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Strength Improvement of Nano-Structured Titanium Processed by Parallel Tubular Channel Angular Pressing

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Abstract: Parallel Tubular Channel Angular Pressing (PTCAP), as a process of Severe Plastic Deformation (SPD), was employed for improving the strength of commercially pure Titanium (Grade 2). In the present research, the tubular samples of pure titanium were severely deformed by one and two passes of PTCAP at the temperature of 450°C. It was found by the results of tensile tests that the yield and ultimate strengths increased by 24% and 29% after applying the second pass of PTCAP, respectively. It was also showed that the Vickers microhardness increased by 46%. Moreover, the micrographs illustrated that the average grain size decreased from ~21 μm in the unprocessed condition to ~143 nm after applying two PTCAP passes. Therefore, applying the technique of PTCAP was successful to produce the nano-structured titanium.

Keywords: Hardness, Microstructure, PTCAP, Strength, Titanium

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Research paper

1 INTRODUCTION

Commercially pure titanium (CP-Ti), as metallic biomaterials, has been used extensively for surgical implants. Titanium is superior to other surgical metals like iron-chromium-nickel alloys (austenitic stainless steels) and cobalt-chromium-based alloys due to high biocompatibility and corrosion resistance. Another favorable property of CP-Ti is that it is more light-weight than other surgical metals. Nevertheless, the main drawback of CP-Ti is its strength. The strength of CP-Ti implants is too low when they are used in load-bearing situations. The addition of alloying elements, such as aluminum and vanadium leads to the significant improvement of titanium's strength. However, the release of aluminum and vanadium ions may cause health problems. Therefore, Al and V ions have aroused concerns about the long-term safety of Ti–6Al–4V alloy implants [1-3].

An alternative method to solve the problem of harmful ion release is to stop using the technique of alloying and improve the strength of pure titanium by nanoscale grain refinement. One of the efficient ways of producing bulk nano-structured materials is the metalworking technique known as Severe Plastic Deformation (SPD). SPD technique is based on the fact that a metal sample is subjected to high plastic strains leading to breaking the coarse grains down into nano-sized (with the size less than 100 nm) grains [4-5].

In order to fabricate nanocrystal line tubes, different SPD methods have been presented. These methods are known as Accumulative Spin Bonding (ASP) [6], High-Pressure Tube Twisting (HPTT) [7], Tubular Channel Angular Pressing (TCAP) [8] and Parallel Tubular Angular Pressing (PTCAP) [9]. PTCAP is a novel method which has some advantages over other methods including the reduction of process force and more strain uniformity in the tube.

Podolskiy et al [10] investigated the microstructure and mechanical properties of CP-Ti by applying equal channel angular pressing (ECAP) at cryogenic temperatures. They achieved higher values of strength and hardness. Also, Zhilyaev et al [11] examined the microstructure and texture homogeneity of ECAP-processed CP-Ti at the elevated temperature of 450°C. They reported that more homogeneous microstructure in mean grain size and texture would be achieved by increasing the pass number of ECAP. To date, there is few research work investigating the microstructure and mechanical properties of CP-Ti processed by PTCAP. The importance and novelty of the research work is to apply the Nano structuring method of PTCAP to CP-Ti at the elevated temperature of 450°C. Successful developments of nanostructured titanium-based medical implants is very important. Nano structuring is a promising technique to further improve the safety, effectiveness and longevity of medical implants made of titanium-based materials. The purpose of present research is to achieve strength improvement in CP-Ti by the Nano structuring technique of PTCAP. For this purpose, tubular samples of titanium were deformed by one and two passes of PTCAP at the temperature of 450°C. Then, the mechanical properties and the microstructure of PTCAP-processed CP-Ti were evaluated by the uniaxial tensile test, micro hardness test, optical microscope (OM) and scanning electron microscope (SEM).

2 MATERIAL AND METHODS

2.1. PTCAP Process

Fig. 1 (a) and (b) schematically illustrate the principle of PTCAP process and die parameters in order to produce nano-structured CP-Ti. As shown in "Fig. 1 (a)", PTCAP process consists of two half passes.

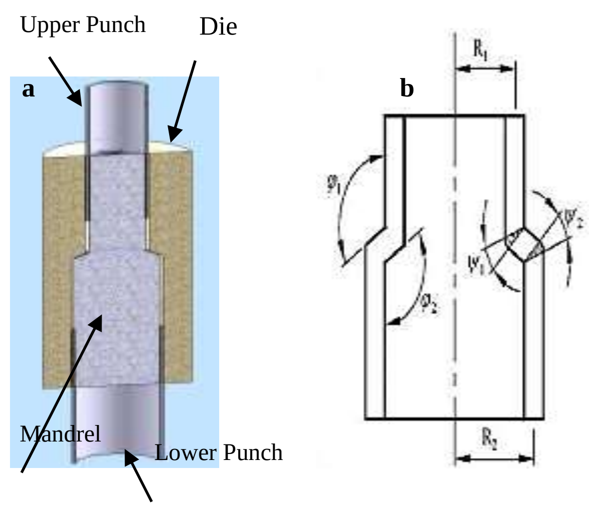

Fig. 1 (a): Schematic illustration of PTCAP process with part label, and (b): Die parameters.

At the first half pass, the tube is pressed into the gap between the mandrel and die including two shear zones in order to enhance the tube diameter to its maximum size. At the second half pass, the tube is pressed back applying the lower punch to reduce the tube diameter to its primary size. The total equivalent strain after N passes of PTCAP can be calculated by the following relationship [9]:

$$\bar{\varepsilon}_{TN} = 2N\left\{\sum_{i=1}^{2}\left[\frac{2\cot\left(\frac{\varphi_i}{2}+\frac{\psi_i}{2}\right)+\Psi_i\csc\left(\frac{\varphi_i}{2}+\frac{\psi_i}{2}\right)}{\sqrt{3}}\right]+\frac{2}{\sqrt{3}}\ln\frac{R_2}{R_1}\right\} \quad (1)$$

Where, φ is the channel angle, ψ is the curvature angle, N is the number of cycles, R_1 is the primary radius and R_2 is the secondary radius.

2.2. Experimental Procedures

The studied alloy in this paper was CP-Ti (grade 2) with the chemical composition shown in "Table 1".

Table 1 Chemical composition of CP-Ti Grade 2 (wt.%)

Ti	Fe	C	N	H	O
Balance	0.053	0.012	0.014	0.002	0.059

Cylindrical tubes of 21 mm in outer diameter, 4 mm in thickness, and 36 mm in length were machined. PTCAP die was fabricated as shown in "Fig. 2 (a)" with the parameters of $\varphi_1 = \varphi_2 = 120°$ and $\psi_1 = \psi_2 = 0$. Two passes of PTCAP process at the temperature of 450°C were conducted using hydraulic press of 50 tons capacity operating at ram speed of 10 mm/s. MoS_2 was also used as lubrication. Infrared thermal camera (IR-384H; Cygnus Electronics) was employed in order to control the temperature of PTCAP process at 450°C. To evaluate the mechanical properties of the samples, tensile test at displacement rate of 1mm/min was conducted according to ASTM E8 using Zwick/Roell-Z250 universal testing machine at room temperature. Tensile test results were reported as the average value of three replicates.

The values of the Vickers micro hardness (HV) were measured according to ASTM E384 using Innovates Nova 240 tester with a load of 2.942 N and a dwell time of 10s. In order to achieve a high degree of accuracy, each reported micro hardness datum was the average of five separate measurements. Metallographic analysis was carried out using an OM (Olympus BX51M) and SEM (S-4800, Hitachi).

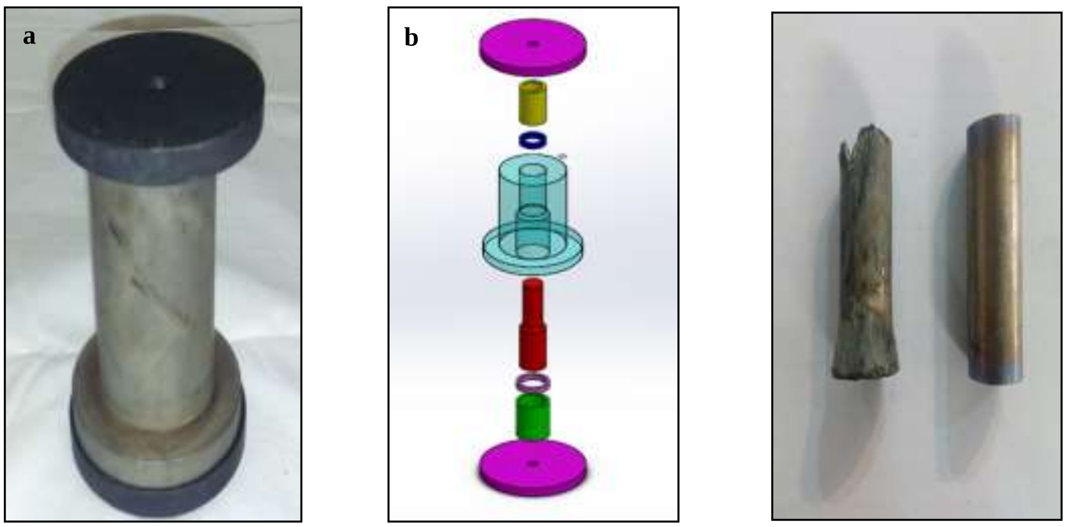

Fig. 2 (a): Fabricated PTCAP die, (b): Exploded view of PTCAP die, and (c): Unprocessed and PTCAP-processed samples.

3 RESULTS AND DISCUSSION

3.1. Tensile and Micro Hardness Tests

Fig. 3 (a) shows the results of tensile tests in 0 pass, 1 pass, and 2 passes of PTCAP process at the temperature of 450°C. As it is seen in "Fig. 3 (a)", there is a satisfactory improvement after two passes of PTCAP with the yield strength increasing from 348 to 449 MPa and the ultimate strength increasing from 508 to 631 MPa. In other words, the yield and ultimate strengths increase by 24% and 29%, respectively. There is also a reasonable ductility in which the elongation decreases from 38% to 27% after two passes of PTCAP process. The same procedure is seen in the results of micro hardness tests. As it is observed in "Fig. 3 (b)", the Vickers micro hardness increases from HV 184 to HV 269 after employing two passes of PTCAP process. In other words, the Vickers micro hardness increases by 46%. "Table 2" summarizes the results of tensile and micro hardness tests.

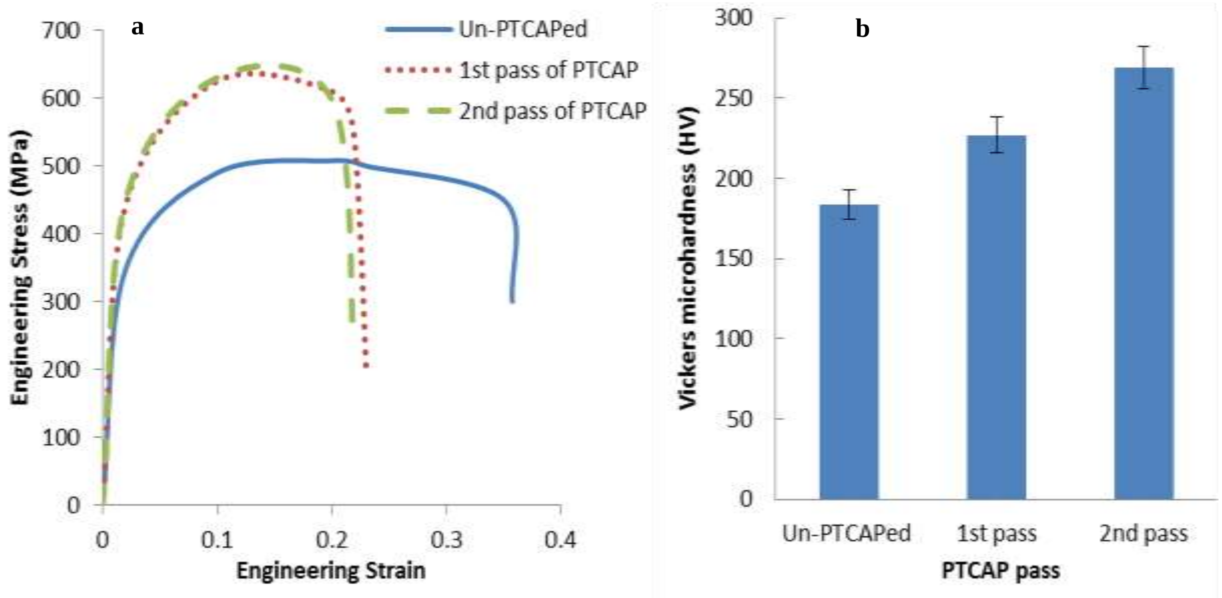

Fig. 3 (a): Engineering stress−strain curves of non-PTCAP and PTCAP-processed specimens, and (b): The average Vickers micro hardness of non-PTCAP and PTCAP-processed specimens.

Table 2 Mechanical properties of CP-Ti before and after PTCAP process

PTCAP pass	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)	Vickers micro hardness (HV)
0	348	508	38	184
1	442	625	29	227
2	449	631	27	269

3.2. Microstructure Analysis

The significant improvement in the mechanical properties of SPD-processed material is mainly attributed to the grain refinement [12-13] and high-density dislocations [14-15]. In accordance with the Hall−Petch equation, the yield stress, as a criterion for the strength of polycrystalline materials, increases considerably by the grain refinement. The Hall-Petch equation is defined according to following relationship [16]:

$$\sigma_y = \sigma_0 + k_y d^{-1/2} \quad (2)$$

Where, σ_y is the yield stress, σ_0 is the friction stress, k_y is the constant of yielding, and d is the grain size.

In “Fig. 4”, the microstructures of CP-Ti before and after applying the PTCAP process are depicted, which the images are obtained by OM and SEM, respectively. In order to identify the behavior of materials, the best way is to examine the microscopic structure [17-19]. It can be observed from “Fig. 4 (a) and (b)” that the grains are refined by PTCAP technique. The magnitude of the average grain size (d) evolved from ∼21 μm in un-PTCAPed material down to ∼143 nm after employing 2 passes of PTCAP process. Therefore, with respect to Hall-Petch relationship (“Eq. (2)”), the improvement in the strength of CP-Ti is mainly attributed to the nano-sized grains. Another reason of strength improvement in nanostructured titanium could be the existence of high-density dislocations. When the pass number of SPD process increases, accumulative strain as well as dislocations continuously increase. As long as a dislocation cell is newly created, it can absorb the high-density dislocations inside the newborn dislocation cell, and then transfer to the cell wall, therefore the whole dislocation cell comes into being. When these dislocation cells turn into high-angle boundaries, the microstructure could be further refined [20].

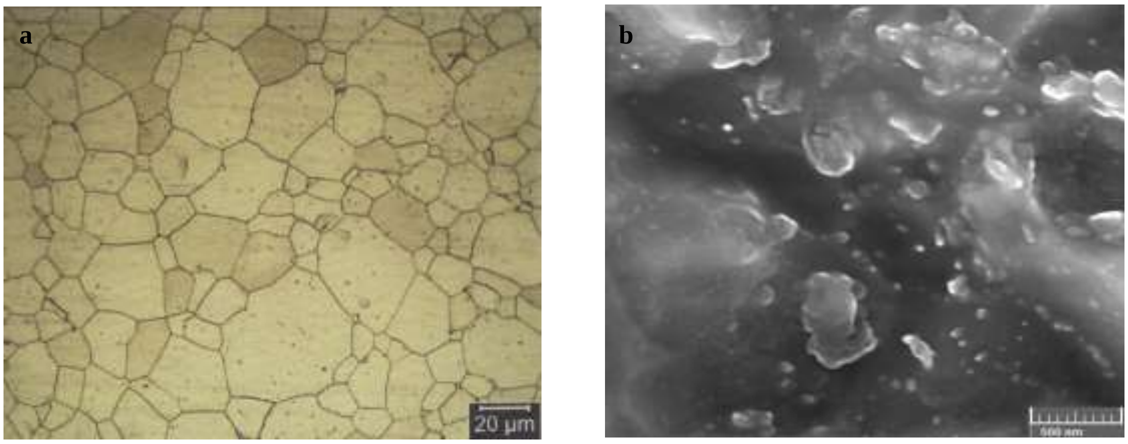

Fig. 4 (a): OM image of specimen before PTCAP process, and (b): SEM image of specimen after two passes of PTCAP process.

Fracture surfaces of non-PTCAP and PTCAP-processed samples after tensile tests are also shown in “Fig. 5 (a) and (b)”. According to “Fig. 5 (a)”, SEM morpholog shows typical ductile failure with the large-sized dimples. The dimples originate from nucleation, growth and coalescence of micro voids under tensile stresses. It is observed that the fracture surface of non-PTCAP-processed sample includes deep dimples. As it is shown in “Fig. 5 (b)”, by applying two passes of PTCAP process, due to the severe deformation, the fracture surface of CP-Ti consists of fine dimples as compare to unprocessed one. Moreover, it is noted that the depth of dimples is reduced in PTCAP-processed sample.

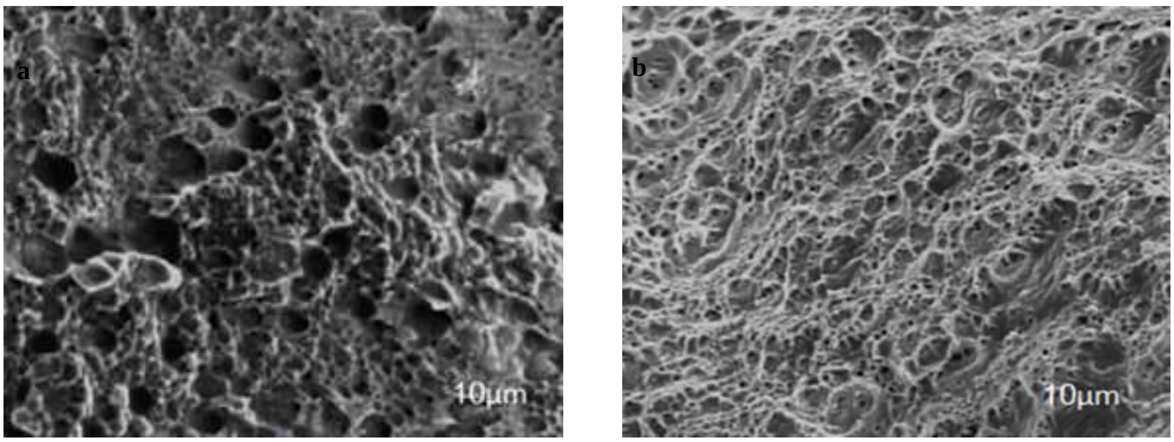

Fig. 5 SEM micrograph of fracture surface in CP-Ti: (a): before PTCAP process, and (b): after two passes of PTCAP process.

4 CONCLUSION

In this research work, tubular samples of CP-Ti (grade 2) were successfully subjected to one and two passes of PTCAP process at the temperature of 450°C. The following conclusions were drawn:

1- The yield and ultimate strengths increased by 24% and 29% after applying two passes of PTCAP process, respectively.

2- The elongation decreased from 38% in the unprocessed condition to 27% after two passes of PTCAP process.

3- The Vickers micro hardness increased from HV 184 in the unprocessed condition to HV 269 after employing two passes of PTCAP process.

4- The average grain size decreased from ∼21 μm in the unprocessed condition to ∼143 nm after applying two PTCAP passes.

5- The strength improvement in CP-Ti was mainly attributed to the existence of Nano-sized grains.

6- Fracture surface analysis demonstrated that after employing PTCAP process, the number of fine dimples increased considerably.

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