Fast automated 3D EDXS Mapping

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Many sophisticated analysing methods were established and combined in the past in order to characterize solids in an appropriate way [1, 2]. The demand for these highly developed investigation techniques arise because optimizing the functionality of materials often depends on a precise control of the size, shape, crystal structure and composition of the material being synthesized. Automated serial sectioning in combination with physical analytics achieved by using a script-able Dual Beam -Focused Ion Beam system (DB-FIB) equipped with an energy dispersive X-ray spectrometer (EDXS) is only one of the possible characterization opportunities mentioned above [3].

We present an excellent example how powerful EDXS-mapping in three dimensions combined with ion channelling contrast formation and the advantages of the block lift out technique [4] provides a better understanding of material properties. Additionally we compare previously achieved 3D data sets using a Si(Li)-detector (much longer acquisition time) to recent fast measurements carried out with a high count rate treatable silicon drift detector (SDD). Today, there are several commonly used alloys like nickel-based superalloys, which consumes up to 30% of the weight in advanced aerospace engines. Some examples of alloys in use are Udimet 720, Waspaloy and Alloy 718, the latter is the most widely used due to its relatively low costs and good formability. Recently, a new nickel-based alloy was developed (Allvac 718 PlusTM) by the company ATI Allvac. More details of the extended mechanical properties of this alloy can be found at Bergstrom [5]. Its properties are attributed to the combined effects of chemistry, heat treatment and microstructure. Especially for hot forged gas turbine disks, the influence of δ-phase is important. However, it is well known that the start of δ-phase precipitation strongly depends on the experimental conditions. Thus, an understanding of the phase stability with time and temperature is essential in order to tailor the microstructure of forged turbine blades for high temperature applications.

Among several investigative tools and techniques like electron back scatter diffraction (EBSD), back scatter electron imaging (BSE) (Figure 1b) and transmission electron microscopy the 3D micro-structural characterization was carried out on the FEI Nanolab Nova200 dualbeam focused ion beam (DB-FIB - FEI company, Eindhoven, The Netherlands) equipped with a formerly installed energy dispersive Si(Li) X-ray detector (10 mm²) system from EDAX (Mahwah, USA) and now working with a Bruker-AXS system (Berlin, Germany, 10 mm^2 SDD, Quantax400, Figure 2a). Final data visualization was performed using the Amira 3.1 software (Mercury Computer Systems SA). The serial–sectioning thickness was selected to be 500 nm using the EDAX-system (Figure 1a) and was reduced to 100 nm with the Bruker-system (six times faster) giving a better resolution in z-direction. Due to the lack of an EBSD system in the DB-FIB additional ion beam imaging giving a

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good channelling contrast (Figure 2b) was performed and therefore rotating, translating and tilting the sample between the cross-section milling position and the SEM imaging position was required. Figure 1a depicts the 3D model, reconstructed from the first (500nm slice thickness) experiment. δ-phase that appear needle-like have a very small angle to the cross section. Hence, a complete plate-like reconstruction is hardly possible. But reducing the slice thickness and therefore increasing the possibility for a more accurate reconstruction procedure is due to a fast acquisition process affordable and helpful to overcome these problems. However, the FIB-SEM investigations confirm the plate-like shape of the δ-phase, which agrees well with previous 2D experiments and data from literature.

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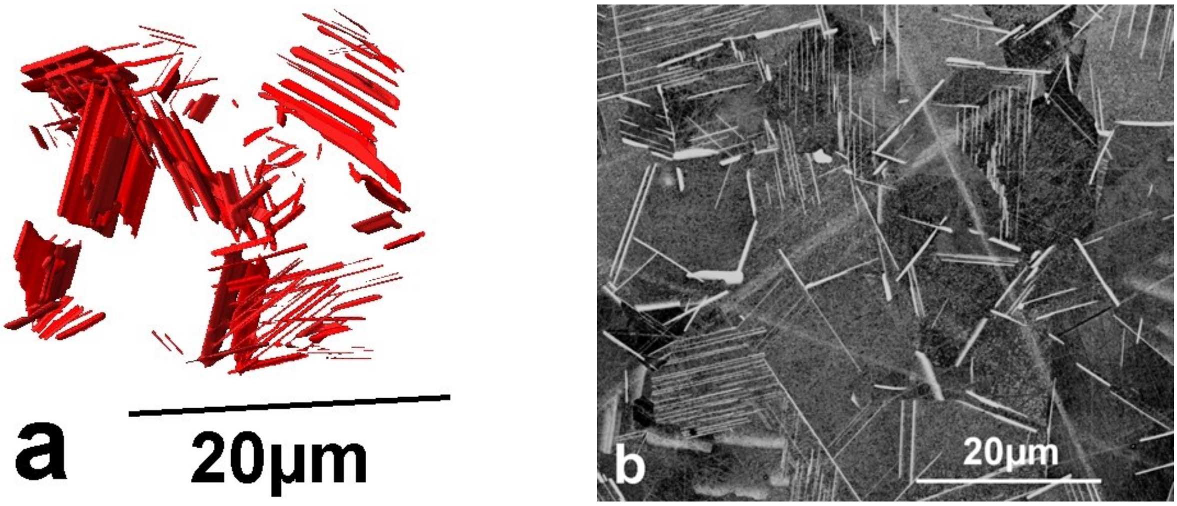

Figure 1. (a) Reconstruction of the δ-phase, (b) BSE-image of δ-phase (white needles)

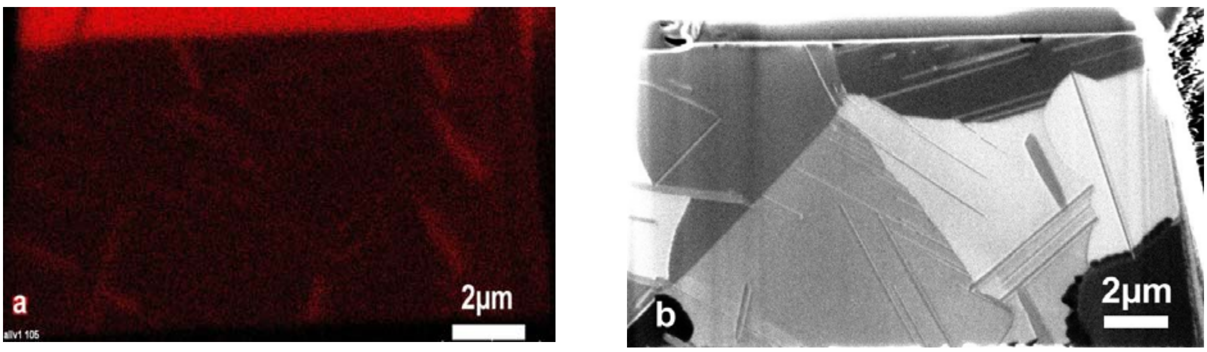

Figure 2. (a) fast (3 min) EDXS-map of the Niobium enriched δ-phase, (b) ion channelling contrast SE-image for grain reconstruction

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