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On the crystallography and composition of topologically close-packed phases in ATI 718Plus®

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ABSTRACT

ATI 718Plus® is a nickel-based superalloy developed to replace Inconel 718 in aero engines for static and rotating applications. Here, the long-term stability of the alloy was studied and it was found that topologically close-packed (TCP) phases can form at the γ-η interface or, less frequently, at grain boundaries. Conventional and scanning transmission electron microscopy techniques were applied to elucidate the crystal structure and composition of these TCP precipitates. The precipitates were found to be tetragonal sigma phase and hexagonal C14 Laves phase, both being enriched in Cr, Co, Fe and Mo though sigma has a higher Cr and lower Nb content. The precipitates were observed to be heavily faulted along multiple planes. In addition, the disorientations between the TCP phases and neighbouring η/γ were determined using scanning precession electron diffraction and evaluated in axis-angle space. This work therefore provides a series of compositional and crystallographic insights that may be used to guide future alloy design.

1. Introduction

The nickel-based superalloy ATI 718Plus® (hereafter 718Plus) has been developed with a view to replace the current workhorse alloy in the gas turbine industry, Inconel 718 (hereafter 718). This new alloy offers an operating temperature increase of about 37 K whilst maintaining workability and weldability [1]. This is achieved through changes in the alloy composition. Most importantly, the Al:Ti ratio is increased and W is added (Table 1), which leads to changes in precipitation behaviour within the γ matrix (Ni, Al, cubic, $Fm\bar{3}m$). In alloy 718 the primary strengthening phase responsible for increased dislocation drag is a fine dispersion of γ″ (Ni_3Nb, $D0_{22}$, tetragonal, $I4/mmm$) with some γ′ (Ni_3Al/Ti, $L1_2$, cubic, $Pm\bar{3}m$), whereas the main strengthening phase in 718Plus is γ′ [2] although some have argued that γ″ may still occur [3]. This is key to performance because γ′ is stable to higher temperatures than γ″, which readily transforms to δ phase above 650 °C [4,5]. The other major difference is that where the dominant grain boundary precipitates in alloy 718 are needle-like δ phase (Ni_3Nb, $D0_a$, orthorhombic, $Pmmn$) the dominant precipitates in 718Plus are η phase ($Ni_3Nb_{0.5}(Al/Ti)_{0.5}$, $D0_{24}$, hexagonal, $P6_3/mmc$).

The morphology of η phase precipitates in 718Plus depends on the thermo-mechanical treatment to which the alloy has been subjected. Often it occurs in colonies in a Blackburn orientation relationship $\{111\}_\gamma \,||\{001\}_\eta$ [7] with one of the adjacent grains. The η phase precipitates then grow in a disc-like morphology on the $\{111\}_\gamma$ matrix planes towards the grain interior and a detailed mechanism has been suggested [8]. It is also common to observe small fractions of fine δ phase sheets within η phase discs [7,8]. In the context of this study it is important to note that after prolonged annealing η precipitates can grow significantly in size, spanning across entire grains as well as growing in aspect ratio to form blocky precipitates with distinct facets [9]. Further, both η and γ′ compete for the same alloying elements, mainly Al, Ti and Nb, resulting in γ′ precipitate free zones (PFZ) around η precipitates and the suggestion that γ′ might play a role in the formation of η [10]. Considering mechanical performance, the η phase is critical for effective grain boundary pinning in sub-solvus forging [11–13] and whether, for example, fine or blocky

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Table 1
Composition of ATI 718Plus and Inconel 718 in wt% [6,7].

Element wt%	Al	Ti	Cr	Fe	Co	Ni	Nb	Mo	W	C + B + P
718Plus	1.45	0.7	18.0	10.0	9.0	Bal.	5.4	2.7	1.0	<0.05
Inconel 718	0.45	1.0	18.1	18.0	–	Bal.	5.4	2.9	–	<0.05

precipitates have formed, and to what extent, will have a strong effect on mechanical properties [9].

Topologically close-packed (TCP) phases may form in most nickel-based superalloys [14,15] when exposed to conditions of high temperature for long periods of time or during solidification and welding [16–18]. Typically, TCP phases are composed principally of the elements Ni, Cr, Co, Mo, and W, and the basic crystallography of common TCP phases is summarised in the supplementary information (Table SI 1). The structures are relatively complex [19,20] but at the simplest level consist of pseudo-hexagonal layers of atoms stacked to form sites with coordination numbers as high as 16 accommodating atoms of widely different sizes. In spite of this, the packing efficiencies are comparable to those of ideal close-packed structures. The TCP phases are potentially detrimental if they occur in significant volume fractions such as to cause the depletion of solute atoms which otherwise aid solid solution strengthening [21]; they cannot be used as strengthening phases themselves due to low number density [22].

The occurrence of TCP phases in 718Plus has so far been little studied and those studies that do report TCP phases [6,16,18,23] in 718Plus have largely neglected their crystallography. Instead, the TCP phases have been identified based mainly on composition, which makes it unclear as to exactly which TCP phases are observed. In terms of the conditions leading to the observation of TCP phases in 718Plus this has mostly been attributed to solidification of the alloy after casting [6] or to welding [16,18,23]. Interestingly, we note that no TCP phases were reported in a study on long term stability of 718Plus (732 °C, 2500 h) by Radavich et al. [24]. In this work, we report the observation of TCP phases in 718Plus subjected to high temperature annealing and characterise TCP precipitates both chemically and crystallographically using (scanning) transmission electron microscopy and diffraction.

2. Methods

2.1. Heat treatments

718Plus samples were provided by Rolls-Royce Deutschland Ltd. & Co KG. The initial ingot material was triple vacuum melted by Allegheny Technologies Inc. for high cleanliness and forged into billet product. This was followed by subsolvus forging to form a black forging and heat treatment A (Table 2) performed by Otto Fuchs KG. Further, long-term anneals B-D were performed by the authors, on separate samples, in order to examine microstructural stability.

Table 2
718Plus samples investigated, with details of respective heat treatment.

Treatment	Details	Treatment	Details
A	843-871 °C for 16 h	+B	+650 °C for 500 h
	954-982 °C for 1 h	+C	+704 °C for 500 h
	788 °C/8 h/FC + 704 °C/8 h	+D	+788 °C for 500 h

2.2. Specimen preparation

Electron-transparent thin film specimens were prepared as follows. First, samples were extracted from the mid-radius of the heat-treated forgings using electric discharge machining (EDM). Slices of 300–500 μm were cut using a saw and 3 mm diameter discs were produced using EDM. These discs were ground to approximately 200 μm thickness and subjected to electrolytic twin-jet polishing using a Tenupol and 10 vol% perchloric acid solution at −5 °C.

2.3. (Scanning) transmission electron microscopy

TEM images and small angle convergent beam electron diffraction (CBED) patterns were acquired using Philips CM30 and JEOL 200CX microscopes operated at 200 kV. Most images and diffraction patterns were acquired using Gatan 2K digital cameras (Orius SC200) although selected images were acquired on photographic film.

Scanning transmission electron microscopy (STEM) was performed using an FEI Tecnai Osiris. The machine was operated at 200 kV and energy dispersive X-ray (EDX) spectrum images and annular dark-field (ADF) images were acquired simultaneously using a scan step size of 3 nm. The specimen was not tilted and the gun lens was adjusted to produce a large current (0.7 nA) for increased X-ray generation, which allowed a modest 200–250 ms dwell time per pixel.

Scanning precession electron diffraction (SPED), in which a PED pattern is acquired at every position in a scan, was performed on a Philips CM300 FEGTEM operated at 300 kV. The scan and simultaneous precession of the electron beam was controlled using a NanoMegas Digistar system, combined with the ASTAR software package [25]. A convergent probe was used, typically with a convergence semi-angle ca. 1 mrad and a precession angle ca. 9 mrad, aligned as described recently [26]. Scans were acquired with a step size of 10 or 20 nm depending on the region of interest. The PED patterns were recorded using a Stingray CCD camera to capture the image on the binocular viewing screen with an exposure time of 40–60 ms. The recorded patterns were corrected for geometric distortions prior to any further analysis.

3. Results

3.1. The occurrence of TCP phases

TCP precipitates were observed in samples produced following all four heat treatments (A-D). In each sample, bright field (BF) images, as shown in Fig. 1, were obtained from approximately 20 TCP particles to assess sites of occurrence within the microstructure, their morphology and size. Across all samples, it was found that almost all (96%) occur at γ-η interfaces and often extend along specific η facets. In a number of cases, it appears that the TCP particles may have nucleated on an η precipitate and continued to grow towards a triple junction or along a grain boundary (e.g. Fig. 1b). TCP particles were also observed at some grain boundaries although it remains possible that these formed initially at a γ-η phase boundary, that was removed during TEM sample preparation. The particles are typically blocky in morphology and exhibit internal faulting. This is in contrast to other alloys in which the TCP particles are plate-like [19], may be a result of different misfit. In the as-received condition (A), particles were measured to have their largest dimension in the range 55–230 nm. After further annealing, the particles had grown size ranges: 50–320 nm, 80–1050 nm and 120–1170 nm for conditions B-D, respectively. In the remainder of this work, TCP precipitates are studied in sample D, which was