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Improving Mechanical Properties of Ultrafine-Grained Titanium Produced through Warm ECAP Using a Novel Lubrication System

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ABSTRACT: In this research, warm equal channel angular pressing of grade 2 pure titanium was investigated. A new design was proposed for tooling with 90° channel angle, which eliminated problems such as the undesired flow of material between the two die halves, increased forming load and sticking of the sample in the die. A lubrication system consisting of copper foils and molybdenum disulfide was found to be effective after some trials. Using this lubrication system, 4 passes of equal channel angular pressing at 260 °C was completed successfully. Tensile test results, demonstrated after the 4th pass, ultimate tensile strength increased by 57% while the reduction of area decreased by 14%. Reducing the process temperature and using appropriate lubrication, and ultimate tensile strength of 799 MPa was achieved which is well above the values already reported from similar researches. The microstructure and fracture surface of the equal channel angular pressing titanium were investigated using a scanning electron microscope. The results show that the initial grain size of 18 µm reduces to 729 nm after four passes. In addition, the fracture surface of the sample shows the formation of fine uniformly distributed dimples, indicating a homogenous structure of the samples.

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1- Introduction

Due to limited slip systems of Titanium (Ti) at room temperature, Warm Equal Channel Angular Pressing (ECAP) is applied to this material for refining the microstructure and improving its mechanical properties. In Ref. [1], a tensile strength of 760 MPa was achieved for grade-2 Ti after 8 passes of ECAP at 300 °C. Increasing the temperature to 450 °C, after 5 passes, a tensile strength of 700 MPa was obtained for this material [2]. A subsequent cold extrusion step increased the strength to 791 MPa but reduced the elongation by 56% [2]. The highest strength achieved for grade-2 Ti was 791 MPa through warm ECAP with a 90° channel angle at a minimum temperature of 300 °C [3]. This paper presents results for warm ECAP of grade-2 Ti at a temperature as low as 260 °C in an attempt to achieve higher strength.

2- Experimental Procedure

Grade-2 Ti rods of 12 mm in diameter and 80 mm in length were used for warm ECAP. An ECAP die with a channel angle of 90° was designed and fabricated. The die includes upper and lower halves which eliminate the challenges like sticking of the specimen and undesired flash formation. Fig. 1 represents the die used in the experiments. The samples were wrapped with 0.05 mm thick copper foil as a solid lubricant. Additionally, a graphite-molybdenum disulfide lubricant (Molyslip EHT) was applied to further reduce the friction. Warm ECAP was tried at 220, 230 and 260 °C and a punch speed of 0.1 mm/s through route B_c. The recovered samples were subject to tensile test and microstructure characterization using scanning electron microscopy.

3- Results and Discussion

At a temperature of 260 °C, four passes of ECAP was performed on grade-2 Ti rods. The recovered samples, shown in Fig. 2, are free of cracks. At 230 °C only one pass of ECAP was successful and at 220 °C the samples were failed during the first pass.

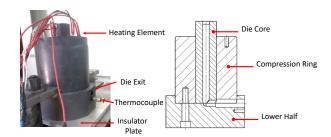


Fig. 1. ECAP die used in the experiments



Fig. 2. Successful ECAP of grade-2 Ti at 260 °C

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Fig. 3 plots the variation of tensile strength and reduction of area for the samples ECAPed at 260 °C. In addition, the relative increase in the tensile strength ($\Delta\sigma_u$) and reduction of area (Δr_A) with respect to raw material are tabulated in Table 1. The results show that the rate of strengthening is the highest (~36%) at the first ECAP pass. The decrease in the rate of strengthening in the subsequent passes could be related to saturation of the dislocations in the crystal structure. The increase in the reduction of area in the last pass could be associated with the formation of subgrains with lower misoriented angles which favors more homogenous structure.

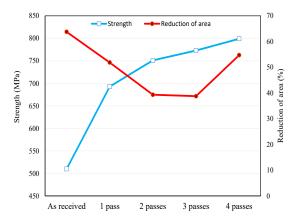


Fig. 3. Tensile strength and reduction of area for ECAPed samples at 260 $^{\circ}\mathrm{C}$

Table 1. Tensile strength and reduction of area for ECAPed samples in comparison to the raw material

| | As received | 1st Pass | 2 nd Pass | $3^{rd}\ Pass$ | 4 th Pass |
|----------------------------|-------------|----------|----------------------|----------------|----------------------|
| σ _u (MPa) | 510 | 693 | 751 | 773 | 799 |
| $\Delta\sigma_{\rm u}$ (%) | 0 | 35.88 | 47.25 | 51.57 | 56.67 |
| r _A (%) | 63.74 | 51.83 | 39.32 | 38.77 | 54.71 |
| Δr_{A} (%) | 0 | -18.69 | -38.31 | -39.17 | -14.17 |

Table 2: Tensile strength achived in this study compared to that of previous works

| | $\Delta \sigma_{\rm u}$ (MPa) | Percent improvement |
|----------------------|-------------------------------|---------------------|
| Present Work | 799 | 0 |
| Purcek et al. [1] | 760 | 4.9 |
| Kang and Kim [2] | 700 | 12.4 |
| Sordi et al. [3] | 790 | 1.1 |
| Stolyarov et al. [4] | 645 | 19.3 |

Comparison of the ultimate tensile strength attained in this study with those of the similar works in Table 2, shows fair improvements.

Scanning electron micrographs of the microstructure and fracture surface of the samples are shown in Figs. 4 and 5. It is observed that the grain size has reduced from $18 \mu m$ to $729 \mu m$ nm after four passes of ECAP this explains the enhancement observed in the tensile strength. In addition, the fracture surface is characterized by dimpled rupture for all samples. With increasing severe plastic deformation the dimples are increased in number and decrease in size which represents a

more homogenous structure.

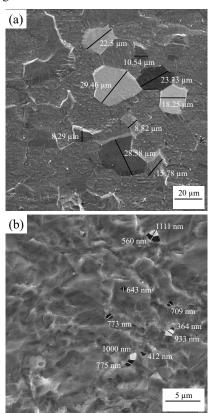


Fig. 4. Scanning electron micrographs of materials in (a) as recived condition and (b) after 4th pass of ECAP

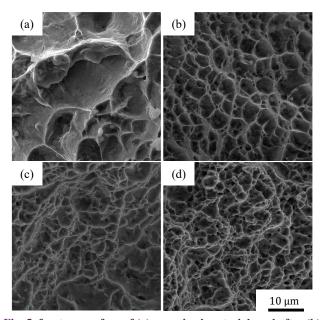


Fig. 5. fracture surface of (a) as recived material, and after (b) 1st, (c) 3rd and (d) 4th pass of ECAP

4- Conclusion

ECAP of grade-2 Ti was unsuccessful at the temperature of 220 °C. At 230 °C only one pass of ECAP was possible. At 260 °C, four passes of ECAP was successfully accomplished which resulted in an ultimate tensile strength of 799 MPa and reduction of the area of ~55%. Characterizations reveal the formation of a homogeneous microstructure with a grain size of 729 nm.

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