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Tuning the scale of α precipitates in β-titanium alloys for achieving high strength

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ABSTRACT

The strength of a commercial β titanium alloy was tuned by systematically controlling the size-scale of α precipitation via two-step heat treatments. While the first step annealing at 350 °C forms the precursor metastable ω phase, the subsequent annealing at 600 °C/1H resulted in fine-scale α precipitation. We find that annealing duration at 350 °C greatly impacts the α size-scale and, this consequently can be used to tune the ultimate tensile strength (UTS) from ~1 to 2 GPa. A combination of high volume-fraction of non-shearable super-refined α precipitates, coupled with their pyramidal arrangement, and a constrained β matrix leads to very high strength.

Over the last few decades, β titanium alloys have largely been exploited as structural alloys owing to richness in their microstructural features [1,2]. These features which lead to a unique combination of high specific-strength and ductility, excellent hardenability, good fatigue performance, and corrosion resistance make these alloys a viable candidate for many applications [3–6]. While the ductility itself is largely dependent on the prior β grain size, other mechanical properties like strength, are expected to be dependent on the volume fraction, morphology, and distribution of α precipitates in the β matrix [7,8]. As is the case with any precipitation-hardenable system, there is a constant push for controlling the formation of α precipitates with desired volume fractions by using a combination of thermal and mechanical processing. Ivasishin et al. published a series of papers in the 2003–2008 period highlighting the importance of thermomechanical processing in β-Ti alloys [9–11]. Once a fine-grain size was achieved via thermo-mechanical processing, two different types of heat treatments were performed: (i) a series of contiguous heat treatments using different heating rates, (ii) heat treatments involving intermediate aging steps. Depending on the heat treatments a wide range of strength-ductility combinations were achieved, which were attributed to the microstructural changes resulting from the heat treatments. Fine-scale α precipitation was observed in the samples that experienced intermediate aging steps. This was attributed to the omega (ω)-phase possibly acting as a pre-cursor to α precipitation, which has more recently been conclusively established in the literature [12,13]. Here we have systematically varied α volume fraction and the corresponding alloy strength (while retaining reasonable ductility) via this phenomenon using a two-step heat treatment. The first step promoted ω formation, while the second step caused ω-assisted α precipitation. We have applied this approach to a well-known β-stabilized alloy, β-21S, which is known to have excellent combination of producibility, high ambient strength, and good elevated temperature properties [4,14].

Sheets of β-21S with nominal composition Ti-15Mo-3Nb-2.7Al-0.2Si wt% were provided by TIMET Company. All specimens were first solutionized at 900 °C for 30 min followed by water quenching. The specimens were then subjected to isothermal annealing at 350 °C for periods ranging from 0H to 150H, to vary the ω volume fraction. Subsequently, specimens were annealed within the α + β phase field at 600 °C for 1H to promote α precipitation. The schematic of all these heat treatments is shown in Fig. 1(a). Microstructural characterization was carried out in a FEI NovaNano SEM230 and a FEI Tecnai F20-FEG TEM operated at 200 kV. Phase compositions were extracted using a 3D atom probe tomography (APT) in a LEAP™ system from Cameca Inc. For both TEM and APT analysis,

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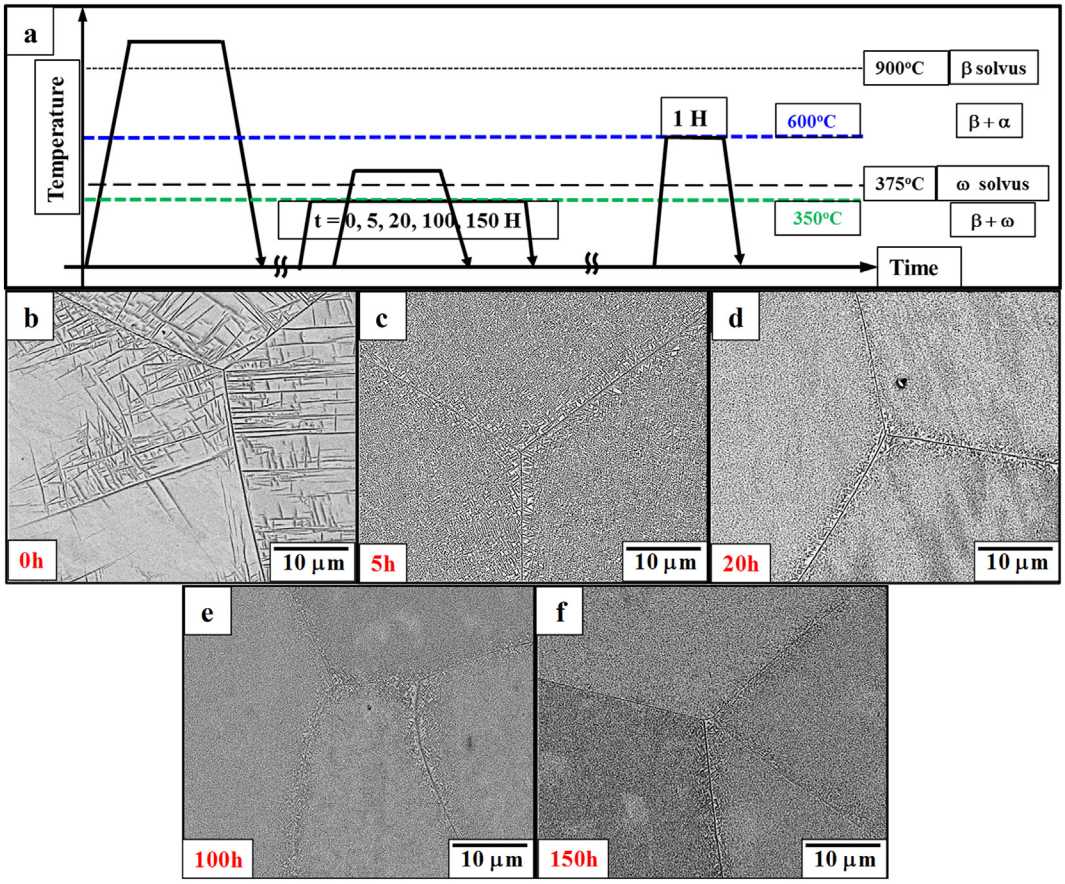

Fig. 1. (a) Schematic showing the heat treatment for the fine scale α precipitation; (b–f) backscattered scanning electron images showing the α precipitates at different aging periods from 0H to 150H.

samples were prepared using a dual-beam FIB-Nova Nanolab 200 system from FEI. Dog-bone shaped tensile specimens of gage length ~3 mm, width ~1 mm and thickness ~0.4–0.6 mm were used for the mechanical testing, which was done under uniaxial tension at room temperature with a strain rate of 10^{-3} s^{-1}. Details of the setup for the mechanical testing are presented elsewhere [15–17].

The microstructures of aforementioned heat-treatments of the β-21S samples are shown in Fig. 1(b–f). As can be seen in Fig. 1(b), the scale of distribution of the α precipitates is not very refined, compared to Fig. 1(c–f), and appears to be very similar to that formed by high temperature duplex-aging condition [4,18]. However, the fine-scale α precipitation observed in case of Fig. 1(c–f) can presumably attributed to the effect of prior isothermal ω precipitation during the first-step of annealing at 350 °C. Such ω-assisted fine-scale α precipitation has been previously reported in other β-Ti alloys [12,13]. The samples aged for 20H and 100H show not only a finer precipitate size, but also a higher number density. Also included, as Fig. 1(f), is the micrograph of the sample that was pre-aged to 150H at 350 °C. On first viewing, there does not appear to be any difference between the samples aged to 20H and 100H, but on further inspection using MIPAR [19], an image processing toolset, we are able to calculate the difference in the values of number density and the equivalent diameter of all these samples. It should be noted that while the changes are very subtle, they do seem to influence the mechanical properties drastically. These will be further discussed in the relevant sections.

The sample which was aged to 150H was further analyzed using TEM and 3D-APT techniques. This was done as the α precipitates in this condition were too fine in size to be analyzed using standard image processing techniques applied to SEM images. The DFTEM image in Fig. 2(a) was obtained by selecting the α reflections near 1/2[112] in the [110]β zone axis, which is shown as inset. This DFTEM image in Fig. 2(a) and the HAADF-STEM image shown in Fig. 2(b), give an idea regarding the number density and size of these extremely fine α precipitates. The STEM image clearly shows that the α laths are no more than ~70–100 nm in length and less than ~20 nm in thickness. One interesting thing to note here is the presence of triangular configuration of α precipitates, highlighted in Fig. 2(b). Such a triangular configuration of precipitates is of great interest to researchers, since it can significantly enhance the mechanical properties [15,20,21]. According to Miyano et al. [21], the triangular configuration results from the invariant lines of three different laths that are not parallel to each other, and as they expand along each direction, they join to form a pyramidal arrangement. The strain-energy criterion is another factor which comes into play when discussing the clustering of the α laths. The stress-free transformation strain resulting from the β to α transformation and the coupling of such strain fields arising from multiple crystallographic α variants, resulting in reduction of the overall strain-energy, can result in the clustering of certain variants of α laths. This mechanism has also been referred to as self-accommodation in the literature, and has been observed not only in the case of Ti-base alloys, but also for Zr-base alloys [22–26]. Specifically, in case of Ti-base alloys, both experimental observations as well as computations of the strain energy have revealed that the clustering of three Burgers oriented α variants, sharing a common ⟨11−20⟩ with the same ⟨111⟩ pole of the parent β grain, leads to a self-accommodating triangular shaped