The 3D microstructure of RR1000 nickel-base superalloy: a FIB-SEM dual-beam approach

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Nickel-base superalloys are aerospace materials that exhibit exceptional mechanical properties and corrosion resistance at very high temperatures. RR1000 superalloy is used in discs in gas turbine engines, where temperatures reach in excess of 650°C with high mechanical stresses [1]. Study of the microstructure at the micron and sub-micron level has conventionally been undertaken using scanning electron microscope images, often meaning the underlying 3D microstructure can be inferred only with additional knowledge. In this work, different methods for image acquisition, image processing and 3D reconstruction are investigated to determine the feasibility and value of imaging nickel-base superalloys in 3D.

RR1000 exhibits Ni_3Al γ' precipitates within an FCC γ matrix and is produced via powder metallurgy using argon gas atomisation. This gives γ' precipitates that vary in size from 5nm to 1µm depending on the heat treatment, cooling rate applied and the selected precipitation ageing heat treatment. For this investigation, a cooling rate, notably slower than those used in disc forgings, was chosen to yield large precipitates that show dendritic-like morphology at a scale suitable for testing the 3D techniques developed in this work [2].

An FEI Helios 600 dual-beam workstation, combining a scanning electron microscope (SEM) with a focused ion beam (FIB), was used to apply a serial sectioning approach to interrogate the 3D microstructure. This was achieved using the FIB to remove slices of the material and the SEM to capture images of the revealed surfaces [3]. A site 15µm x 20µm in area was selected for investigation, and a layer of platinum deposited to protect it during the preparation stage and to minimise “curtaining” during the serial sectioning. Trenches are milled out around the site of interest to stop redeposition of material and to allow an unobstructed view of the imaging face.

Various detectors may be used while imaging with the electron beam during the serial sectioning. The simplest signal in principle for detecting the contrast between the γ and γ' is from backscattered (BS) electrons, as this gives contrast directly dependent on the elemental composition of the material. However, due to the geometry of the imaging conditions present in the FIB-SEM, there is a significant loss of signal as some BS electrons do not reach the in-column Thru-the-Lens Detector (TLD). To counter this, a magnetic immersion lens helps focus type-II secondary electrons (SEs) up the column to the TLD. Type-II secondary electrons are generated by interaction of the BS electrons with the sample and therefore this signal contains compositional contrast.

In total, a volume of 9.5µm x 8.7µm x 11.3µm was interrogated through the serial sectioning using a slice thickness of 50 nm. Figure 1 is an image from the data set and shows the typical microstructure for this alloy, composed of γ' precipitates in a γ matrix. In addition, pores and minority phases (carbides/oxides) are evident. For the reconstruction of the microstructural elements of the material, specialist software Avizo® Fire was used. Due to the complex nature of the variations of the intensities across the imaging face, a simple global thresholding could not be used, and the data set had to be segmented into the different features manually. The software is then used to generate a 3D surface of the γ' precipitates by linking the precipitate features in sequential slices, see figure 2.

Volumetric data is then extracted for 3D analysis of the reconstruction. One example of the use of this data is to plot the surface area of the precipitates versus the precipitate volume, as shown in figure 2. This shows that the smaller precipitates follow the growth expected for a spherical precipitate with A α $V^{2/3}$, with the larger precipitates exhibiting more dendritic growth, with the surface area increasing at a faster rate.

We have demonstrated how a FIB-SEM system can be used to reconstruct 3D morphology and distribution of precipitates in RR1000 nickel-base superalloy. The wealth of data provided from the FIB-SEM serial sectioning technique is enabling a better understanding of the microstructural development of RR1000 and its relationship with the underlying mechanical properties [4].

References

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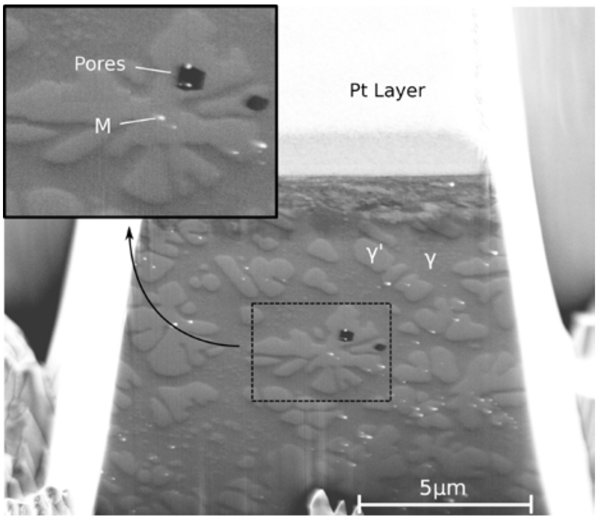

Figure 1. *An unprocessed secondary electron image, at 4kV, from the serial sectioning of RR1000 alloy. The inset shows a magnified part of the image. The “flowers” are γ', the white dots are the minority phases, labelled M, are likely to be carbides or oxides and the black regions are pores.*

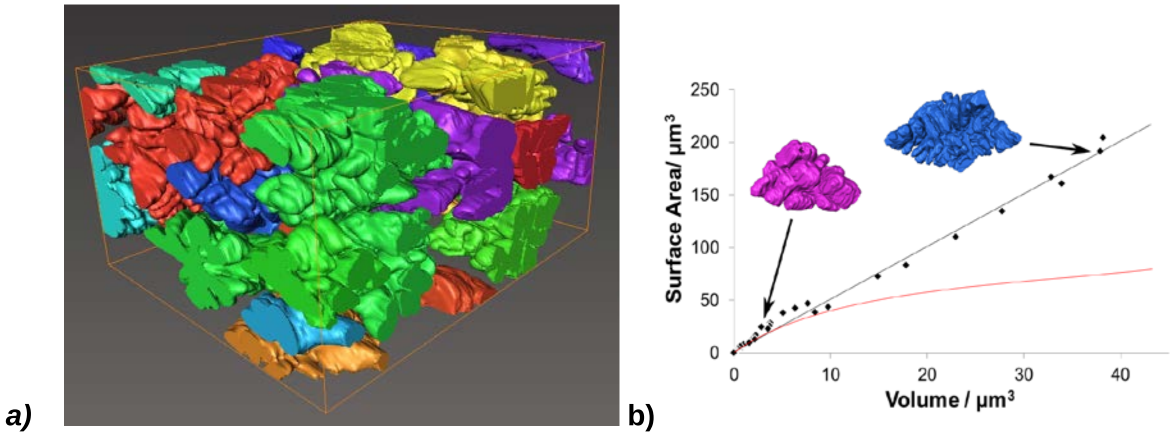

Figure 2. a) *Reconstruction of the γ' microstructure of RR1000. The bounding bounding box measures 9.5μm x 8.7μm x 11.3μm, the precipitates are coloured to distinguish individual precipitates.* ***b)*** *Graph showing the precipitate volume plotted against the surface area (data points). The standard of a sphere of increasing radius is used as a reference (red line). Two precipitates are shown (not to scale), and their corresponding data points are indicated by arrows.*