Quantitative chemical analysis of the γ- and γ'-phases in nickel base superalloy PWA1483

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Macroscopic physical properties are correlated to the microstructure in nickel base superalloys. In these materials mechanical properties are strongly influenced by the presence of sub-micrometer sized γ'-phase precipitates. In this presentation the detailed phase compositions of single crystalline superalloy PWA1483 (nominal composition $Ni_{60}Cr_{14}Co_9Al_8Ti_5Ta_1W_1Mo_1$) is elucidated. PWA1483 forms globular to cubical, 0.2 to 0.6 µm sized γ'-phases surrounded by 0.1 to 0.3 µm broad γ-matrix channels. The chemical compositions of the highly alloyed solid solutions determine the crystal structure and the γ and γ'-phase fractions formed during heat treatment [1]. The two phases γ (Cr,Co-rich) and γ' (Al,Ti-rich) are described in the literature as Ni (space group: Fm$\bar{3}$m) and as Ni_3Al (space group: Pm$\bar{3}$m) crystal structure, however, chemical and structural details are frequently not reported.

In transition metals X-ray absorption and overlapping X-ray lines are challenges for a quantitative analysis [2]. SEM-EDX and STEM-EDX were applied for a quantitative EDX analysis. The nominal composition of PWA1483 was measured and confirmed by SEM-EDX with an accuracy of up to 1.5 at.% using multiple scan areas of 0.25 mm². STEM-EDX spectra were acquired on the same sample on a Jeol 2200FS (EDX-detector: Jeol Centurio SSD) at Siemens and a Fei Tecnai F20 (EDX-detector: EDAX Apollo XLTW SSD) at the USTEM. Only FIB-prepared lamellae were used mounted on a Cu grid.

Tabulated k-factors of the standardless Cliff-Lorimer method were found to be inaccurate up to 20% [2]. Quantitative analysis of the nominal composition yielded modified k-factors using STEM-EDX mappings acquired over an 8 µm² wide area of the TEM lamellas. After recalibration of the k-factors the chemical composition of the individual phases were determined yielding $Ni_{54}Cr_{24}Co_{11}Al_5Ti_2Ta_1W_2Mo_1$ for the γ-matrix and $Ni_{67}Cr_4Co_5Al_{12}Ti_8Ta_2W_1Mo_1$ for the γ'-phases. Both TEM systems at USTEM and at Siemens yielded the same chemical composition for the two phases within the error bars, which proves the significance of EDX analysis for such complex materials. The chemical composition of the individual phases together with the nominal composition yielded the volume fraction of the γ-matrix and was calculated as of 48 ±5%. This result was confirmed by SEM secondary electron greyscale images.

The presented analysis of the γ and γ'-phases by STEM-EDX provides a unique and quick method for analyzing the chemical composition of the individual phases with high accuracy. From this data the phase fractions can be determined if the nominal composition of the superalloy was known.

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