Acta Materialia 124 (2017) 489–500

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

On the role of boron on improving ductility in a new polycrystalline superalloy

Paraskevas Kontis [a], Enrique Alabort [b], Daniel Barba [b], David M. Collins [a], Angus J. Wilkinson [a], Roger C. Reed [a, *]

[a] Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, UK
[b] Department of Engineering Science, University of Oxford, Parks Road, Oxford OX1 3PJ, UK

ARTICLE INFO

Article history:
Received 10 May 2016
Received in revised form
27 August 2016
Accepted 3 November 2016
Available online 22 November 2016

Keywords:
Nickel-based superalloys
Grain boundaries
Strain mapping
In-situ

ABSTRACT

The role of boron in promoting ductility at high temperature in a prototype nickel-based superalloy designed for industrial gas turbines is studied. Both a boron-containing and boron-free variant are tested in tension at 750 °C, with further *in-situ* tests carried out using scanning electron microscopy (SEM), to clarify the mechanism of ductility improvement. The improvement in ductility is observed to be greater at the lowest investigated strain rate, where the grain boundary character plays a significant role on the mechanical properties; no ductility improvement was observed at the highest investigated strain rate. The *in-situ* tests were also performed at 750 °C and revealed directly the greater susceptibility of the grain boundary morphology in the boron-free case to fracture and – in the boron-containing case – the mechanism of ductility enhancement. The findings are supported further by high-resolution electron backscattered diffraction (HR-EBSD) strain mapping which confirms that the distribution of elastic strain and geometrically necessary dislocation (GND) content are influenced markedly by boron addition. The mechanism through which boron indirectly enhances the mechanical properties at elevated temperatures is discussed.

1. Introduction

The ductility of the nickel-based superalloys can be exploited for high temperature applications [1]. But what factors influence it? When in polycrystalline form, intergranular fracture is common particularly under the high temperature conditions of greatest technological relevance; it follows that the effects occurring at the grain boundaries must play a significant role in promoting – or limiting – ductility [2,3]. But more fundamental studies are needed to elucidate the physical mechanisms which are at play.

One difficulty is that traditional approaches rely upon post-mortem studies for the deduction of microstructure/property relationships in such materials. For example, emphasis is often placed on fracture surfaces or dislocation structures examined at ambient conditions, when in fact it is the effects occurring at temperature and with the load applied which are clearly of the greatest relevance. *In-situ* studies circumvent these difficulties and are therefore of significant value in the search for a greater appreciation of the phenomena which control properties [4,5].

In this paper, a study is made of the behaviour of a prototype polycrystalline superalloy designed recently for power generation applications. A boron-free and a boron-containing variant are tested in tension at different strain rates and at elevated temperatures. *In-situ* tensile experiments are also carried out in a scanning electron microscope at elevated temperatures to examine the factors promoting ductility. High-resolution electron backscatter diffraction (HR-EBSD) measurements are made at interrupted intervals of tensile deformation, thus enabling a cross-correlation based strain mapping method to be undertaken. In this way, insights are provided into the role played by boron in promoting ductility in these alloys.

2. Background

As will become apparent later in this paper, a significant role is played by boron in the serration of grain boundaries in this superalloy. Therefore attention is paid here to the prior literature on

---

* Corresponding author.
E-mail address: roger.reed@eng.ox.ac.uk (R.C. Reed).

http://dx.doi.org/10.1016/j.actamat.2016.11.009

this topic. The serration of grain boundaries has been reported to have an influence on mechanical properties in polycrystalline nickel-based superalloys by impeding grain crack propagation [6–8]. In particular, voids and cavities forming at serrated grain boundaries are often assumed not to easily interlink and this prevents the formation of a continuous crack path [9,10]. Moreover, serrated grain boundaries invoke longer diffusion paths along grain boundaries [11]. Environmental effects at elevated temperatures are thus retarded. Whilst some theories exist on the exact formation mechanism, a consensus on the formation of grain boundary serrations remains absent. Furthermore, the definition of whether a grain boundary is serrated is currently rather arbitrary; for example, a boundary may be considered serrated when its 'amplitude' is larger than 0.5 μm [6].

Different theories have been developed that attempt to rationalise the formation of serrated grain boundaries in nickel superalloys. Serrated grain boundaries are often strongly associated with heterogeneous nucleation of γ' in their vicinity [10,12]. In this case, a serration results from the pinning of a grain boundary at a primary γ' precipitate in conjunction with its movement between the primary γ' particles [13]. The grain boundary migration continues until γ' particles form homogeneously throughout the grain – thus forming a physical barrier that prevents further migration. Moreover, it is well established that the occurrence of a serration depends strongly on the cooling rate from the solution treatment temperature through the γ' precipitation zone [11,13–15]. Low cooling rates result in serrated grain boundaries, since there is then sufficient time for grain boundaries to grow. Rapid cooling leads to smaller heterogeneously formed γ' particles; consequently planar grain boundaries form [11,14]. The serration amplitude and wavelength increase as cooling rate decreases and solution temperature increases [16,17]. For formation of serrated grain boundaries, it is also often thought that the solution heat treatment temperature should be well above the solvus of the major grain boundary carbides, e.g. $M_{23}C_6$, [10]. That way the carbides are dissolved and the grain boundary can move in the absence of obstacles. By contrast, different studies have postulated that serrated grain boundaries form in the presence of $M_{23}C_6$ carbides at the grain boundaries, whereas planar grain boundaries occur in their absence [18].

It has also been suggested [6] that alloy composition plays a role – for instance by providing a driving force for grain boundary serrations from discontinuous chromium and carbon segregation along the grain boundaries. Inhomogenous segregation of carbon and chromium are expected to provide varying magnitudes of lattice distortion along the grain boundaries, while this is not observed when the segregation is continuous. This has been rationalised using a calculation by McLean [19], where the lattice strain energy for carbon, given as 48 kJ mol^{-1}, was reported to be far greater than chromium, at 1.2 kJ mol^{-1} [6]. As a consequence, grain boundary serrations are not expected in cases of low carbon content in solid solution. If the accumulation of strain energy results in a distortion of the grain boundary itself, one can infer that serrated grain boundaries may occur due to discontinuous segregation. It was suggested that the non-uniform distortional lattice strain along the grain boundary acts as the driving force for the onset of grain boundary serrations. Contrary to this, in a study of a ternary Ni-xCr-0.1C model alloy, chromium was found to increase the serration amplitude [20].

In addition to carbon and chromium, boron has also been found to segregate to serrated grain boundaries in Astroloy, as confirmed by atom probe studies [21]. Here, serrated grain boundaries were observed in a boron-containing alloy, but planar grain boundaries occur in the absence of boron.

3. Experimental procedures

3.1. Materials

The prototype nickel-based polycrystalline superalloy STAL15-CC investigated in this study has a composition shown in Table 1 and grain size of ~750 μm. The chemical analysis was conducted in an independent laboratory where the carbon content was measured using a LECO CS444 analyser and for the other elements the inductively coupled plasma OES (ICP-OES) method was utilised. To investigate the effect of boron, test-bars with no boron (boron free – BF) and boron-containing (BC – 0.05 at.% B) content were produced by conventional casting (CC). Castings in the form of tapered rods were prepared at Doncasters Precision Castings Ltd., using casting stock melted by Ross & Catherall (Sheffield, United Kingdom). After conventional casting, a hot isostatic press (HIP) was used to consolidate the as-cast bars, in order to eliminate microporosity and improve mechanical properties. The HIP process was performed at 1195 °C for 5 h under 175 MPa pressure. The process of HIP was followed by a stage of primary ageing at 1120 °C for 4 h and a subsequent second stage of ageing at 845 °C for 24 h, both followed by air-cooling. Note that the same heat treatment conditions were applied to both alloys.

3.2. Mechanical tests at elevated temperature

Tensile tests were performed using an Instron 8800 electro-thermal mechanical testing (ETMT) device at 750 °C under displacement control with speed of 10 μm s^{-1} and 0.1 μm s^{-1}, corresponding to strain rates, $\dot{\varepsilon}$, of approximately 2×10^{-3} s^{-1} and 2×10^{-5} s^{-1}, respectively. Specimens were machined from fully heat treated bars by electro-discharge machining (EDM). The gauge volume of the specimens was designed as 5 mm length × 2 mm width × 1 mm thick. Specimens were mechanically ground with abrasive media prior to tests to remove the oxidised layer present from the EDM. Heating was achieved by passing direct current (DC) through the gauge length of the specimen controlled by a thermocouple which was spot-welded to the centre of the gauge length. The displacement between the grips was measured with the use of a linear variable differential transducer (LVDT). Following tensile failure of the boron-free and boron-containing samples, their fracture surfaces were characterised using an optical Alicona microscope.

In-situ tensile tests were performed at 750 °C and under displacement control with speed of 0.1 μm s^{-1}, corresponding to strain rate, $\dot{\varepsilon}$, of approximately 2×10^{-5} s^{-1} to failure. A Kammrath-Weiss *in-situ* module, equipped with a resistance heater, was mounted on a Zeiss EVO SEM with a LaB_6 filament. Double dogbone specimens were machined from fully heat-treated bars by EDM with a gauge volume of 5 mm length × 2 mm width × 1 mm thick. Once again the surfaces were mechanically ground with abrasive media to a 1 μm finish. Secondary electron images were obtained at 750 °C with surfaces etched using Kalling's solution to reveal grain boundaries. Two thermocouples were used to monitor temperature during the tests; one was fitted between the sample and the heater and the other was spot-welded onto the sample surface. In order to

Table 1
Summary of chemical compositions of the different STAL15-CC variants investigated in this work (at.%).

Alloy	B	C	Co	Cr	Mo	W	Al	Ta	Hf
Boron-free	<0.005	0.44	5.50	16.45	0.61	1.26	10.00	2.41	0.02
Boron-containing	0.05	0.47	5.50	16.55	0.59	1.26	10.09	2.40	0.02