Application of different imaging techniques for characterization and visualization of micro and nanostructural elements in Allvac 718Plus superalloy

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Allvac 718Plus (718Plus) is a newly developed Ni-based superalloy, high strength, corrosion resistant and has improved higher temperature performance compared to the Inconel 718 superalloy. The 718Plus superalloy is used for applications in power generation, aeronautics and aerospace. The combination of a different chemistry and adequate heat treatments causes precipitation mainly the γ′ and δ or η- phases. The 718Plus microstructure consists of a γ matrix with spherical precipitates of ordered face centred cubic γ'-Ni₃(Al,Ti) type phase, some orthorhombic δ-Ni₃Nb and hexagonal η-Ni₃Ti, η*-Ni₆AlNb or Ni₆(Al,Ti)Nb particles precipitated at the grain boundaries.

The aim of this study was characterization of phases and 3D visualization of microstructural element morphology using several microscopy techniques, mainly HRSTEM-HAADF, STEM-EDX spectrometry. The present work concerns also the application of TEM and FIB-SEM electron tomography for imaging and evaluation of qualitative and quantitative information about microstructure of materials. Performed analysis in atomic level (HRSTEM-HAADF) of selected precipitates in the 718Plus superalloy revealed the complex nature of these precipitates, as shown in Fig.1. X-ray spectroscopic imaging (STEM-EDX), enables the mapping of local concentrations of selected chemical elements. This technique was used for qualitative and quantitative evaluation of η- phase precipitates with resolution about of 0.2 nm. STEM-EDX maps of selected elements forming η particles in 718Plus superalloy are presented in Fig.2.

The electron tomography is currently a relatively new technique in materials science that uses a TEM or FIB-SEM to 3D imaging of microstructural elements in various engineering materials. The TEM electron tomography technique enables obtaining 3D model of the investigated object(s) from the multiple 2D projection images, acquired over a range of viewing directions (±75°). The TEM investigation was performed on FIB lamella by a Cs-corrected Titan™ G2 60-300 with EDX ChemiSTEM™ technology, which allowed to achieve high X-ray signal over a large tilt angle of sample and collect a tomographic series of 2D elemental maps at the angular range from -40° to +60° (with a step of 4°) of tilting the sample. The STEM-EDX imaging by ChemiSTEM™ provides new opportunities for 3D visualization of changes in the concentrations of particular chemical elements in nanoparticles or analysis of the microstructure of thin foils. Tomographic reconstruction of a tilt series images was performed using SIRT method, which allowed visualizing the three-dimensional distribution of selected elements (Al, Cr) in the analysed volume. Application of elemental maps imaging (STEM-EDX) acquired during tilting the sample was used for 3D imaging of coherent γ′ precipitates in 718Plus superalloy (Fig.3a).

FIB-SEM tomography is based on a serial slicing technique using a FIB-SEM dual beam workstation. Dual-beam SEM enables the acquisition of serial images with small (few nanometers) and reproducible spacing between the single imaging planes - because no mechanical stage tilting is necessary between the FIB milling and the electron beam SEM imaging steps. Meso-scale FIB-SEM tomography technique, was used for characterization of spatial distribution and metrology of the η- phases in 718Plus superalloys with resolution of 12 nm. SEM backscattered electrons (BSE) image of 718Plus superalloy presents different shapes of the η- phase precipitated in the γ matrix. Fig. 3b shows three-dimensional visualization of FIB-SEM tomographic reconstructed η- phase. Fig. 3b shows at different angle of view a morphology of selected η particles precipitated at the γ grain boundary. Platelets, occasionally as a lamellar structure at grain boundaries and in the grains were observed in the microstructure of the 718Plus. The η- phase precipitates at the γ grain boundaries had much higher thickness (270 nm) than the thickness of the η- phase plates (56 nm) in lamellar precipitates inside γ grains (Fig. 3b). Application of HRSTEM-HAADF imaging and tomographic techniques (STEM-EDX, FIB-SEM) allowed for visualization the precipitates in 718Plus superalloy, their quantitative assessment, spatial distribution and morphology.

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Fig. 1. Result of analysis of selected precipitates in the 718Plus superalloy at atomic level. a) STEM-HAADF image of the microstructural elements in the analyzed region. b) Experimental and simulated HRSTEM-HAADF images for platelet precipitate observed in the microstructure of 718Plus superalloy.

Fig. 2. STEM-EDX maps of selected elements of η particles in 718Plus superalloy.

Fig. 3. Result of STEM-EDX and FIB-SEM tomography techniques application for visualization of precipitates in 718Plus superalloy. a) 3D visualization of γ' using STEM-EDX tomography. b) Reconstruction using FIB-SEM tomography and 3D visualization of complex particle and η- phase in 718Plus superalloy.