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An Atom Probe Tomography study of site preference and partitioning in a nickel-based superalloy



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ABSTRACT

Atom Probe Tomography (APT) has been utilised for an in-depth examination of the commercial polycrystalline Ni-based superalloy RR1000, assessing compositions of the primary, secondary and tertiary γ' phases. Clear differences in the phase chemistries are noted, particularly for the tertiary γ' to which much of the alloy strength is attributed. Trace amounts of Hf are found to segregate strongly to the primary and secondary γ' phases, but also exhibit an extended diffusion profile across the γ - γ' interface up to 80 nm wide. Ti, Al and Mo demonstrate similar, yet not as pronounced diffusion profiles, indicating assumed phase chemistries may not be representative of those regions adjacent to the γ - γ' interface. Within γ' , unique element site-occupancy preferences for this alloy were identified. Finally, the grain boundary chemistry across a γ - γ' interface and that of an intragranular boride were analysed, identifying the latter as a mixed M_5B_3 boride rich in Mo and Cr. These demonstrate further the depth of information on Ni-alloys accessible by APT, while the overall implications of results in comparison with other in-service/model alloys are also discussed.

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1. Introduction

Where do the atoms in an alloy reside? This question is of fundamental importance to the field of materials science and the discipline of physical metallurgy from which it emerged. Unfortunately, for structural alloys – which are usually based upon one of Al, Fe, Ni or Ti – truly unequivocal answers are not yet available. One explanation for this is complexity. These alloys are usually multicomponent in nature with many alloying elements added. Strengthening phases are nearly always present, and these can be ordered, so that a site preference arises. Moreover, precipitate distributions are often multimodal. Alloys are mainly polycrystalline, so that segregation and/or grain boundary precipitation can arise. To what extent can a modern high-resolution characterisation techniques answer this critical question?

The nickel-based superalloys are a class of structural alloy for which the above description of complexity is particularly appropriate. First, these are multicomponent alloys with ten or more

alloying elements present; arguably they represent some of the most complex alloys designed by man. An ordered strengthening phase, based upon the $L1_2$ crystal structure, plays an important role in the alloy microstructure; thus preferential partitioning of elements is expected not only to this phase but also to the sub-lattice atomic sites within it. Elements such as B and C are added which are known to segregate to grain boundaries [1,2], however the extent of this segregation and whether secondary phases such as carbides and borides form as a result remains unclear. Finally, elements such as Hf and Zr are sometimes added in trace quantities for a variety of intended benefits including gettering sulphur at grain boundaries [3] and to prevent oxide scale spallation [4]; however the mechanisms for these are still not well understood. Characterising chemical distribution at the atomic-scale is therefore a key step in developing an understanding of the role of individual elements, their interactions in the microstructure and ultimately the performance of the material. Increasingly demanding applications for components made from such materials, most notably for jet propulsion [5] and power generation [6], drives continued development of these alloys to perform in extreme conditions. Detailed characterisation of these materials is essential to enable such progress.

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In this study, the modern high-strength polycrystalline nickel-based superalloy RR1000, an alloy developed by Rolls-Royce plc, is subjected to detailed high resolution analysis using atom probe tomography (APT). Due to the complex chemistry of this alloy, a technique is needed which is capable of resolving the numerous components with equal sensitivity, whilst providing the spatial resolution needed to map microstructures of the differently sized phases. APT complements more conventional analyses such as electron microscopy [7–9] and X-ray/neutron scattering [10,11]. Examples of insights provided by APT into these types of alloys have been previously reviewed [12,13], while focussed studies include composition-dependent interactions between solutes [14], evolution of lattice spacings and precipitates during ageing [15,16], informing dislocation-precipitation interaction simulations [17], behaviours of individual solute elements including Re [18–20], Pt-group metals [21–24], Nb [25], segregation at grain boundaries [1,26,27] and oxidation around crack tips [28,29]. For the newly discovered class of Co-based alloys, APT has also been used effectively to investigate chemical partitioning and the nature of certain microstructural features [30–32].

RR1000 is in use in current generation aero engine components such as high-pressure turbine discs. Despite the extensive use of APT to look at various model and commercially-used superalloys, few detailed studies have been published on RR1000 or similar alloys using this technique. Therefore, we examine in detail element partitioning behaviour and inter-relationships in the γ' phases of this alloy, and assess how specific elements segregate to grain boundaries. The latter is becoming increasingly important in disc alloys required to operate for longer periods at elevated temperatures in modern flight paths, leading to rising concerns over creep and low cycle fatigue characteristics [33].

2. Experimental

Samples of RR1000 were provided by Rolls-Royce plc. This alloy was manufactured using powder metallurgy, and following hot isostatic pressing and recrystallization during extrusion it was isothermally forged to shape. After a solution heat treatment at 1120 °C (sub-solvus) for 4 h, it was air-cooled then precipitation aged at 760 °C for 16 h to yield a fine-grained (~5 μm average dia.) microstructure.

For optical and electron microscopy, samples were cut from the as-received material using an ISOMET™ 5000 precision saw. These were successively ground using a series of SiC grit papers before being diamond polished to a final 1 μm surface roughness finish, then colloidal silica polished up to 0.04 μm finish. Samples prepared in this manner were analysed using a combination of Zeiss NVision 40, Zeiss Merlin and JEOL 6500 Scanning Electron Microscopes (SEM), varying the voltage between 5 and 15 kV to suit the signal-to-noise requirements of different high resolution secondary and backscattered electron micrographs. Energy Dispersive X-ray Spectroscopy (EDX) was performed on selected areas of the samples in a Zeiss Merlin SEM equipped with two Oxford Instruments detectors and Aztec software. Separate samples for optical/SEM studies were etched in 10 vol% orthophosphoric acid in H₂O at 10 V for 5–10 s to preferentially dissolve the γ matrix.

For APT analysis, matchsticks (0.5 × 0.5 × 15 mm) cut from the alloy were initially electropolished in a 10 vol% perchloric acid/acetic acid solution, then finished in a solution of 2 vol% perchloric acid in butoxyethanol to suitably sharp needle-shaped samples. To examine grain boundaries and site-specific regions, bulk samples mechanically polished as described above were utilised to prepare needles by the lift-out procedure [34] using a Zeiss NVision dual-beam FIB, taking care to minimize Ga⁺ beam-damage with a final 2 kV/30 pA polish. APT data was collected using a Cameca LEAP™

3000X HR instrument in the Department of Materials, University of Oxford. The majority of samples were run in voltage mode at a specimen temperature of 50 K, with 200 kHz pulses at a pulse fraction of 20%. The small subset of potentially more fragile samples containing grain boundaries were run in laser mode, with pulse energies of 0.2–0.6 nJ.

3. Results

3.1. SEM/EDX

Fig. 1a–b shows SEM secondary electron micrographs of the etched RR1000 microstructure at two different magnifications. Together these figures reveal a trimodal size distribution of γ' precipitates and their relative location in the microstructure. The largest (>500 nm) primary γ' precipitates are the least prevalent, located along grain boundaries, as indicated in Fig. 1a). The secondary γ' (~250 nm in size) are the most abundant by volume fraction, while the smallest tertiary γ' (≤ 50 nm) precipitates appear uniformly distributed but are most easily visible at grain boundaries.

Also highlighted in Fig. 1a is the presence of two intragranular precipitates. Fig. 1c) isolates these at high magnification, along with a series of EDX maps identifying them as a Mo-Cr rich boride and a smaller Ta-Hf rich carbide. These were not observed within every grain. Some borides also appeared enveloped within a γ' shell. Site-specific APT analysis, enabled by FIB lift-out specimen preparation, was undertaken to more precisely examine the composition of the larger intragranular borides, as discussed below.

Smaller elongated borides (approximately 1/10th the size of intragranular borides) were also noted infrequently along grain boundaries; most boundaries examined however were boride free. Furthermore etching of samples to expose phases reduces EDX resolution through creation of surface topographical features. These can partly shield the signal making smaller particles challenging to identify. However the composition of the smaller borides appeared very similar to that of the intragranular one shown in Fig. 1, enriched in Mo and Cr.

3.2. Atom Probe Tomography

APT analysis across numerous samples, comprising 35 million ions in total, provided a measurement of the overall alloy composition, in Table 1, with sufficient sensitivity to detect all minor alloying elements. The measured concentrations are in close agreement with specified values for the major elemental additions, with the exception of Zr (0.1 at.% nominal). Trace levels of Fe (0.4 at.%) and Si (0.06 at.%) were also identified. The primary γ' phases are retained from sub-solvus processing, whilst the secondary and tertiary γ' phases nucleate during different stages in the cooling [35]. The primary γ' particles, which are the largest, incoherent, and located at the grain boundaries form during alloy solidification, prior to any thermal-mechanical processing. The alloy is then aged below the solvus temperature in order to preserve these, where they act to pin the grain boundaries, inhibiting grain coarsening to create a finer grain size distribution. The secondary and tertiary γ' particles however form during subsequent thermal ageing treatments. An in-depth modelling study on RR1000 [36] has correlated thermal treatments to different size distributions of secondary and tertiary γ' , and their relative contributions to the overall strength of the alloy.

Hence, the matrix composition differs relatively for each stage at which these phases respectively form, leading to slightly different γ' chemistries. The chemistries of tertiary γ' precipitates are the most distinct of the three, containing significantly more Mo and Al

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