Mechanical properties of cryo-rolled commercially pure titanium

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Abstract. Commercial pure Titanium (CP-Ti) was processed by cryo-rolling with a thickness reduction of 20% and 50% at a cryogenic temperature (T = –196°C). It was found that cryo-rolling to the total reduction of thickness 50% leads to form ultra-fine grains. The results showed that strength and hardness were enhanced by decreasing grain size. Both strength and hardness of the cryo-rolled CP-Ti were noticed higher than as-received material. The mechanical properties of the cryo-rolled 20% and 50% were significantly increased: the yield strength by 9% and 45%, and the ultimate tensile strength by 26% and 60%, respectively, compared to as-received CP-Ti. While the elongation was decreased by 38% and 78% compared with the as-received CP-Ti.

Keywords: Commercially pure titanium; Cryo-rolling; Ultra-fine grain; Mechanical properties; Nano-indentation

1. Introduction
Titanium and titanium alloys are introduced in many applications such as aerospace engineering and biomedical technology. Commercially pure Titanium (CP-Ti) has good corrosion resistance and biocompatibility than commonly used titanium alloys [1, 2]. Titanium and its alloys have the highest strength to weight ratio. CP-Ti shows high ductility before fracture. Based on the Hall–Petch law, strength and ductility can be improved by successful applications such as grain refinement [3].

Severe plastic deformations (SPDs) is an effective process to fabricate materials with an ultra-fine grain (UFG, 100 nm < grain size <1000 nm) or nano-grain (NG, grain size <100 nm). Many techniques have been introduced to synthesize materials with UFG or NG, including cold rolling at low temperature, equal channel angular pressing (ECAP), multiple forging (MF), high-pressure torsion (HPT), ball milling (BM), accumulative roll bonding (ARB), hydrostatic extrusion (HE), and other processes [4-7]. The cold rolling process can be used to obtain a high degree of deformation. However, cold rolling of the CP-Ti at room temperature can cause dynamic recovery because of rising temperature during the rolling process. Dynamic recovery phenomenon is associated with rearrangement of dislocations, leading to a suitable
boundary configuration[8]. The dislocation climb mechanism can entangle by decreasing vacancy concentration. Thus, material deformation at low temperature or sub-zero temperature can inhibit dynamic recovery by adjusting dislocations and subsequent annihilation at grain boundaries. Cryo-rolling deformation at -196 °C has been proved to be an effective method to improve yield strength and tensile strength for various metals such as Ti, V, Al, and Zr [8-11].

This work has studied the severe plastic deformation (SPD) process through cryo-rolling to refine the CP-Ti grain size. Furthermore, the CP-Ti has investigated after processing by cryo-rolling, considering several deformation reductions of thickness, which may produce different mechanical behavior under tensile. The microstructure and mechanical properties of the cryo-rolled samples were studied.

2. Material
Commercial pure titanium (CP-Ti, grade-2, ASTM B265) with a purity level of 99.45% was used in this study. The chemical composition and mechanical properties are listed in tables 1 and 2, respectively.

Table 1. Chemical compositions of the CP-Ti [12].

| Chemical composition (wt. %) | C   | Fe  | O₂  | N   | Ti  |
|-----------------------------|-----|-----|-----|-----|-----|
|                             | 0.02| 0.13| 0.13| <0.01| Bal |

Table 2. Mechanical properties of the CP-Ti [12].

| Yield Strength (MPa) | Ultimate tensile stress (MPa) | Elongation (%) |
|----------------------|-------------------------------|---------------|
| 332-365              | 431-488                       | 31-36         |

3. Experimental Procedures
The dimensions of as-received CP-Ti plates were (5±0.05 mm × 25 mm × 70 mm) in thickness, width, and length. The plates were socked in liquid nitrogen (LN) container for 20 minutes before cryo-rolling. The LN was used to achieve cryogenic temperature. The plates were immediately rolled after being removed from LN, as shown schematically in Figure 1. The cryo-rolling was performed without lubricate using a STANAT roller mill with a diameter of 101.6 mm and a rotation speed of 24 rpm. The samples were processed in a single pass with a total reduction of 20% and 50%.

![Figure 1. Schematic of the cryo-rolling process.](image_url)
The microstructure of the materials was characterized by an optical microscope (OM). X-ray diffraction (XRD) analysis of the samples was recorded in a range of 30-80° using a Siemens D500 at 40 kV and 30 mA with a Cu Kα radiation source. Nano-indentation and tensile tests on an Agilent G200 and an MTS were carried out at room temperature to study the cryo-rolled materials' mechanical properties. A tensile test was performed using displacement-control mode with a cross-head speed of 0.5 mm/min. Tensile specimens with 25mm gauge length were machined by an electric discharge machine (EDM) in the rolling direction (RD)[12].

4. Results and discussion

4.1 Microstructure of cryo-rolled samples

The present study proves that cryo-rolling is useful in synthesizing the grain refinement of the CP-Ti. Figure 2 shows the optical micrographs(OM) of the as-received CP-Ti and after cryo-rolled to 20%, respectively. The as-received CP-Ti shows equiaxed recrystallized grains with an average grain size of 14μm (Fig. 2a). In addition, it can be seen twins in some grains. After a 20 % reduction of thickness, the microstructure changes significantly (Fig. 2b). The grain size has refined, and many dislocation tangles can be noticed within most grains compared with as-received CP-Ti. The twinning was produced from most of the grains, and some were involved in secondary twinning. During the cryo-rolling, dislocations increases as a result of increasing the strain. Dasgupta et al. [8] reported dislocation accumulation with the increase of cold work. D’yakonov et al. [13] studied the microstructure evolution of commercial-purity titanium during cryorolling and obtained increasing dislocation gliding with increasing strain.

![Figure 2. Optical micrographs of the CP-Ti microstructure: (a) as-received CP-Ti; (b) after 20% cryo-rolling.](image-url)
Figure 3 shows the XRD pattern of the as-received CP-Ti, 20%, and 50% cryo-rolled. All diffraction peaks are related to $\alpha$-Ti. The diffraction peak locations remain unchanged after cryo-rolling, while some peaks' intensity shows a noticeable change. The peaks are broader and lower in intensity due to large amounts of strains introduced into the lattice during cryo-rolling. Wu et al. [4] reported peaks broadening following plastic deformation. The relative height of the basal plane (002) decreased significantly after cryo-rolling. This indicates that the material shows the evaluation of texture. The peak intensity referred to the relative number of crystal planes parallel to the surface. Also, it can prove that there was no phase transformation during cryo-rolling. Zherebtsov et al. [14] studied the formation of nanostructures in commercial-purity titanium via cryorolling. They obtained texture evaluation after cryo-rolling.

![Figure 3. XRD patterns of the as-received CP-Ti, 20% and 50% cryo-rolled.](image)

4.2. Hardness measurements
Nano-indentation measurements were conducted on polished samples. Berkovich indenter was used with a test load of 30gf and a dwell time of 15s. Figure 4 shows the room temperature results of the nano-indentation measurements of the as-received CP-Ti, 20% and 50% cryo-rolled. The result shows that the hardness values were significantly increased after cryo-rolling. The average hardness was 2.21, 2.32, and 3.19 GPa for the as-received CP-Ti, 20% and 50% cryo-rolled, respectively. The hardness of 20% and 50%...
cryo-rolled has increased by about 5% and 44%, respectively, compared to the as-received CP-Ti. The difference in the thickness reduction causes a quite difference in the indentation hardness values. This is assigned to grain refinement and high dislocation density during cryo-rolling. Sergueeva et al. [1] studied advanced mechanical properties of pure titanium with ultrafine-grained structure. They observed that the hardness of CP Ti was increased with grains refinement.

![Figure 4. Nano-indentation measurements of the as-received CP-Ti, 20% and 50% cryo-rolled.](image)

**4.3. Tensile test measurements**

Figure 5 shows the stress-strain curves of the as-received CP-Ti, 20% and 50% cryo-rolled. The results show that the ultimate tensile strength (UTS) and 0.2% yield strength (YS) were enhanced following cryo-rolling compared to the initial CP-Ti. The cryo-rolled materials (20% and 50%) showed ~ 9% and 45% higher YS and ~ 26% and 60% higher UTS than the as-received CP-Ti. The elongation of the cryo-rolled samples decreased by 38% and 78% for the 20% and 50% cryo-rolled, respectively. The remarkable increases in the UTS and YS were associated with grain refinement and high dislocation density during cryo-rolling. The UFG metals produced by cryo-rolling has been studied. The significant increase of YS has been related to the Hall-Petch (H-P) relation,

\[
\sigma_{ys} = \sigma_0 + kd^{-1/2}
\]

where \(\sigma_{ys}\) is the yield stress, \(\sigma_0\) is a material constant (dislocation motion movement resistance), \(k\) is Petch parameter (the strengthening coefficient), and \(d\) is average grain size[1, 9]. This work shows that
the grain size has been refined after 20\% and 50\% cryo-rolling (Fig.2b). In addition, the XRD pattern (Fig. 3) exhibits a remarkable increase in peak width, which demonstrated the grain refinement and higher dislocation density. Zherebtsov et al. [14] reported increasing CP-Ti mechanical properties with an increase of deformation strain following cryo-rolling. The tensile results agree with the results reported in the literature for CP-Ti titanium with ultrafine-grained structure [1, 4, 15].

Figure 5. Stress-strain curves of the as-received CP-Ti, 20\% and 50\% cryo-rolled.

5. Conclusion
From the strength and hardness evaluation of cryo-rolled at −196 °C of the CP-Ti, the following conclusions were drawn:

1. Grain refinement of the CP-Ti was obtained using the cryo-rolling process, which conforms with the Hall-Petch relation.
2. XRD analysis showed peaks broader with an increase of the amounts of strains cryo-rolling.
3. The plasticity evaluation from nano-indentation measurements shows that the hardening of the CP-Ti corresponds with the grain size.
4. The hardness of 20\% and 50\% cryo-rolled has increased by about 5\% and 44\%, respectively, compared to the as-received CP-Ti.
5. The cryo-rolled 20% and 50% mechanical properties were enhanced: the yield strength by 9% and 45%, and the ultimate tensile strength by 26% and 60% compared with the as-received CP-Ti.

6. The elongation was decreased by 38% and 78% compared with the as-received CP-Ti.

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