



# Yield point elongation in fine-grained titanium

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## ABSTRACT

Fine-grained (FG) titanium with average grain size of around 1  $\mu\text{m}$  was produced by annealing nano-grained (NG) Ti at 500  $^{\circ}\text{C}$  for different time. The tensile deformation behaviors of this FG Ti were investigated. The phenomenon of yield point elongation (YPE), namely, a plateau following yielding in the stress–strain curve, is observed in FG Ti. It is found that, with increase of the grain size, the YPE in FG Ti appeared and increased to a maximum value, then decreased and, finally disappeared. Microstructures of FG Ti samples before and after the YPE were investigated. The results show that this YPE phenomenon is attributed to the dislocation behaviors and characteristics of grain size in the FG Ti.

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## 1. Introduction

The phenomenon of yield point elongation (YPE), namely a yield “plateau” in the stress–strain curve following yielding, has been observed in steels [1–3] and other polycrystalline metals [4–6]. This phenomenon is mostly reported to be caused by the dislocation glide [4–6]. As for the case of dislocation glide, the initial mobile dislocation density should be low and the yielding is accompanied by rapid multiplication of dislocations with slower moving velocities under lower stress [3]. In addition, a recent study found YPE phenomenon in a dual-phase high-manganese steel, which was attributed to martensite transformation induced by the localized strain concentration of delta-ferrite [1]. These YPE phenomena were observed during the tensile tests at room-temperature. Furthermore, tensile testing of a 304 stainless steel at temperatures below  $-60^{\circ}\text{C}$  also shows a YPE phenomenon, which was attributed to the dynamic strain softening and/or transformation-induced plasticity (TRIP) [7]. Undoubtedly, the occurring of YPE phenomenon is closely connected with the microstructure evolutions during deformation.

The above YPE phenomena were all found in face-centered cubic (FCC) and body-centered cubic (BCC) metals, while YPE in hexagonal close-packed (HCP) metals was rarely reported. Recently, Barnett et al. [8] reported a YPE phenomenon in a HCP magnesium alloy and they claimed that this YPE phenomenon occurred by twinning. Here in our study, we demonstrate a YPE

phenomenon in a HCP Ti, which was caused not by twinning but by dislocation movements and characteristics of grain size.

The deformation behaviors of coarse-grained (CG), ultrafine-grained (UFG) and nano-grained (NG) Ti have been studied intensively [9–12]. However, the behavior of fine-grained (FG) Ti with grain sizes around 1  $\mu\text{m}$  was less reported. Therefore, another aim of the present study is to shed light on the deformation behaviors of this FG Ti.

## 2. Material and methods

In this study, NG commercially pure (CP) Ti (Grade 2) produced by a combination of asymmetric and symmetric rolling (ASR plus SR) was used as the starting material. The as-processed NG Ti has an average grain size of 80 nm which was elaborated in our previous report [10]. Using a muffle furnace with a temperature accuracy of  $\pm 1^{\circ}\text{C}$  in the hot zone, the NG Ti was annealed at 500  $^{\circ}\text{C}$  for 5, 10, 20, 30, 40, 50 and 60 min, to produce Ti samples with different grain sizes. The microstructures of the annealed Ti samples, both before and after 3% tensile deformation, were investigated using a JEM-2100F field emission transmission electron microscope (TEM) operating at 200 kV. The observed sections for TEM were perpendicular to the normal direction (ND) of the Ti sheets. The tensile testing specimens were cut from the annealed Ti sheets with thickness of 0.3 mm along the longitudinal rolling direction (RD). The surfaces of the Ti sheets samples were polished before test. The gage length of the tensile specimen was 20 mm and gage width was 3.5 mm. The normal tensile tests at ambient temperature were carried out by using a Shimadzu AG-10kNA tension tester at the strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$ .

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### 3. Results and discussion

Tensile deformation behavior of the as-processed NG Ti by ASR plus SR was demonstrated in our previous paper [10]. Fig. 1 displays the tensile deformation curves of the Ti samples annealed at 500 °C for different time. Detailed information of tensile properties, including the length of YPE, is shown in Table 1. The Ti samples annealed for 5 min showed high strength and relatively low elongation. The increase of annealing time from 5 to 10 min resulted in the decrease of strength and enhancement of elongation. Both of these two samples exhibited gradual yielding. It is interesting that YPE occurred in the Ti sample annealed for 20 min. A short plateau appeared following the incipient yielding in the stress–strain curve. The sample annealed for 30 min showed a longer plateau (YPE) of about 2.5%. The increase of annealing time from 30 to 40 min resulted in a little reduction of the YPE length. Further increasing the annealing time to 50 min led to remarkable reduction of the YPE length. The YPE phenomenon was not observed in the Ti samples annealed at 500 °C for 60 min.

Fig. 2 gives the typical TEM images and statistical grain size distributions of Ti samples annealed at 500 °C for different time. It can be seen that all the annealed samples possessed equiaxed microstructure and the samples with longer annealing time exhibited larger grain size as well as wider grain size

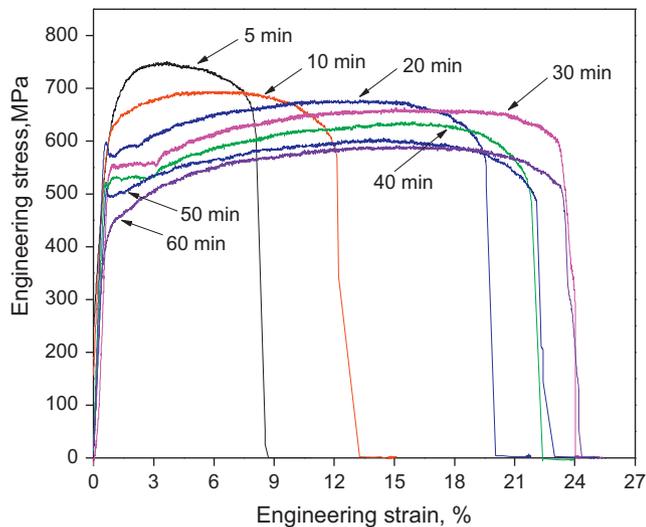


Fig. 1. Tensile deformation curves of the titanium samples annealed at 500 °C for different time.

Table 1

Tensile properties and grain sizes of the Ti samples annealed at 500 °C for different time.

Annealing time (min)	Tensile properties				Grain size	
	Length of YPE (%)	YS (lower) (MPa)	UTS (MPa)	A (%)	Average value ( $\mu\text{m}$ )	Number of analyzed grains
5	0	684.6	748.8	8.5	0.29	554
10	0	619.0	693.2	12.2	0.44	488
20	0.34	575.3	677.7	19.6	0.88	677
30	2.37	555.6	658.9	23.4	1.34	557
40	2.03	531.4	635.1	22.1	1.87	446
50	0.38	499.1	603.8	22.4	2.12	520
60	0	455.6	588.0	23.6	2.26	493

distribution. Average grain sizes of the Ti samples annealed at 500 °C for different time was also given in Table 1. The average grain size of Ti with annealing time of 5 min was about 0.29  $\mu\text{m}$ , and it increased to about 2.26  $\mu\text{m}$  when the annealing time was increased to 60 min. Combined the annealed microstructure with the tensile deformation behavior, it is evident that the YPE phenomenon is closely related with the annealing time, further associated with the characteristics of grain size, i.e. the average grain size and grain size distribution in the Ti samples.

In order to further understand the mechanism of the YPE phenomenon, the microstructures of Ti samples after annealed at 500 °C for 30 min and tensile tested for a plastic strain of 3% were investigated by TEM observations. As for the 500 °C/30 min annealed samples, the plastic strain of 3% corresponds to the end of the YPE (Fig. 1). The typical TEM image of the tensile tested samples for a plastic strain of 3% is given in Fig. 3. There were relatively larger and smaller grains, and a large number of dislocations at or near grain boundary or within the grain, but no twins and stacking faults were observed. Thus, the YPE phenomenon in this FG HCP Ti was not associated with twins. In the study by Barnett et al. [8] on HCP magnesium alloy, twins initiate twinning events in neighboring grains and twinning spreads its way over the sample during YPE. Therefore, YPE of FG Ti in the present study is different from that of the magnesium alloy. Generally, YPE in BCC iron and steels was explained by the effect of carbon or nitrogen atmospheres around dislocations [13]. However, in the present study, the solubility of carbon or nitrogen in pure Ti was too limited to cause this effect. In addition, the content of oxygen in tensile tested Ti samples was expected to have no change after short time annealing at 500 °C; since Ti possesses excellent oxidation resistance due to the highly protective surface oxide film and the surfaces of Ti samples were polished before tests. Furthermore, it is impossible that the content of oxygen has the same variation regularity with the length of YPE, i.e. increase with the growth of grain (corresponding to annealing time) and then decrease with the further enlargement of grain size. Therefore, the YPE phenomenon in this study was expected to have no relation with oxygen as well as other impurity atoms, such as carbon and nitrogen in pure Ti.

Comparing Fig. 2c with Fig. 3, it can be seen that the plastic strain of 3% (YPE) led to significant multiplication of dislocations in the FG Ti. In addition, the TEM observations revealed that the average size and distribution of grains had no obvious variation after YPE. Furthermore, the YPE in this Ti occurred only when the grain size possessed certain characteristics, i.e. appropriate average value and distribution. Therefore, it is reasonable to conclude that the YPE in this FG Ti was attributed to the dislocation behavior and characteristics of grain size.

The effects of grain size on YPE phenomenon have been recognized in the study of other metals. The research by Lloyd and Morris [14] showed that the YPE in an FG Al alloy is inversely dependent on grain size. However, in this FG Ti, the length of YPE began to decrease when the average grain size was reduced to smaller than 0.88  $\mu\text{m}$ , and the YPE phenomenon disappeared when the average grain size was smaller than 0.44  $\mu\text{m}$ .

In this study, the as-annealed Ti samples possessed a very low dislocation density. When it was subjected to tensile stress, the Ti samples displayed yielding accompanied by rapid multiplication of dislocations. In this yielding stage, it is assumed that the dislocation behaviors led to plastic flow without increase of tensile stress when the grain size possessed certain characteristics; therefore, the YPE phenomenon occurred. However, the specific interaction between dislocations and grains, which led to the YPE phenomenon, is still not very clear. Further systemic

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