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# Comparison of laboratory-scale and industrial-scale equal channel angular pressing of commercial purity titanium



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#### ABSTRACT

This study contrasts the extent to which laboratory and industrial scale variants of equal channel angular pressing (ECAP) impart desirable microstructures and mechanical properties in Grades 2 and 4 titanium. Industrial-scale ECAP-Conform (ECAP-C) with post-ECAP thermo-mechanical processing (TMP) enhanced performance levels beyond those achieved with the same material processed in the laboratory by ECAP only. ECAP-C processed titanium demonstrated exceptional tensile properties and fatigue strength, superior even to conventional Ti-6Al-4V.

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#### 1. Introduction

Development of improved metals and alloys is often driven by requirements for improved mechanical performance. For medical device applications, improving biocompatibility is also required. Titanium and its alloys generally satisfy these requirements and are widely used for production of medical implants [1]. Ti-6Al-4V, the most highly produced Ti alloy, is often used for bone replacement and dental implants [1,2]. However, the potential toxicity of aluminium and vanadium [3,4] favours use of commercially pure titanium as an alternative to Ti-6Al-4V [5-8]. Unfortunately, the absence of the alloying elements leaves commercial purity (CP) grades with lower mechanical strength [2]. One remedy to this drawback is to process CP titanium by severe plastic deformation (SPD) to achieve ultrafine grain structures and associated high strength levels [5-8]. Among the SPD techniques, equal channel angular pressing (ECAP) was shown [5-10] to be particularly efficient in refining the grain structure of CP Ti and substantially improving mechanical properties.

One variant of ECAP, viz. ECAP-Conform, also known as ECAP-C, is capable of producing continuous long rods of titanium, which is essential for large scale manufacturing [11,12]. While laboratory and pilot scale implementations of ECAP-C have demonstrated very good properties [13], it has not heretofore been demonstrated that industrial scale implementation of ECAP-C can accomplish the same. The present work contrasts the effects of laboratory and

industrial implementations of ECAP processing on the mechanical properties of two grades of CP titanium, Grade 2 and Grade 4.

#### 2. Experimental section

Materials: Rods 12 mm in diameter of commercially pure titanium Grade 2 (wt% < 0.01 C, 0.18 Fe, 0.12 O, < 0.01 N, balance Ti) and Grade 4 (wt% 0.04 C, 0.14 Fe, 0.40 O, 0.01 N, balance Ti) were received from TIMET and Dynamet (USA), respectively. These materials were processed by two methods: (I) Equal channel angular pressing (ECAP) with back pressure (at 300 °C with a pressing speed of 1 mm/min via Route B<sub>C</sub> with 90° angle between the die channels) to a total accumulated strain of 460%; and (II) ECAP-Conform (ECAP-C) followed by proprietary thermo-mechanical processing to a total accumulated strain of 510%. The TMP process after ECAP is essential in industrial practice to produce standard commercial rod diameters. Henceforth, these two processing histories will be referred to as ECAP (I) and ECAP(II). Samples produced in this study are designated by their grade and processing history, for example, Gr2ECAP(I) for a sample of Grade 2 CP titanium subjected to the ECAP(I) processing conditions. The diameters of samples after ECAP(I) and ECAP(II) were 10 mm and 6 mm, respectively.

*Microstructural analysis*: Samples for microstructural analysis with thickness of 2 mm were cut from titanium rods using a Buehler low-speed saw. Metallographic surfaces for optical microscopy were polished to a mirror-polished condition by grinding with 1200 and 2400 grit papers, followed by polishing with a mixture of 90% colloidal silica (Struers OP-S) and 10% hydrogen peroxide. The microstructures

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of the as-received coarse-grained materials were examined in polarised light using a Leica DMILM optical microscope with a Leica DFC 290 camera.

Transmission electron microscopy (TEM) was performed with a Philips CM 20 microscope operated at 200 kV. Specimens for the TEM study, typically  $\approx$  150  $\mu m$  thick, were extracted from transverse

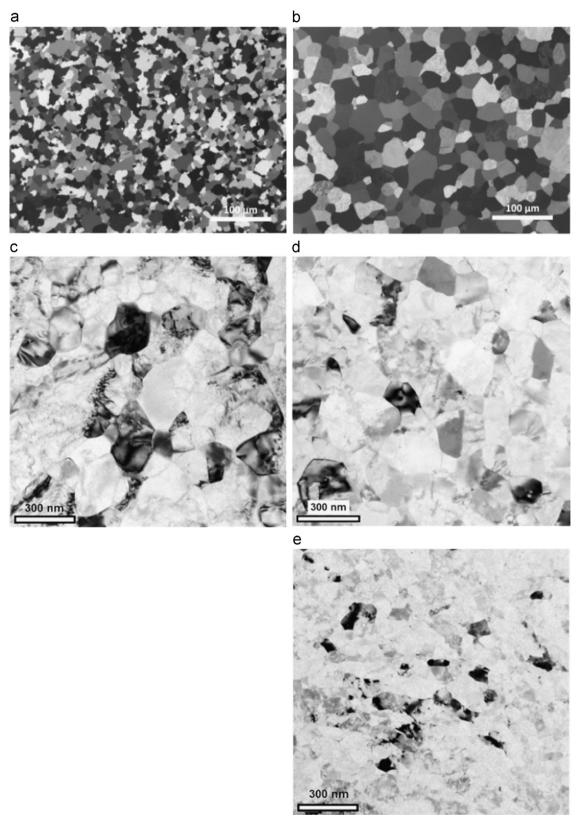


Fig. 1. Microstructure of pure titanium in the following conditions: (a) Grade 2 as-received; (b) Grade 4 as-received; (c) Grade 2 ECAP (l); (d) Grade 4 ECAP (l); (e) Grade 4 ECAP (II). (a-b) OM in polarised light; (c-e) TEM in bright field.

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