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The effect of chromium and cobalt segregation at dislocations on nickel-based superalloys

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ABSTRACT

The segregation of solutes at dislocations in a polycrystalline and a single crystal nickel-based superalloy is studied. Our observations confirm the often assumed but yet unproven diffusion along dislocations via pipe diffusion. Direct observation and quantitative, near-atomic scale segregation of chromium and cobalt at dislocations within γ' precipitates and at interfacial dislocations leading to the partial or complete dissolution of γ' precipitates at elevated temperatures is presented. Our results allow us to elucidate the physical mechanism by which pipe diffusion initiates the undesirable dissolution of γ' precipitates.

Superalloys derive their outstanding strength from $L1_2$-ordered γ' precipitates [1]. However, the presence of dislocations enhances the kinetics of their dissolution during exposure to elevated temperatures and results in either recrystallisation or directional coarsening of γ' known as rafting [2,3]. Both have an undesirable impact on the performance of superalloys [4,5]. Albeit often studied, the mechanism underpinning this dissolution remains elusive [6–9].

It is often assumed that the presence of a high density of crystalline line defects, i.e. dislocations, in the γ matrix have the potential to enhance transport of elements by so-called pipe diffusion [8,10]. As a consequence, local chemical inhomogeneities are promoted, which lead to the destabilisation and dissolution of the γ' precipitates. Such fundamental insights into the stability of the γ' precipitates and their dissolution mechanisms are required for the development of accurate models to predict the lifetime of critical components such as turbine blades [11,12]. Nevertheless, accurate quantitative chemical studies to near-atomic scale are lacking.

In search for proof on the true role of dislocations in this process, we subjected a commercial polycrystalline superalloy, and a single-crystal nickel-based superalloy, to microstructural degradation and observed recrystallisation and rafting, respectively. Regions of the microstructure with high dislocation density revealed by controlled electron channeling contrast imaging (cECCI) were analysed at the near-atomic scale by atom probe tomography (APT) in search for segregation of solutes to dislocations within the deformed γ/γ' microstructure and rationalisation of the dissolution of γ' particles via pipe diffusion.

In the case of recrystallisation the polycrystalline superalloy IN792 was studied. Specimens from fully heat treated bars of IN792 with chemical composition Ni-13.9Cr-8.8Co-1.1Mo-1.3 W-7.6Al-4.9Ti-1.3Ta-0.4C-0.1B-0.012Zr (at.%), were ground and polished with abrasive media to a 1 μm finish and were isothermally and statically exposed in air at 750 °C for 50 h. In the case of rafting the single crystal superalloy MC2 was investigated. Creep specimens were machined from fully heat treated bars of MC2 with chemical composition Ni-9.3Cr-5.1Co-1.3Mo-2.6 W-11.2Al-1.9Ti-2.0Ta (at.%) and close to perfect [001] crystallographic orientation. Tensile creep tests were performed under constant load (initial applied stress = 120 MPa) and under thermal cycling conditions, using the following cycle: 15 min at 1050 °C + 1 min at 1105 °C + 15 min at 1050 °C + 1 min at 1160 °C. After completion of the thermal cycling creep tests, the specimens were cooled down to 800 °C (i.e. in the γ' precipitation domain) with a cooling rate of 10 °C/s, and then at a slower rate. The tests were performed using a radiant furnace and a contactless extensometer following procedures described in references [5,13].

In order to identify regions of high dislocation density, the cECCI method was utilised, which is an advanced scanning electron microscopy technique that allows to map crystalline defects within a wide field-of-view [14]. A Zeiss Merlin scanning electron microscope (Carl Zeiss SMT AG, Germany) with a Gemini-type field emission gun electron column and a Bruker e-FlashHR EBSD detector (Bruker Corporation, USA) was used. To reveal the optimum diffraction contrast of crystal defects,

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the ECCI under controlled diffraction conditions method was employed to rotate and tilt the crystal into two-beam diffraction conditions [14]. The computer program TOCA was used to select suitable rotation/tilt angles from simulated electron channeling patterns based on a prior EBSD analysis at the region of interest [14]. The microscope was operated at 30 kV accelerating voltage and 3 nA probe current. The specimen was positioned at 7 mm working distance and was tilted to an angle in the range [−4°, 20°].

APT specimens were prepared from site specific lift-outs from regions of the IN792 alloy with high dislocation density and from the fully rafted microstructure of MC2 alloy using a dual beam focused-ion beam FEI Helios 600 following procedures described in reference [15]. Specimens from IN792 alloy were analysed on a Cameca LEAP 3000 HR instrument operating in laser pulsing mode with a repetition rate of 100 kHz, laser pulse energy 0.6 nJ and at a base temperature of 70 K. The specimens from the MC2 alloy were analysed on a Cameca LEAP 5000 XR instrument operating in laser mode with a repetition rate of 125 kHz, laser pulse energy 45 pJ and at a base temperature of 50 K.

We first focus on the polycrystalline, fully heat-treated nickel-based IN792 superalloy subjected to a short degradation treatment during a static and isothermal exposure at 750 °C in air for 50 h. It has been suggested that environmental degradation results in the oxidation of MC carbides connected to the surface, causing substantial volume expansion that can cause localised plastic deformation [16–19]. Fig. 1a shows a cECCI micrograph from the vicinity of an intragranular oxidised carbide connected to the surface (evidence of severe oxidation of the carbides and their surface eruptions is shown in the supplementary figure) revealing the dissolution of the γ' precipitates as well as significant recrystallisation in its vicinity. Note that recrystallisation was observed only next to the oxidised carbide and not along the surface of the specimen. A high density of dislocations, imaged in bright contrast, is readily visible in the original microstructure near the recrystallised grains. These originate from the volume expansion of the oxidised carbide that continuously generate misfit dislocations. At such a relatively short exposure time at 750 °C, a temperature substantially below the γ' solvus, bulk diffusion is expected to be too slow to cause this dissolution. We assume that these dislocations contribute to the local dissolution of the γ', releasing them from topological constraints so that they can travel through the soft recrystallised grains to the heat-treated regions of the microstructure. This explains why the dislocation density is higher in the region between the recrystallised grains and the original microstructure. Conversely, the dislocation density is lower far away from the carbide.

Fig. 1b shows an APT reconstruction from a specimen prepared from a region of the γ/γ' microstructure exhibiting a high dislocation density near the interface with the recrystallised region. The reconstruction contains a γ' precipitate and its interface with the γ matrix. We also observe small γ precipitates within γ', which are believed to result from the applied heat treatment [20,21]. Within the γ' precipitate, we observe local enrichment, along tubular features, of chromium and cobalt of up to 8.0 and 12.0 at.%, respectively. These are akin to segregation to dislocations often encountered in APT [22–24] and previously confirmed through correlation with transmission electron microscopy (TEM) [25]. A cylindrical region of interest perpendicular to a representative dislocation, indicated in Fig. 1b (arrow #1), reveals chromium and cobalt enrichment at the dislocation as clearly shown in Fig. 1c. Aluminium and titanium are depleted, whereas the nickel concentration at the dislocation is the same as that of nickel in the γ' precipitate. No connection between the dislocations and the γ precipitates was observed by APT, thus the chromium and cobalt segregation to the dislocations originates uniquely from the γ matrix and not from the γ precipitates within the γ' precipitate.

The segregation of chromium and cobalt to dislocations causes local chemical inhomogeneities and leaches these elements from the γ

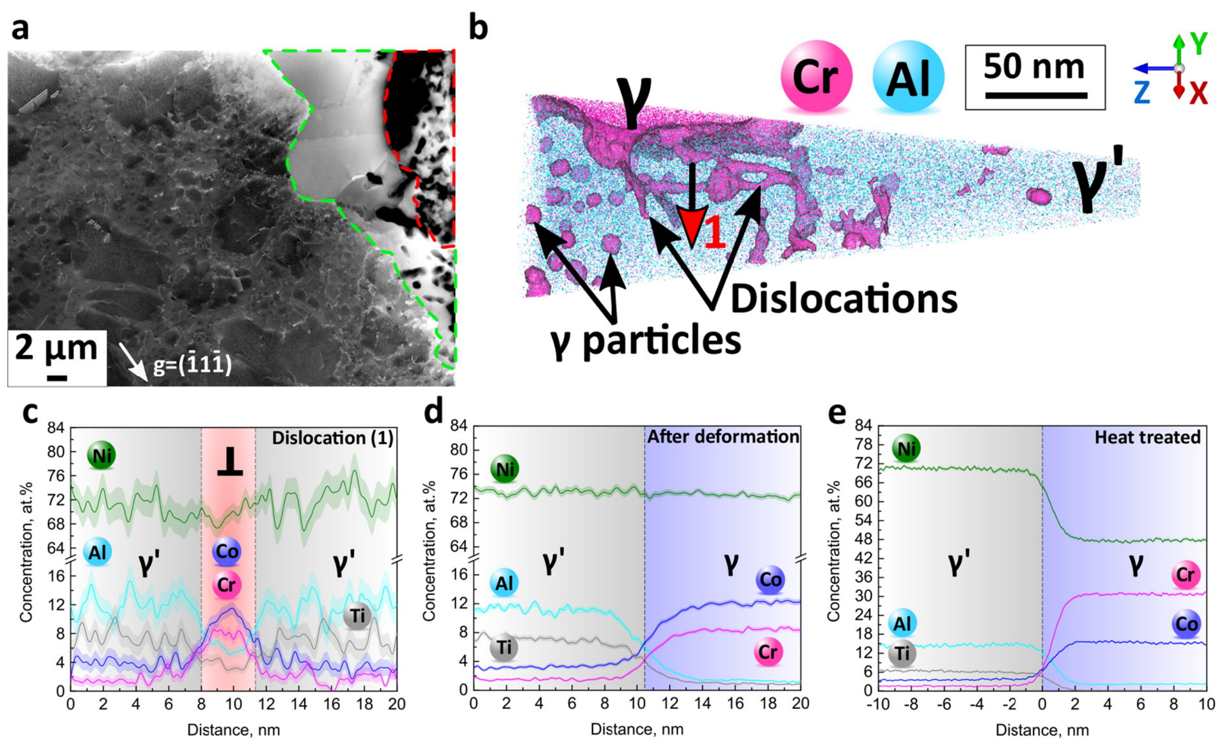

Fig. 1. (a) cECCI micrograph from the cross section of an oxidised intragranular MC carbide in IN792 alloy after static and isothermal exposure at 750 °C for 50 h showing a high dislocation density due to the plastic deformation from the volume expansion during oxidation. Part of the oxidised MC carbide is denoted by the red dashed line and the recrystallised zone is denoted by the green dashed line. (b) Atom probe reconstruction from a high dislocation density γ/γ' region, showing a γ/γ' interface, dislocations and γ precipitates within the γ' precipitate. The γ/γ' interface and dislocations are shown with an isocomposition surface at 4.6 at.% Cr. (c) 1D concentration profile perpendicular to the denoted dislocation in Fig. 1b (arrow #1). (d) 1D concentration profile across the γ/γ' interface from the deformed region due to volume expansion of the oxidised MC carbide. (d) Concentration profile across the γ/γ' interface of IN792 after full heat treatment and prior to any deformation. Error bars are shown as lines filled with colour and correspond to the 2σ counting error. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)