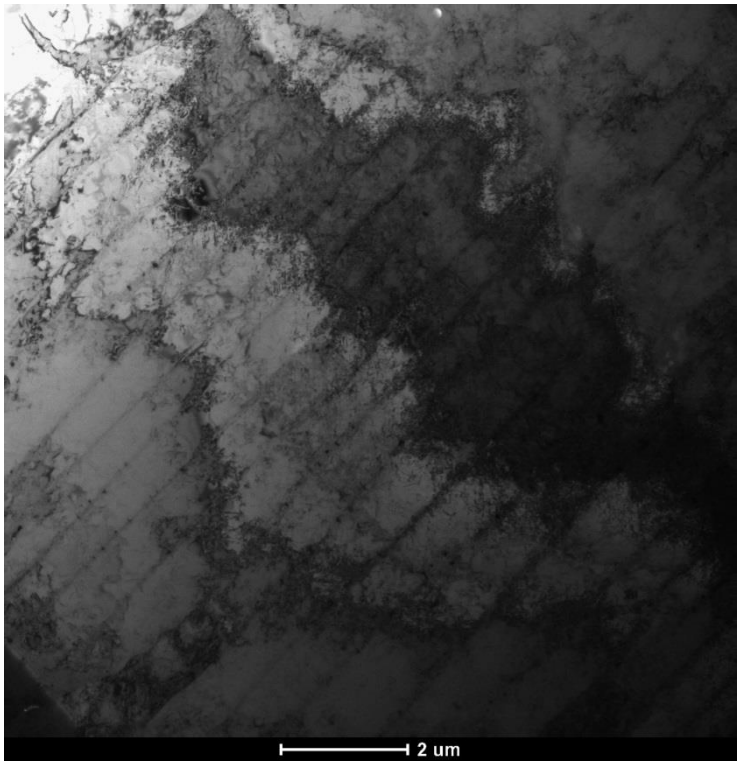

Figure 2 BF-TEM image of columnar dislocation cells in AB sample.

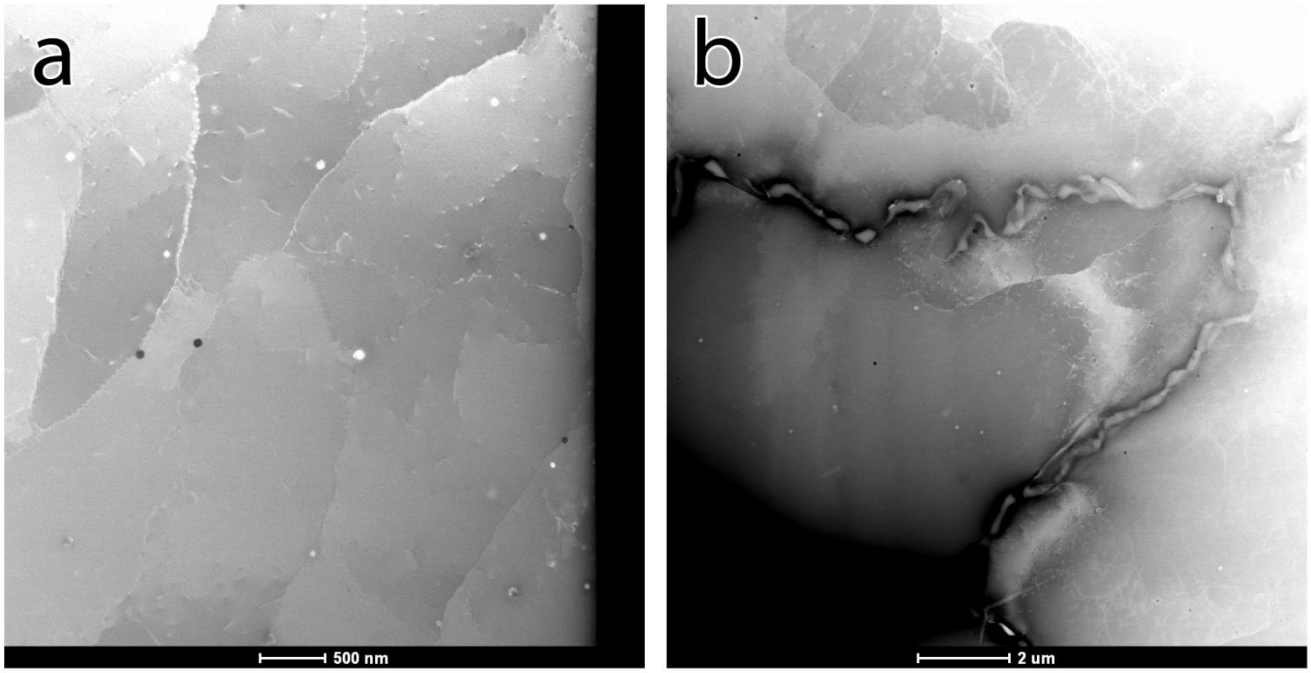

Figure 3 HAADF-STEM images of (a) dark and bright precipitates and (b) $Cr_{23}C_6$ phase, both in the HIP sample.

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