On the effect of incremental forming on alpha phase precipitation and mechanical behavior of beta-Ti-10V-2Fe-3Al

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Abstract. A combination of good ductility and fatigue resistance makes $\beta$-titanium alloys interesting for many current and potential future applications. The mechanical behavior is primarily determined by microstructural parameters like (beta phase) grain size, morphology and volume fraction of primary / secondary $\alpha$-phase precipitates, and this allows changing and optimizing their mechanical properties across a wide range. In this study, we investigate the possibility to modify the microstructure of the high-strength beta titanium alloy Ti-10V-2Fe-3Al, with a special focus on shape and volume fraction of primary $\alpha$-phase. In addition to the conventional strategy for precipitation of primary $\alpha$, a special thermo-mechanical processing is performed; this processing route combines the conventional heat treatment with incremental forming during the primary $\alpha$-phase annealing. After incremental forming, considerable variations in terms of microstructure and mechanical properties can be obtained for different thermo-mechanical processing routes. The microstructures of the deformed samples are characterized by globular as well as lamellar (bimodal) $\alpha$ precipitates, whereas conventional annealing only results in the formation of lamellar precipitates. Because of the smaller size, and the lower amount, of $\alpha$-phase after incremental forming, tensile strength is not as high as after the conventional strategy. However, high amounts of grain boundary $\alpha$ and lamellar $\alpha$-phase in the undeformed samples lead to a significantly lower ductility in comparison to the matrix with bimodal structures obtained by thermo-mechanical processing. These results illustrate the potential of incremental forming during the annealing to modify the microstructure of the beta titanium Ti-10V-2Fe-3Al in a wide range of volume fractions and morphologies of the primary $\alpha$ phase, which in turn leads to considerably changes, and improved, mechanical properties.

1. Introduction

Excellent mechanical and corrosion properties are key reasons for the use of titanium alloys, for instance in structural applications in the aerospace industry [1, 2]. The properties of different alloys are strongly affected by the volume fractions of hexagonal close-packed (hcp) $\alpha$-phase and body-centered cubic (bcc) $\beta$-phase. Compared to the $\alpha$-phase, the $\beta$-phase is characterized by a lower strength, but higher formability. A combination of good ductility and fatigue resistance makes $\beta$-titanium alloys interesting for many current and potential future applications. The mechanical behavior of titanium alloys is primarily determined by microstructural parameters like ($\beta$-phase) grain size, morphology and volume fraction of primary / secondary $\alpha$-phase precipitates, and this allows changing and optimizing their mechanical properties across a wide range [3-5].

A typical example of light-weight titanium structural elements, hollow shafts, are manufactured along various processing routes. This typically involves cold extrusion, or deep hole drilling - but since the
latter is characterized by a material utilization of only 40%, it is quite inefficient for high-grade materials [6]. Incremental forming at elevated temperatures has recently been developed as an alternative for the fabrication of hollow shafts. One such process, which makes use of a partially rotating forming technology to produce axially symmetric hollow shafts directly from solid semi-finished bars, has been termed spin extrusion [7]. The metastable beta-titanium alloy Ti-10V-2Fe-3Al (Ti-10-2-3) was used in first experimental studies with a focus on spin-extrusion at high temperatures and on its effect on microstructural evolution [6]. Ti-10-2-3 is a candidate material for aerospace applications, and it is well known for its excellent formability at higher temperatures [1, 2]. Conventional heat treatments of this alloy consist of a solution treatment and a two-stage annealing for the precipitation of primary and secondary α phases. Annealing at various temperatures in the primary α phase field (from about 600 to 790 °C) leads to different volume fraction and sizes of the precipitates, but does not affect their morphology. Thermo-mechanical processing, in contrast, is known as an effective method to control the size and morphology of different phases in various titanium alloys. Several studies [8-10] have, for example, shown that a change in the morphology of α layers from lamellar to globular α can occur during hot working of Ti-6Al-4V alloy, which considerably improves the mechanical properties of the material. While the microstructural characteristics of Ti-6Al-4V alloy after thermo-mechanical processing have been well documented in the literature, there is to our knowledge no systematic report on the effectiveness of concurrent hot deformation and annealing in the primary α region on the formation of different microstructures in Ti-10-2-3. In this study, we incrementally deform Ti-10-2-3 samples at different temperatures in the primary α phase field to simulate spin extrusion on a laboratory scale. We analyze how different microstructural processes such as phase transformation and work hardening are activated simultaneously, and how they influence the kinetics of microstructural evolution and the resulting changes in mechanical properties.

2. Experimental

The β-titanium alloy Ti-10V-2Fe-3Al (Ti-10-2-3) was obtained in solution-annealed condition from Otto Fuchs KG (Meinerzhagen, Germany). To modify the microstructure (i.e., morphology and volume fraction of the primary α-phase), two different heat treatment strategies were used in this study: In the first one (referred to hereafter as “conventional heat treatment” strategy), the material was solution annealed above the β-transus temperature (830 °C) for 30 min to achieve a fully recrystallized microstructure. The material was then water quenched to produce material fully in the β-phase. To investigate the influence of the primary α-phase on mechanical properties, the material was then heated again up to 600 °C, 650 °C, 700 °C or 750 °C for 60 min followed by air-cooling. From this procedure, four different material conditions with different sizes and volume fractions of the primary α-phase were obtained for further microstructural and mechanical analysis.

In addition to this conventional strategy, a special “thermo-mechanical processing” (TMP) approach was studied. This processing route combines the conventional heat treatment with an incremental forming during the primary α-phase annealing (the procedure is shown schematically in Figure 1). The forming was also realized at 600 °C, 650 °C, 700 °C, and 750 °C with 250 forming increments in 60 min. Each increment consisted of 1 mm compression and 1 mm tension. The whole process (forming, annealing and (air) cooling) was performed in a hydraulic universal testing machine of type SchenckPTT250 with electrical resistance heating of the samples (see Figure 2 for the geometry of the TMP sample, which most notably has a parallel length of 20 mm – further details on this special sample type have been explained in detail in [11]).
Figure 1. Schematic illustration of the applied heat treatment for the Ti-10V-2Fe-3Al alloy. First, a solution treatment is performed at 830 °C for 60 min with subsequent water quenching, followed by primary α phase annealing (at 4 different temperatures to obtain different primary α volume fractions: 600 °C, 650 °C, 700 ° and 750 °C) and air cooling. The thermo-mechanical processing includes an incremental forming during the α phase annealing.

Small (“mini”) tensile specimens were cut out of the TMP samples by spark erosion. Depending on the forming temperature, 3 to 4 specimens could be taken out of one TMP-sample, as shown in greater detail in Figure 2. A systematic characterization of the stress-strain behavior as a function of the annealing temperature could be performed using a UPM 1475 Zwick/Roell tensile testing machine. Because of the small size of the mini tensile specimens, axial elongation was measured with an optical digital image correlation System (Aramis by GOM). This technique tracks the displacement fields of a speckle pattern on the specimen surface. The deformation field is recorded during testing and can subsequently be converted into strain data. The uniaxial tensile tests were carried out at an initial strain rate of 10⁻³ 1/s at RT.

Finally, the microstructures of all specimens were analyzed by optical microscopy and by scanning electron microscopy (SEM), using a Zeiss Neon 40ESB operated at 10 kV with a 4 quadrant backscatter detector.

Figure 2. The TMP sample of Ti-10-2-3 during the thermo-mechanical processing in the primary α phase field (600 °C - 750 °C) is deformed with 250 forming increments of ± 1 mm during a period of 60 min (left). After TMP, a “mini” tensile specimen is cut out by spark erosion (right). This specimen has a total length of 20 mm and a measuring length of 5 mm.
3. Results and discussion

3.1 Forces during thermo-mechanical processing

We first focus on an evaluation of the thermomechanical behavior during processing. During TMP, each sample was deformed in 250 forming increments (each increment consisted of a total deformation of 1 mm in uniaxial tension, followed by a reverse deformation up to 1 mm in compression). Processing forces, which give an indication of thermal stability of the materials during incremental forming, were recorded during the experiments. Typical results are shown in Figure 3. Forming forces obviously depend on temperature. Interestingly, at 600 °C, the maximum force in each increment is nearly constant at 30 kN (for both loading directions) during the whole TMP procedure. In contrast, the incremental forming forces steadily drop from about 9 kN to 5 kN at 750 °C. It is to be expected that forming forces are lower at higher temperatures, but the transition towards softening during incremental forming at higher temperatures warrants a more detailed analysis. This softening effect can be directly related to the microstructures of the material at the different temperatures considered here (see also Figure 4 and the more detailed discussion in section 3.2): The samples deformed at 600 °C exhibit a high volume fraction of primary α precipitates (about 50 %, Table 1). Furthermore these precipitates are homogeneously distributed in the β grain structure. During the incremental forming process, some of the precipitates become oriented parallel to the direction of the highest shear stress, which is closely related to the formation of shear bands. Cyclic deformation then seems to occur primarily in those shear bands, and at constant stresses. In contrast, the samples deformed incrementally at 750 °C only have a primary α volume fraction of about 8 %; the precipitates are also distributed inhomogeneously. We did not observe the formation of shear bands and therefore the effect of decreasing forming forces is likely to be directly related to cyclic softening of the beta phase, which has been documented at high temperatures [12].

![Figure 3](image)

**Figure 3.** Force-time curves measured during incremental forming at 600 °C and 750 °C, respectively. At 600 °C the force remains nearly constant at about 30 kN (both in compression and in tension) for 60 minutes, whereas the processing force at 750 °C drops from 9 kN to 5 kN during the course of the incremental forming procedure.

3.2 Microstructural investigations

To fully document the microstructural evolution in our Ti alloy during incremental forming, we first consider the characteristics of the initial microstructure in the undeformed Ti-10-2-3 samples after air-cooling. Figures 4a-d show the microstructures of conventionally heat-treated samples (i.e., with annealing between 600 °C and 750 °C, and without incremental deformation). In all heat-treated conditions the material is characterized by a large β-grain size of approx. 300 μm. It is obvious that most
primary α phase has an acicular morphology, which is formed in the β grains. The volume fraction of the precipitates decreases with rising of the annealing temperature from 51% at 600 °C to 19% at 750 °C (Table 1). The size of the α needles and the interlamellar spacing between the α layers increase with increasing temperatures as well.

Table 1. Volume fraction (in %) of primary α after the conventional heat treatment vs. thermo-mechanical processing at different temperatures. The data was estimated from the SEM micrographs using simple image analysis algorithms.

|        | 600 °C | 650 °C | 700 °C | 750 °C |
|--------|--------|--------|--------|--------|
| conv. HT | 51     | 48     | 39     | 19     |
| TMP    | 50     | 38     | 15     | 8      |

Grain boundary α (GB-α) could be observed in all samples after the conventional heat treatment (Figures 4a-4d, highlighted with arrows). In the SEM micrographs (most notably in Figures 4c and 4d) it is visible as a continuous thin layer with a thickness of approx. 1 µm. At 700 °C and 750 °C, the GB-α is surrounded by a zone without any precipitates. In contrast, the GB-α layers at 600 and 650 °C are considerably thinner, and are not associated with a precipitate-free zone. We highlight that this is an important observation because GB-α represents a preferred path for crack growth and can therefore lead to a lower amount of ductility. Finally, at lower annealing temperatures (Figures 4a and 4b), the primary α precipitates form the well-known basket-weave microstructure.

In Figures 4e-h, we present the corresponding SEM micrographs obtained from samples of the TMP material. A comparison between the two heat treatment strategies shows that the morphology of the primary α phase is altered by incremental deformation. Instead of needles, the precipitates form a bimodal structure that consists of both globular and lamellar α-phase. While it seems reasonable to expect that the size of the primary α precipitates is reduced by incremental forming, our microstructural observations point out a different effect: compared to the conventional heat treatment without deformation, precipitate size seems to be unchanged at 700 °C and 750 °C to the conventional treatment; at 600 °C and 650 °C the precipitates in fact are significantly larger in comparison to those of the undeformed material, and no basket-weave structures could be observed. Moreover, the TMP samples show no tendency to precipitate GB-α at lower temperatures. Only at 750 °C, small amounts of GB-α could be observed. Most notably, mechanical loading during incremental forming does have an effect on precipitate orientation: all precipitates are orientated parallel to the direction of maximum shear stresses. Even at lower temperatures (600 and 650 °C) the primary α is related to the formation of clearly recognizable shear bands (see also Figures 5c and d). Comparing the conventional heat treatment and the TMP condition, we also observe that the conventionally annealed material systematically exhibits a higher volume fraction of primary α in comparison to the TMP samples. This difference increases with increasing temperature (see Table 1).
Figure 4. SEM micrographs of the conventional heat treated (a - d) and the TMP samples (e - h) after annealing at 4 different temperatures: The conventional heat treatment leads to finely dispersed lamellar αₚ in the β-matrix. Lower temperatures result in higher αₚ volume fraction. GB-α (highlighted by arrows) can be recognized at all temperatures. The TMP samples show a bimodal microstructure with globular and lamellar αₚ precipitates at 600 and 650 °C.
In Figure 5, we present some interesting microstructural features of the samples heat-treated at 600 °C and 650 °C, respectively, at higher magnification. On the left, (Figures 5a, b) SEM micrographs from undeformed conditions are shown, highlighting the basket-weave microstructures in both cases. At 650 °C, the α-phase volume fraction is slightly lower, so the precipitates have more space to grow which results in larger lamellae. In addition to the basket-weave structures, we also observed regions where the primary α precipitates are predominantly oriented in parallel and hence form no triangles (Figure 5a, dark areas, highlighted by arrows). Further research is required to fully analyze how these regions affect the mechanical behavior. The TMP samples (Figures 5c, d), in contrast, exhibit no basket-weave microstructures. Again, it is obvious from these micrographs that the αp-phase is oriented parallel to the direction of maximum shear stress (i.e., inclined by about 45° to the uniaxial loading direction). Incremental forming at 600 °C leads to the formation of distinct shear bands. Inside these bands, the α-phase is also strongly oriented parallel to the growth direction of the bands. The samples deformed at 650 °C exhibit a bimodal structure with both globular and lamellar α-phase; no significant occurrence of shear banding could be observed.

![Figure 5](image)

**Figure 5.** SEM micrographs of the conventionally heat-treated (a, b) and the TMP materials (c, d) after annealing at 600 °C (a, c) and 650 °C (b, d): The conventional heat treatment leads to a typical basket-weave structure and smaller precipitates. There are also regions where the primary α is primarily oriented in one direction (5a, dark areas, highlighted with arrows). The TMP samples exhibit a bimodal microstructure with globular and lamellar α. Also the localization of deformation in shear bands can be clearly observed (as highlighted in c). The αp - phase is oriented parallel to the orientation of maximum shear plane stress during incremental forming in all TMP samples.

### 3.3 Mechanical properties

Stress-strain curves of the undeformed and the conventionally heat-treated materials are shown in Figure 6. Clearly, precipitation of primary α significantly increases the yield strength (YS) and the ult-
mate tensile strength (UTS) in comparison to the solution treated material (which only consists of the $\beta$-phase). After the conventional heat treatment the YS is increased from approx. 480 MPa (ST) to approx. 800 MPa after heating to 700°C or 750 °C, to 900 MPa after heating to 650 °C, and to a maximum value of 1050 MPa after heating to 600 °C. Ductility, however, decreases with lower annealing temperatures. The uniform elongation (UE) decreases from 39 % (ST) to 7 % (750 °C) and drops further to 2 % when the material is heat-treated at 600 °C. As expected, these results confirm that a large volume fraction of primary $\alpha$ precipitates increases the strength but decreases the ductility of Ti-10-2-3. We also observe that there is no significant strain hardening for the samples heat-treated at 700 °C and 750 °C samples, whereas those treated at lower temperatures (and the ST material) show some hardening.

**Figure 6.** Engineering stress-strain curves of the samples heat-treated without incremental forming in comparison to the solution treated (ST) material. Yield strength and ultimate tensile strength are significantly increased in comparison to the ST material. Ductility decreases with lower annealing temperatures.

In comparison to materials subjected to the conventional heat treatment, the mechanical behavior of the samples subjected to incremental forming is different (Figure 7). After TMP the YS is increased from approx. 520 MPa (cyclic deformation at 750 °C) to approx. 650 MPa (700 °C), 700 MPa (650 °C) and to a maximum of 750 MPa (600 °C). The strength of all TMP conditions is lower than that of the corresponding materials that were heat-treated without incremental forming. This can be related to the larger amount of $\alpha$-phase precipitates in the conventional heated samples (Table 1), which results in a stronger hardening of the $\beta$-matrix and thus leads to higher strength. In contrast, the ductility of the TMP samples is higher than for the conventional conditions. The samples that were cyclically deformed at 750 °C reach an UE of 22 %. The UE decreases with lower forming temperatures, but even the samples deformed during TMP at 600 °C still exhibit a UE of 6 %. This difference is mainly due to the fact that GB-$\alpha$ and the lamellar $\alpha$ morphology in the conventionally heated samples leads to lower ductility in comparison to the TMP samples that contain only globular or bimodal $\alpha$ precipitates. It is also known that the basket-weave structure, which occurs in the materials heat-treated at 600 °C and 650 °C, considerably reduces ductility. The strain hardening rate of the TMP samples is relatively low. Only those samples cyclically deformed at 750 °C exhibit a significant hardening behavior. Due to the small amount of primary $\alpha$ (about 8 %, Table 1), the strain-hardening behavior of this material condition is actually quite similar to the strain-hardening observed for the solution annealed material that is fully in the $\beta$-phase.

**Figure 6.** Engineering stress-strain curves of the samples heat-treated without incremental forming in comparison to the solution treated (ST) material. Yield strength and ultimate tensile strength are significantly increased in comparison to the ST material. Ductility decreases with lower annealing temperatures.
4. Summary and conclusions

This paper presents first results of an ongoing investigation of the possibility to modify the microstructure of primary α, in particular with respect to precipitate shapes and volume fractions. In addition to the conventional strategy for the precipitation of primary α, a special route of thermo-mechanical processing was performed. This processing approach combines the conventional heat treatment with incremental forming during the primary α-phase annealing (performed in this study at 600 °C, 650 °C, 700 °C and 750 °C, each for 60 min). After the incremental forming, considerable variations in terms of microstructure and mechanical properties were observed for the different conditions. The microstructure of the deformed samples is characterized by globular and lamellar (bimodal) α precipitates, whereas conventional annealing leads to the formation of lamellar precipitates. Furthermore the primary α is oriented in the direction of maximum shear stresses during incremental forming, and forms no specific basket-weave structures. Due to the smaller size and the lower amount of α-phase after incremental forming, the strength lower than in materials treated with the conventional strategy. A high amount of grain boundary-α and lamellar αp-phase in the undeformed materials leads to a significantly lower ductility in comparison to the matrix with bimodal structures. Our study demonstrates that incremental forming of Ti-10V-2Fe-3Al during the annealing step of the thermo-mechanical treatment can be used to modify the microstructures in a wide range of volume fractions and morphologies of the primary α-phase.

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