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Nucleation of recrystallisation in castings of single crystal Ni-based superalloys

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ABSTRACT

Recrystallisation in single crystal Ni-based superalloys during solution heat treatment results in a significant cost to the investment casting industry. In this paper two sources of surface nucleation have been identified in the alloy CMSX-4®. Firstly, Electron Backscattered Diffraction (EBSD) has revealed micro-grains of γ′, between 2 and 30 μm diameter in the layer of surface eutectic found in the upper part of the casting. These have high angle boundaries with respect to the bulk single crystal and a fraction coarsen during solution heat treatment. Secondly, in the lower regions where surface eutectic does not form, locally deformed regions, 5–20 μm deep, form where the metal adheres to the mould. The local strain causes misorientations up to ≈20° with respect the bulk single crystal, and after heat treatment these regions develop into small grains of similar low-angle misorientations. However, they also form twins to produce further grains which have mobile high-angle boundaries with respect to the bulk single crystal. Experiments have shown that micro-grains at the surface grow to cause full recrystallisation where there is sufficient strain in the bulk material, and by removing these surface defects, recrystallisation can be completely mitigated. Etching of the cast surface is demonstrated to be an effective method of achieving this.

1. Introduction

The occurrence of recrystallisation in single crystal superalloy components during the solutioning heat treatment has long posed a significant mystery: how do you nucleate mobile high angle boundaries in a single-crystal material undergoing very moderate strains? In the past investigations have focussed on the strain necessary to drive the migration of boundaries [1–3], but avoid this central issue. The nucleation of recrystallisation is normally associated with the migration of existing boundaries experiencing a significant local difference in dislocation density on either side [4–6]. In the absence of grain boundaries, severe deformation of 20–25% by, for example, indentation [3,7–10] or compression [11–13] is required to trigger recrystallisation, often magnified by the presence of strain concentrators such as carbides [14]. Recrystallisation has also been observed during thermo-mechanical fatigue, nucleating at the intersection of deformation twin bands [15,16]. None of these possibilities arises in cast single crystal superalloys where potential strains are low and carbides are absent by design. Nucleation is always at, or very close to, the cast surface [17], but is unlikely to occur spontaneously. This work, for the first time, provides evidence of viable nuclei in the surface layers of single crystal castings and demonstrates that under suitable conditions of very moderate strain these can develop into sizable grains equipped with mobile high angle boundaries.

2. Background

Single crystal superalloy components are vulnerable to grain boundaries as the elements strengthening grain boundaries are removed to give a more effective solution heat treatment. Boundary misorientation angles above 10° lead to a catastrophic drop in creep

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rupture life [18]. Grain boundaries act as crack initiation and propagation sites during creep deformation and lead to a significant reduction in the creep rupture life following recrystallisation [18–22]. Fatigue crack nucleation and propagation rates are also higher in recrystallised samples, [23,24]. Hence, any sign of recrystallsation, or indeed the inability to examine a casting through, for example, the formation of surface scale [25], results in the rejection of the components and a significant increase in cost. Recrystallisation also constrains component design, since complex geometries are more prone to developing higher local casting strains [1,2,13]. Furthermore, work by Hill [26] has shown that the susceptibility to recrystallisation depends on the alloy composition; the critical strain for recrystallisation was nearly halved for some recrystallisation-prone alloys, which thus become uneconomic despite other good properties.

Recrystallisation is driven by the strains induced from the different thermal response of the mould and the casting. It is a complex function of the material expansion coefficients and modulae, the component design and the casting process parameters. Quantifying the threshold strain to propagate recrystallisation following nucleation is not straightforward as the dislocation density is not a monotonic function of strain and the dislocation configuration and stored energy depends on the deformation temperature [1,27]. However, observations of the deformation in castings prior to heat treatment show that the dislocation configurations most closely resemble those produced at high temperatures, demonstrating that the majority of the strain is induced during the very early stages of cooling after solidification [1]. By applying the solution heat treatment to samples deformed to known strains at these temperatures the critical strain to fuel recrystallisation has been identified at 1–2% [1,2] in agreement with previous work [3]. Modelling of the strains induced during cooling has demonstrated that in specific locations the metal can experience sufficient stress to induce the plastic deformation necessary for recrystallisation, and experimental castings of various geometries have validated this modelling [1,2]. One strategy to control recrystallisation is to reduce deformation during cooling by changing the ceramic mould and core materials, reducing the strength of the mould, but increasing the risk of failure or distortion. An alternative approach is to eliminate or reduce the nucleation of recrystallsation.

3. Experimental

3.1. Materials

CMSX 4,[1] a second-generation superalloy (composition given in Table 1) was cast parallel to [001] under conditions consistent with normal foundry production practice at Rolls-Royce plc. The bars were ≈12.5 mm in diameter and ≈200 mm in length, Fig. 1. Post-processing steps that can induce strain, such as grit-blasting, were not employed. Several bars received a two-stage heat treatment at Bodycote plc. The first stage was a ramped solution heat treatment with a final step of ≈1315 °C for 6 h; the second stage primary age, was at 1140 °C for 6 h. Other bars remained in the as-cast state.

3.2. Sample preparation

The bars were sectioned lengthways, parallel to [001], with silicon carbide (SiC) blades, at the positions indicated in Fig. 1. The samples were mounted in conducting Bakelite, and ground with SiC paper from 1200 grit to 4000 grit. Final polishing was with a 3 μm diamond suspension (≈5 min), followed by a dilute 0.04 μm colloidal silica suspension (≈3 min).

A sample for transmission electron microscopy (TEM) was extracted from a specific location using the focussed-ion beam (FIB) technique in the Helios Nanolab 600 dual-beam field emission gun scanning electron microscope (FEGSEM). Sample preparation was done using standard techniques (technical details included in supplementary material) but the final stages of thinning at the lowest beam currents were not possible, consequently, some ion-beam damage was observed in the final microstructure.

3.3. Characterisation

Electron imaging with backscattered (BSE) and secondary electrons (SE), and energy dispersive x-ray spectroscopy (EDX) were performed on the CamScan MX2600 or JEOL 5800LV microscopes. Generally, imaging was done at 15 kV with a working distance of 10–15 mm, and the EDX data was acquired using Inca software from Oxford Instruments at 25 kV with either 35 mm (CamScan MX2600) or 10 mm (JEOL 5800LV) working distance.

The electron backscattered diffraction (EBSD) data was acquired at 25 kV with 30 mm working distance using the CamScan MX2600 FEGSEM and the CHANNEL 5 HKL software from Oxford Instruments. Orientation data is presented as inverse pole figures (IPF) and local misorientation maps (kernel method, 11 × 11 pixel matrix) acquired using 0.4–0.6 μm step size. Only misorientations ≥5° were considered, as misorientations of ≈3° were measured between adjacent dendrites.

Electron probe microanalysis (EPMA) was done using the Cameca SX100 microscope. The wavelength dispersive spectrometers used and the element spectral lines used are given in the supplementary information. The data was acquired at 20 kV with a 1 μm spot size and 40 nA current, and the acquisition time was 30 s except for Hf and Re, where 60 s was used due to weaker signals.

Transmission Electron Microscopy on the sample prepared by FIB was performed on a JEOL 200CX microscope operating at 200 kV.

3.4. Mechanical testing and annealing

Half-cylinder samples ≈12 mm in length were cut slowly with SiC blades from the as-cast bars to be deformed on an Instron 8800 servo-hydraulic low cycle fatigue machine. Fully heat-treated RR3010 was used as platen material. Samples were compressed at 0.2% min^{-1} along the [001] axis at room temperature to induce 3% plastic strain. To reduce friction with the platens carbon sheets and Cu grease were used.

To remove ≈100 μm of the surface, the deformed samples were electrolytically etched in a solution of 7 vol% perchloric acid in ethanol. This was done in an ice-bath at 15 V, and uniform etching was achieved at a rate of 5–10 μm min^{-1}. Although etching reduced nucleation from the damage caused by the platen, the depth was not sufficient to eliminate it altogether.

The etched and un-etched samples were sealed in fused silica tubes backfilled with Ar, and were given the standard solution heat treatment, with one sample interrupted after 30 min at 1315°. For examination, the polished surfaces were immersed in Kalling's etchant (10 gms $CuCl_2$ + 50 ml HCl + 50 ml ethanol) for 1–2 min to reveal the grain structure in the optical microscope.

4. Observations from the cast surface

The CMSX 4 bars are shown in the as-cast condition, Fig. 1a, and after the standard heat treatment, Fig. 1b. Almost 65% of the upper part of the as-cast bar has a shiny-silver colour (upper mould

[1] CMSX-4 is a registered Trade mark of the Cannon Muskegon Company.