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MS-4-P-1984 The 3D imaging of 718Plus superalloy microstructure by FIB-SEM tomography

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Allvac 718Plus is a high strength, corrosion resistant nickel-chromium-iron based superalloy used for applications in power generation, aeronautics and aerospace [1]. Its typical chemical composition is: Ni-18Cr-10Fe-9Co-5.1(Nb+Ta)-1W-2.7Mo-0.7Ti-1.5Al-0.03C (wt%). The 718Plus microstructure consists of a γ matrix (Ni-based solid solution) with ordered face centred cubic γ' $Ni_3(Al,Ti)$-type phase and some orthorhombic δ (Ni_3Nb)-type particles precipitated at the grain boundaries. The primary strengthening mechanism for this alloy is a precipitation hardening, therefore properly sized and distributed precipitates are critical for good alloy performance. The aim of this study was to describe 718Plus microstructure and apply FIB-SEM tomography to 3D imaging and metrology of the precipitates.
Microstructural investigation and phase chemical compositions were performed using SEM-NEON CrossBeam 40EsB (Zeiss) and TEM – a probe Cs corrected Titan3 G2 60-300 (FEI) equipped with ChemiSTEM, a Super-X EDS detector with 4 windowless diodes and an X-FEG high brightness electron gun. The TEM study was conducted on the lamella prepared by FIB. FIB-SEM tomography is based on a serial slicing technique employing a FIB-SEM dual beam workstation [2]. The NEON 40EsB CrossBeam with Ga-ion beam was used to perform a precise in-situ milling. Consequently, the acquired stack of images was transformed directly into a 3D data volume with a voxel resolution of 12×12×12 nm. Repeated removal of layers as thin as a 12 nm allowed exploring a total volume of 10.7 x 7.2 x 7.7 µm.
Fig. 1 shows 718Plus microstructure observed using SEM-EsB detector (Z-contrast). Needle shaped particles, identified as δ-phase by EDS analysis, were observed at grain boundaries and occasionally at twin boundaries. Distribution of chemical elements between γ, γ' and δ phases was determined using STEM-EDS (ChemiSTEM) (Fig.1c). It can be seen that precipitates with round-to-blocky morphology are randomly dispersed within the matrix. Some primary Nb-rich MC and Ti-rich M(C,N) particles were also observed. Fig. 2 shows 3D visualization of the shape and distribution of δ phase at γ grain boundary in 718Plus. The results achieved confirm the ability of FIB-SEM tomography to reconstruct 3D structures with dimensions in the range of 100 nm or even smaller. Such 3D reconstructions can serve as a basis for quantitative analysis of complex structures in a nanoscale. Further investigation of 718Plus superalloy by analytical TEM is in progress.

References

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[2] A. Kruk, A. Czyrska-Filemonowicz; Archives of Metallurgy and Materials, 58(2013)387

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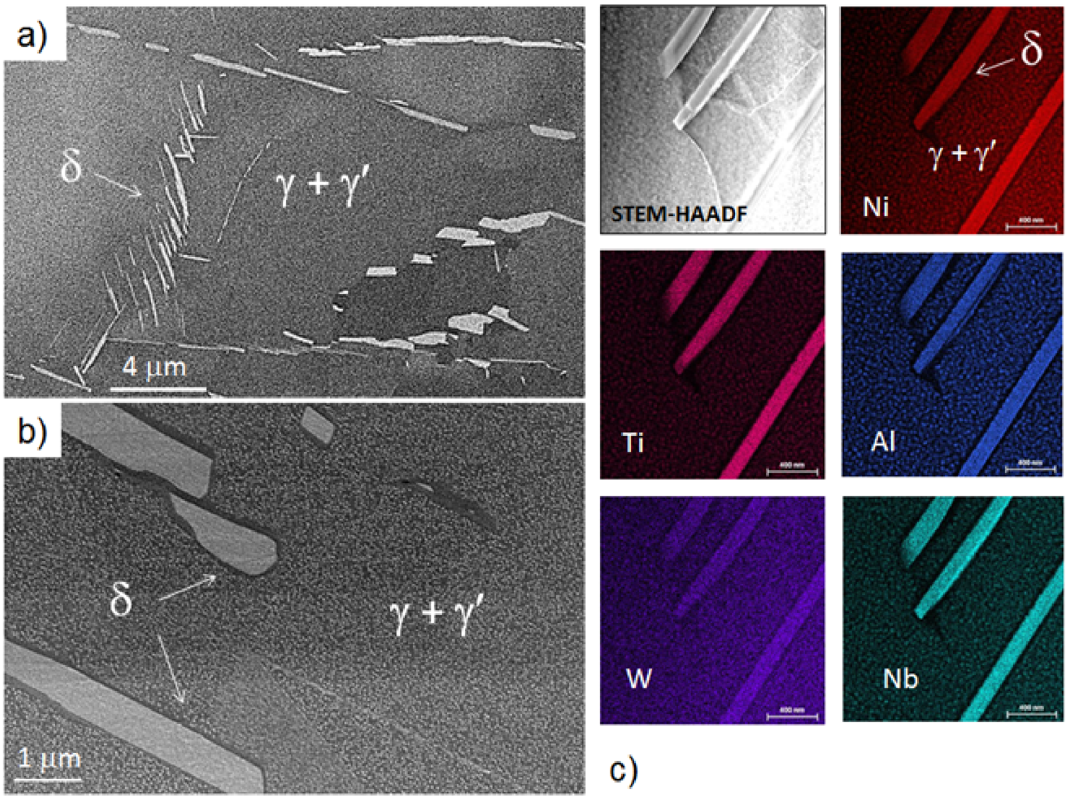

Fig. 1: Microstructure and chemical composition of 718Plus superalloy: a, b) microstructure of non-etched sample observed in Z-contrast (SEM – EsB detector), c) chemical element distribution maps obtained by STEM-EDS.

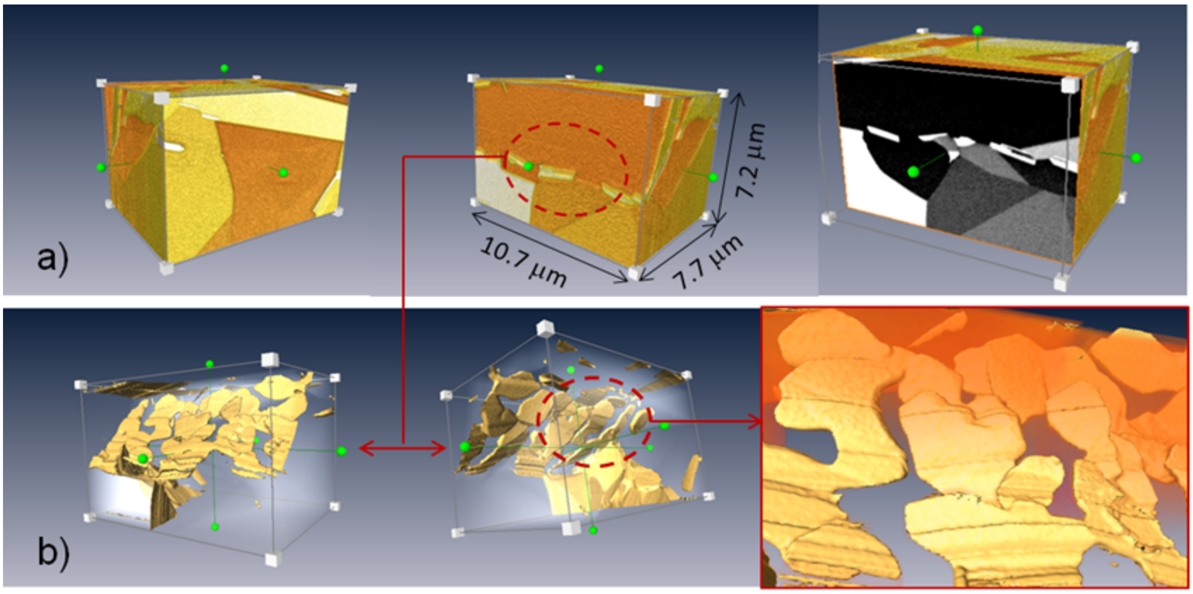

Fig. 2: Three-dimensional visualization of tomographic reconstructed volume of 718Plus superalloy by FIB-SEM tomography: a) reconstructed volume 10.7 x 7.2 x 7.7 μm at different angle of view, b) shape and distribution of δ phase precipitated at the γ grain boundary.