Mechanical properties and deformation modes of grade 1 commercially pure titanium at cryogenic temperature

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Abstract. In this study, we have investigated the deformation behaviour of Grade 1 commercially pure titanium at 20°C, -100°C and -170°C. Optical microscope and electron backscatter diffraction were applied to characterise the twins activated during tensile testing and the crystal orientations of the sample, and further to analyse the orientation dependent slip activity. The higher density of twins was exhibited at lower deformation temperature. The twinning activity was also higher in the parallel loading (RD) than transverse loading (TD) to rolling direction. According to the Schmid factor analysis, the grains were most favourable for <a>-prism slip in the RD sample, whilst the large fraction of grains was favourable for <a>-basal slip in the TD samples. The activation of slip systems is anticipated to be changed with decreasing temperature due to the different effect of temperature on critical resolved shear stress between slip systems. The work-hardening was affected by twinning- and dislocation-induced hardening depending on the temperature and crystal texture. Consequently, the high work-hardening capacity increased or maintained the total elongation (EL) with decreasing temperature (EL: 66% vs. 70% (RD) and 55% vs. 51% (TD) between 20°C and -170°C).

1 Introduction

Titanium and its alloys are considered as the advanced materials for the cryogenic applications, such as aerospace-craft and cryogenic fuel storage tank, due to their low ductile-to-brittle transition temperature and excellent mechanical properties at cryogenic temperature [1]. Prior studies showed the superior ductility of (near) α-Ti at cryogenic temperature compared to that at room temperature [2, 3], and the deformation twinning significantly increased with decreasing temperature. As the twinning-induced grain refinement and lattice reorientation can change the mechanical properties of titanium, it has been suggested that the cryogenic processing techniques have a potential for enhancing processing and mechanical properties of titanium with excellent mechanical properties.

The underlying deformation mechanism of α-Ti is largely ascribed to its low crystallographic symmetry inherent to the hexagonal close packed (HCP) structure compared to the other cubic metals [4]. According to the Von Mises criterion [5], the plastic deformation of polycrystalline materials requires at least five independent deformation systems.
modes to accommodate an arbitrary imposed strains. The $\langle a \rangle$-dislocation slip on prismatic and basal planes are commonly activated due to the lowest critical resolved shear stress (CRSS) [6], but these two slip systems only offer four independent deformation modes. Therefore, the $\langle c+a \rangle$-dislocation slip on pyramidal planes and twinning must be activated to deform the polycrystalline titanium.

The activation of deformation modes is significantly affected by the CRSS for the operating dislocation slip or twinning systems. It has been reported that CRSS for dislocation slip rapidly increases with decreasing temperature because of the increase of lattice friction, whereas CRSS for twinning is relatively insensitive [3, 7]. For that reason, deformation twinning was promoted at cryogenic temperature, and meanwhile dislocation slip is still operative [8]. However, CRSS values for deformation modes of $\alpha$-Ti were evaluated at low temperature range via macroscopic experiments using the bulk samples with microstructural uncertainty, which suggests that the increment of CRSS for dislocation slip is different between individual slip systems. The temperature dependent CRSS for deformation modes complicates the fundamental understanding of deformation mechanism of $\alpha$-Ti, and there still remain many questions concerning the twinning and slip activity at cryogenic temperature.

The purpose of this study is therefore to examine the cryogenic deformation behaviour in polycrystalline $\alpha$-titanium regarding to the crystal texture. The uniaxial tensile experiments were performed within the cryostat at 20°C, -100°C and -170°C. The twins in deformed microstructure were observed by optical microscopy (OM), and the favourable slip systems in the grain orientation analysed with the EBSD measurement. The effects of temperature and crystal texture on work-hardening behaviour were described by the twinning- and dislocation-induced hardening mechanisms.

2 Experimental methods

The microstructure and crystal texture of as-received Grade 1 CP-Ti plate with thickness of 1 mm were characterised by electron backscatter diffraction (EBSD) technique, as shown in Fig. 1. A coupon sample with a size of 10 × 10 mm was sectioned from the rolled plate for the microstructural analysis using EBSD. The sample surface was ground on SiC paper to 1200 grit, and subsequently electropolished within an etchant of CH$_3$OH (400 ml) + C$_8$H$_{14}$O$_2$ (245 ml) + HClO$_4$ 60% (40 ml) using a LectroPol-5 (Struers) with 22 voltage for 45 seconds at room temperature. EBSD measurement was conducted in a FE-SEM (Hitachi SU5000) using a velocity EBSD camera (EDAX). The map area of 1000 × 1000 μm$^2$ was scanned with a step size of 1 μm and an accelerate voltage of 15 kV. The measured EBSD data was processed with a confidence index of > 0.1 using OIM 8.6 software.

![Fig. 1.](image)

(a) Inverse pole figure map (IPF) and (b) pole figures (PFs) showing the microstructure and texture of as-received Grade 1 CP-Ti plate.

The dog-bone shaped samples with a dimension of 6 mm in width and 25 mm in length were sectioned parallel (RD) and transverse (TD) to the rolled direction, and stretched to
fracture at a strain rate of $1 \times 10^{-3}$ s$^{-1}$ and temperatures of 20°C, -100°C and -170°C. For the tensile tests at low (-100°C) and cryogenic temperature (-170°C), the tensile samples were performed within the cryostat equipped with the proportional integral differential (PID) temperature controller, liquid nitrogen (LN$_2$) injection system and air circulator. The LN$_2$ was injected from the LN$_2$ nozzle and spread by the air circulator to lower the temperature in the cryostat. Once the temperature reached to the target temperatures (i.e., -100°C or -170°C), it was stabilised by the PID temperature controller. The tensile sample was held in the cryogenic conditions for ~10 minutes. In the holding period, the load was automatically controlled to zero using 'load zero function' before the tensile load was imposed, because the undesired deformation can be occurred due to the material contraction at low temperature.

The regions of fracture samples ~10 mm away from the fractured point were cross-sectioned to examine the deformed microstructure. The cross-sectional surface was ground and polished using a solution mixed with hydrogen peroxide and colloidal silica (0.04 μm) in ratio of 1:5. The polished surface was etched using the Kroll's reagent. An optical microscope (Carl Zeiss, Axio SCOPE A.1) with polarised light filter was used for the observation of microstructure.

### 3 Results and discussion

The equiaxed grain shapes were exhibited with a mean grain size of 13.4 μm in the EBSD map (see Fig. 1(a)), and the (0001) pole figure (see Fig. 1(b)) displayed a typical rolling texture of rolled α-Ti plate, in which the c-axes of hcp lattices tilted about 30-40° from normal direction (ND) in rolled plane toward TD axis [8]. The different crystal orientations are determined with respect to the applied loading directions, and it is anticipated that the deformation behaviour is significantly dependent on the initial crystal texture.

Fig. 2. shows the tensile stress-strain behaviours for the loading conditions (RD and TD) and deformation temperatures (20°C, -100°C and -170°C). The average mechanical properties were listed in Table. 1. The yield strength (YS) and ultimate tensile strength (UTS) increased with decreasing temperature. The YS is higher in the TD than RD samples, but the difference between YS and UTS is higher in the RD than TD samples, and it is more significant at lower temperature. This indicates that the work-hardening capacity is significantly affected by the crystal texture and temperature conditions.

![Fig. 2. Engineering tensile stress-strain curves for the (a) RD and (b) TD loading directions and deformation temperatures of 20°C, -100°C and -170°C.](image)

In Fig. 3., the hardening rate was calculated as a function of true stress. The initiation of necking was determined according to Considere's criterion. The hardening rate remained higher in the RD than TD samples over the plastic flow region, and also increased with decreasing temperature. The onset of necking occurred at the last stage of plastic deformation.
at relatively low temperature of -100℃ (RD) and -170℃ (RD/TD), whilst it was early occurred at relatively high temperature of 20℃ (RD/TD) and -100℃ (TD).

Table. 1. Tensile mechanical properties of Grade 1 CP-Ti for the RD and TD loading directions and 20℃, -100℃ and -170℃; the average yield strength, ultimate tensile strength and tensile elongation are denoted as YS, UTS and EL, respectively.

| Temperature | YS (σ0.2)   | UTS             | EL       |
|-------------|-------------|-----------------|----------|
| RD/TD       |             |                 |          |
| 20℃         | 149/194 MPa | 282/273 MPa     | 66/55%   |
| -100℃       | 188/201 MPa | 450/380 MPa     | 71/39%   |
| -170℃       | 227/294 MPa | 608/470 MPa     | 70/51%   |

In general, the high work-hardening capacity contributes to the resistance to necking localisation under tension, and thereby increasing the ductility of materials [3, 9]. Compared to the result at 20℃ (see Fig. 2. and Table. 1), the total elongation (EL) was rather increased in the RD samples at -100℃ and -170℃. For the TD, the ductility deteriorated at -100℃, but at -170℃, it was comparable to that at room temperature. The results are contrary to the most of metals showing the ductile-to-brittle transition at low (or cryogenic) temperature. Therefore, the hardening mechanisms of α-Ti should be discussed for the effect of crystal texture and (cryogenic) temperature.

![Fig. 3. Work-hardening rate vs. true strain curves for the (a) RD and (b) TD loading direction and temperature of 20℃, -100 and -170℃; the onset of necking according to Considere's criterion corresponds to the data of open circle.](image)

In order to elucidate the hardening mechanism, the twins were observed in the fractured samples, as shown in Fig. 4. The total elongation was rather equivalent in each of RD and TD samples; except for that of TD sample at -100℃. This indicates that the analysis of twins is comparative in each loading direction for the impose plastic strain. The density of twins was higher at lower temperature. The twinning-induced lattice reorientation introduces the new grain boundaries into their parent matrices. The twin boundaries act as the obstacles for the dislocation glide and contribute to the work-hardening in terms of the Hall-Petch relationship. Therefore, it is evident that the enhanced twinning activity delayed the initiation of necking due to the high twinning-induced hardening at low temperature.

As the density of twins was approximated in the fractured RD and TD samples with different imposed strains, it is rather difficult to conclude that the twinning activity depends on the crystal texture. However, at room temperature, although the imposed strain is 11% higher in the RD than TD sample, the density of twins was seen to be comparable with each other. Note that the work-hardening behaviour is stronger in RD than TD sample at 20℃ (see Fig. 3). This suggests that other hardening mechanism governed the deformation of Grade 1 CP-Ti together with twinning mechanism, depending on the crystal texture.
One of the hardening mechanisms is dislocation hardening. The CRSS and Schmid factor \( m \) are two main factors to determine the activation of slip systems [10]. The \( m \) value is defined as follow:

\[
m = \cos \varphi \cdot \cos \lambda
\]  

where, \( \varphi \) and \( \lambda \) are the angles between the applied stress and slip plane normal, and the applied stress and dislocation glide direction (i.e., slip direction), respectively. The \( \langle a \rangle \)-prism and \( \langle b \rangle \)-basal slip are most easily activated in the \( \alpha \)-Ti due to their low CRSS values (the ratio of 1:1.2 [6]), and the \( m \) value mainly depends on the inclination angle (\( \theta \)) between c-axis of hcp structure and applied tensile loading direction (LD). Thus, in Fig. 5, the maximum \( m \) value for \( \langle a \rangle \)-prism and \( \langle b \rangle \)-basal slip was calculated as a function of \( \theta \) angle of grain orientation, and the fraction of grain orientations was analysed in the EBSD data of Fig. 1(a) with respect to the angle relationship between the \( \theta \) angle and RD/TD loading direction.

According to the SF analysis, the SF distribution for \( \langle a \rangle \)-prism slip is higher above the \( \theta \) angle of \( \sim 65^\circ \) relative to that of \( \langle b \rangle \)-basal slip. In the RD, the most of grains are oriented to favour the \( \langle a \rangle \)-prism slip, whilst further the \( \langle a \rangle \)-basal slip in the TD sample. These two slip systems showed different dislocation glide motions [6, 11]. Furthermore, the dislocations
would be more accumulated in deformation at low temperature, due to the suppressed dynamic recovery [12]. This explains the higher work-hardening behaviour at lower temperature (see Fig. 3). It was also reported that the CRSS for slip systems depends on the deformation temperature [13]. However, there still remain several questions for the rigorous evaluations of CRSS in deformation modes at subzero temperatures.

4 Summary

In the present study, we have performed the tensile experiment in the cryostat to explore the deformation of rolled Grade 1 CP-Ti at cryogenic temperature. The total elongation between room and low (or cryogenic) temperature was comparable with each other. The work-hardening behaviour became stronger with decreasing temperature. The onset of necking is likely delayed due to high hardening capacity, thereby showing no ductile-to-brittle transition at low temperature. Meanwhile, the work-hardening was larger in the RD than TD samples, and the elongation was improved in the RD samples with increase of work-hardening capacity. The OM observations demonstrated that the enhanced twinning activity increases the work-hardening with decreasing temperature. In addition, the orientation dependent slip activity was analysed by EBSD measurement, and it is suggested that the anisotropic work-hardening behaviour is also related to the activation of slip systems in polycrystalline α-titanium, depending on the temperature and crystal texture.

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