Scripta Materialia 117 (2016) 81–85

Contents lists available at ScienceDirect

Scripta Materialia

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

Regular Article

Precipitation of the ordered α_2 phase in a near-α titanium alloy

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ARTICLE INFO

Article history:
Received 18 September 2015
Received in revised form 8 February 2016
Accepted 10 February 2016
Available online 10 March 2016

Keywords:
Titanium alloys
Precipitation
Atom probe tomography
Transmission electron microscopy (TEM)

ABSTRACT

Precipitate evolution in a near-α alloy was studied using transmission electron microscopy (TEM) and correlative atom probe tomography (APT) after ageing at 550–700 for times up to 28 days. It is found that precipitation occurs much faster and is more prolific in samples heat treated at higher temperatures. Particles were spherical after ageing at 550 °C, while after ageing at 700 °C they become ellipsoids with the major axis lying close to the [0001] direction. At longer ageing times, the α_2 precipitates were found to contain greater amounts of Sn + Si, indicating that Sn and Si are stronger Ti_3(Al,Sn,Si) formers than Al.

Recent years have seen dramatic increases in the demand for commercial air travel and the cost of fuel, along with reductions in the cost of capital and the social acceptability of emissions. Strong competition has therefore driven efforts to improve the efficiency of aircraft engines [1] by running them at higher temperatures [2]. As a consequence, the materials used have to be resistant to increasingly extreme conditions. Creep resistant α Ti alloys have a high specific strength, making them ideal for high temperature compressor applications [3].

Many studies have been carried out to develop near-α Ti-Al alloys containing additions of zirconium (Zr), tin (Sn), molybdenum (Mo), niobium (Nb) and silicon (Si) [4–8]. Near-α Ti alloys are utilised for compressor discs and blades [9,10] with improved tensile strength, fatigue resistance and creep performance at temperatures up to 660 °C [11–13]. However, as a result of solute partitioning in primary α grains, formation of the α_2 (Ti_3Al) phase has been reported [14,15]. A detrimental effect of Ti_3Al precipitates on fracture toughness and low cycle fatigue properties is well documented [16]. Moreover, it is known that the low cycle fatigue resistance is reduced due to promotion of strain localisation as a result of the presence of α_2 [17]. It was previously reported that when slip occurs in a material containing Ti_3Al precipitates dislocations travel in pairs [18]. Cross slip is restricted and therefore deformation tends to occur by non-homogeneous planar slip [17,18]. This is a significant problem since the resulting lowered fatigue resistance can reduce the lifetime of compressor discs [19].

IMI 834 is a near-α alloy currently deployed in high temperature compressor disc applications with a β transus temperature of 1060 °C [20]. This alloy has a reduced volume fraction of β phase with ~15 vol.% of the fine primary α phase. This is achieved by reducing the proportion of β stabilisers, such as Mo. Sn and Zr are added as α stabilisers [21] in quantities up to 6 wt.%. Above this, embrittlement occurs due to the formation of Ti_3Al [7]. Small amounts of Si are added to increase the high temperature strength by the formation of fine ordered precipitates on the lamella boundaries, having a stoichiometry of $(Ti,Zr)_6Si_3$ [22,14].

IMI 834 has a good combination of creep, low cycle fatigue (LCF) and crack propagation properties, but a potential problem with this alloy is the precipitation of the α_2 phase when the solubility limit of Al is exceeded. In service, near-α Ti alloys are used for many thousands of hours at temperatures above 500 °C and it is possible that they are subjected to an uncontrolled decomposition transformation. It is worth noting that, although limited formation of the α_2 phase is sometimes used in commercial Ti alloys to improve strength, the precipitation process is still not well understood. Hence, a detailed investigation of the kinetics of the formation and nucleation of α_2 precipitates in the α phase is essential for predicting the behaviour of materials at elevated temperatures.

Previous atom probe tomography (APT) and transmission electron microscopy (TEM) work on decomposition transformation in the α phase of IMI 834 has been minimal and the information on the kinetics of the transformation is still unclear.

Lenssen [6] noticed the precipitation of the ordered particles in the α grains after 4 and 100 h of ageing at 700 °C and linked this with a slight reduction in mechanical performance. After ageing for 500 h at 700 °C

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http://dx.doi.org/10.1016/j.scriptamat.2016.02.015
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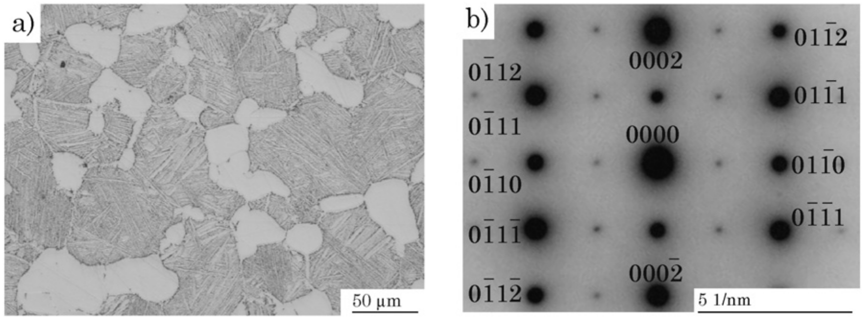

Fig. 1. a) As-received bimodal microstructure and (b) Selected area $[2\bar{1}\bar{1}0]$ diffraction pattern in the 625/14d sample. The intensity scale has been inverted to highlight the superlattice reflections.

much larger precipitates were observed. Although ordered regions approximately 2.5 nm in diameter and 10 nm apart were seen in the α phase after 4 h at 700 °C, no accurate information about the chemical composition was given. Zhang et al. [23] investigated the influence of ageing time and temperature on the precipitation and growth of α_2 ordered domains in a series of alloys with compositions similar to IMI 834. It was reported that Al content and ageing temperature influenced the distribution and growth rate of the α_2 phase. The higher the Al content and ageing temperature, the quicker the observed growth of the α_2 phase.

The new generation of APT instruments, with higher detector efficiencies and wide fields of view enable this system to be revisited, which is the focus of the current work. If large particles, like those observed by Lenssen [6], form during service, the consequences could be serious. Therefore, we examined features observed in the samples of aged IMI 834 to find out the composition of phases and the effect of ageing parameters on their nucleation and growth.

The IMI 834 material used was received from Rolls-Royce plc, of composition (measured by ICP-OES) Ti-5.8Al-4.0Sn-3.5Zr-0.7Nb-0.5Mo-0.3Si-0.10O, in wt.% (10.3Al-1.6Sn-1.8Zr-0.4Nb-0.2Mo-0.0.6Si-0.3O at.%). The alloy was forged, heat treated in the $\alpha + \beta$ phase region to give 10–15% primary α_2 and then aged for 2 h at 625 °C. The microstructure is shown in Fig. 1. Small samples were then encapsulated in quartz with an Ar atmosphere and heat treated, following Lenssen [6], for 14 days at 625 °C (625/14d), 28 days at 550 °C (550/28d), 100 h at 700 °C (700/100 h) and for 16 days at 700 °C (700/16d). A larger experimental matrix was initially examined; the conditions reported here correspond to the earliest times at which some precipitation could be observed.

TEM experiments were performed using a JEOL 2000FX 200 kV TEM. Samples were prepared by two methods: (i) in-situ lift-out [24] and (ii) electrolytic thinning [25]. To produce specimens by the first method (i) a focussed ion beam/scanning electron microscope (FIB/SEM) Helios NanoLab 600 equipped with an Omniprobe™ was used. To prepare specimens by the second method (ii) thin slices were sectioned from the alloy, mechanically thinned down to 100 µm and discs of 3 mm diameter were cut by spark eroding. The discs were electrolytically thinned in 3% perchloric acid, 40% butan-1-ol and 57% methanol using a Tenupol-5 twin-jet electropolisher at −40 °C and a voltage between 20 and 25 V.

Atom probe samples were prepared by the FIB lift-out method. A description of the lift-out and sharpening of needle-shaped tips can be found in reference [26]. APT experiments were carried out using both a 4000X Si and 3000X HR local electrode atom probe (LEAP). The 4000X Si offers higher detection efficiencies ~57% at the expense of mass resolution, while the 3000X HR is less efficient ~37%, but has improved mass resolution. Each set of needles was analysed using laser and voltage-pulsing modes, in order to ensure that the observed segregation behaviour was not an artefact induced by (thermal) laser effects. The results were independent of the instrument and operating mode, but laser mode gave higher yields. A laser pulse energy of 0.1 nJ and frequency of 200 kHz were used. In voltage-pulsing mode on the LEAP 4000X Si, the voltage pulse fraction was 15%. For both modes the sample stage temperature was held at 55 K. IVAS™ data analysis software (Cameca) based on the standard algorithm [27] was used for reconstruction of the collected data [28,29]. The iso-surface concentrations were selected to highlight the α_2 phase.

A new phase may either (i) form as a consequence of spinodal decomposition or (ii) it may form by nucleation with a long incubation time [6,30,31]. In the case of spinodal decomposition a compositional wave forms, which grows until a new phase is formed. In case (ii) a long incubation time can be explained by slow nucleation. To

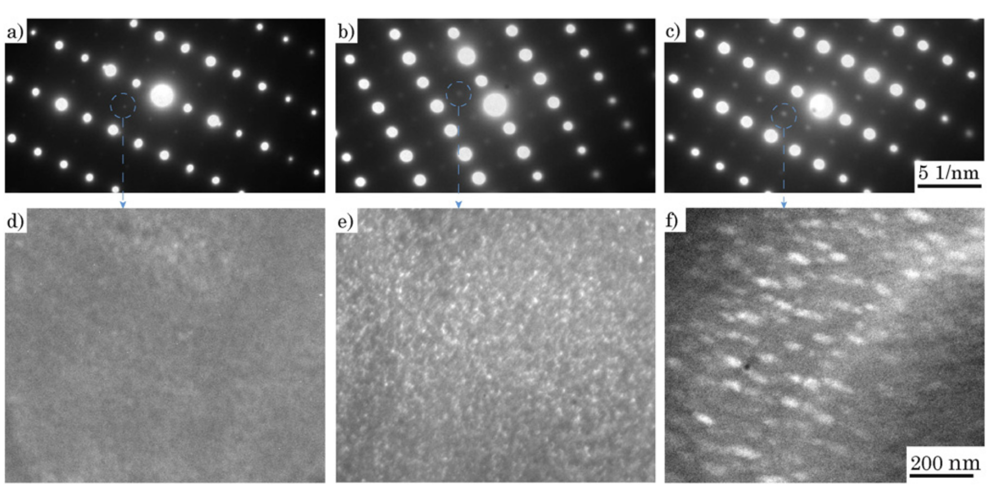

Fig. 2. Selected area $[2\bar{1}\bar{1}0]$ diffraction patterns after heat treatment at a) 550/28d b) 700/100 h c) 700/16d. Corresponding dark field images of the ordered regions taken from the marked superlattice spot, d), e) and f) respectively.