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The Effects of Severe Plastic Deformation on some Properties Relevant to Ti Implants

Anibal Andrade Mendes Filho*, Vitor Luiz Sordi, Maurizio Ferrante

*Department of Engineering Materials, Federal University of São Carlos – UFSCAR,
Rod. Washington Luis, Km 235, CEP 13565-905, São Carlos, SP, Brazil*

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In some types of surgical implants, such as bone screws and plates, Grade 2 Ti is seriously considered as a replacement for the Ti-6Al-4V alloy. Advantages are lower cost and the absence of Al and V, which have been identified as potentially harmful to human health. The present paper shows that the lower strength of the commercially pure metal can be enhanced by Severe Plastic Deformation followed by conventional cold rolling, so as to reach a strength level higher than the technical requirements applicable to the alloy. This was ascertained by tensile and Vickers hardness tests from which it was concluded that the best combination of properties are obtained by submitting the metal to Equal Channel Angular Pressing (four passes at 300 °C) followed by a 70% thickness reduction by cold rolling. Although the present results are valid for the material only, and not for the product considered, that is, bone screws, it appears that this solution is a step towards the replacement of the Ti6-4 alloy by Grade 2 Ti, at least for some types of metallic medical implants.

Keywords: *ECAP, cold rolling, titanium, orthopaedic implants, bone screws*

1. Introduction

Surgical implants make intensive use of the well known Ti-6Al-4V alloy. Cost consideration and concerns regarding the effect of Al and V on human health constitute a strong motivation for the replacement of said alloy[1], and a viable candidate is the lower cost Ti Grade 2, from which a better corrosion resistance is expected, although its tensile strength compares unfavorably with that of the alloy, viz. 350 MPa against 1000 MPa.

The main effect of any Severe Plastic Deformation (SPD) technique is the increase of mechanical strength due to grain size reduction, and among those techniques only Equal Channel Angular Pressing (ECAP) is capable of producing billets of commercial dimensions. Besides the Hall-Petch effect, grain size reduction exerts a positive effect on two important requirements of implant materials: i) osteoblasticity, a phenomenon related to the integration of the implant with the surrounding bone tissue[2], and ii) fatigue strength[3].

As for the effects of ECAP on Ti and its alloys, there is a fair amount of information. For instance, a recent paper by Zhao[4] shows that, contrarily to previous assumptions[5], Ti can be deformed at room temperature, and eight passes in a 120° die were thus carried out. This process reduced the grain size from 23 to 0.2 μm and as a consequence the yield strength was raised to 710 MPa, that is, a 160% increase over that of annealed Grade 1 Ti. The authors emphasize that reduction of pressing speed is a key detail to successfully deform Ti at room temperature. However, the literature mentions many instances of higher tensile properties when warm ECAP deformation is followed by cold rolling; Thus Stolyarov[6] and collaborators obtained final yield strength equal to 1020 MPa but only 6% elongation. Recalling that ECAP performed at room temperature resulted in an elongation of 19%[4], this procedure appears to be more advantageous than the sequence - warm ECAP plus cold rolling-except when pressing forces are considered.

The relationship between strain level, grain size and strength is illustrated in a number of papers. For instance[7] it was shown that grain refinement mainly takes place after the first pass, saturation being reached by the sixth and eighth passes. This confirms previous findings by Langdon[8] which were obtained on Al samples. It is important to point out that this effect of the first pass on grain size appears to be a fundamental occurrence, not confined to this or that material. As for the influence of strain level on strength, Table 1, reproduced from a recent investigation, shows a relatively slow evolution of hardness with pass number, except when the first pass is considered[9]. ECAP deformation was carried out at 300 °C in a 120° die, following route B_c; after four passes the grain size was reduced from 28 to 0.25 μm.

To understand the apparent inconsistency between the statement that the grain size is defined by the first pass and the observed increase - although slow - of the hardness, hence of tensile strength, it may be useful to summarize the present knowledge regarding grain refinement mechanisms. The basic model describes the formation of a banded structure of elongated subgrains, which under increasing strain transform into an equiaxed array of grains[10]. This transition from subgrains to grains is in actual fact a transition from low angle boundaries ($2 \leq \xi < 15°$) to high angle boundaries ($\xi \geq 15°$) a phenomenon which is currently under intense study. For instance, using Electron Back-Scattered Diffraction (EBSD) the evolution from low to high angle boundaries was followed on ECAP deformed samples of commercial Al. After one pass the proportion of the latter type of boundaries was 15%, a figure which doubled after

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*e-mail: anibalmendes@gmail.com.br

four passes[11]. Additionally, the same investigation correlated tensile strength with grain boundary character and concluded that strength increases with the proportion of high angle boundaries. This result suggests that such microstructural features, sometimes called “non-equilibrium boundaries”, can be more efficient in limiting plastic flow than low angle boundaries, and a possible explanation is that they would experience difficulties to emit dislocations into the neighboring grain[12]. Of course, final strength is dictated not only by the above described action of the grain boundaries, but also by dislocation hardening. On this respect a recent calculation performed on a ECAP-deformed Al-4%Cu alloy showed that grain boundaries and dislocation hardening, account for 54 and 20% of the total strength, respectively, the balance being attributed to precipitation hardening[13].

Finally, it must be recalled that, besides the positive effects above discussed, grain size reduction to submicron level enhances the corrosion resistance of commercial Ti, as observed by Balyanov et al. on commercially pure Ti[14].

The present work is directed to the upgrading of the mechanical strength of Grade 2 Ti, using a SPD sequence composed by ECAP followed by cold rolling (CR). After a preliminary discussion regarding the effect of such process on Grade 2 Ti tensile behavior, the feasibility of using that material in the manufacture of cortical screws will be analyzed.

2. Experimental

2.1. Material

Grade 2 Ti, with chemical composition complying with the ASTM B 348[15] Standard. From the initial extruded and annealed bar, samples Ø 10 and 70 mm length were machined out and reannealed at 710 °C/2 hours, in order to guarantee a homogeneous grain size.

2.1.1. ECAP deformation and CR

The former was conducted at 300 °C in a $\Phi = 120°$ die constructed with heat resisting steel. ECAP deformation consisted of four passes following Route B_C (sample rotated 90° clockwise between passes) at ram speed of ≈5 mm/min. Additionally a one pass experiment was performed at room temperature. Samples identification scheme is in Table 2.

Additionally, samples in the non-deformed (0X) and ECAP-deformed condition (4XH) were cold rolled up to a thickness reduction equal to 70 and 90%. Identification was the same shown in Table 2, followed by the letters CR; numbers within parenthesis indicate the thickness reduction (70, 90%). Rolling was performed in a PFENN dual rolling mill with a linear speed of 300 mm/s.

2.2. Mechanical tests

2.2.1. Tensile and hardness

Miniature specimens having 8 mm^2 section and 12 mm gauge length were tested in an INSTRON D5500 machine at a deformation rate equal to 10^{-3}/s. The tensile samples were cut along the longitudinal axis of the ECAP billet and along the rolling direction. Data on yield and ultimate strength and on uniform and total elongation are the average of at least two tensile tests per condition. Vickers hardness measurements were performed under a load of 0.20 kN and each value is the average of four measurements.

Table 1. Vickers hardness of Grade 2 Ti after ECAP[9].

Condition	Nominal equivalent strain	HV 20
Coarse-grained	-	145
1 pass	0.66	205
2 passes	1.32	208
3 passes	1.98	213
4 passes	2.64	269

Table 2. Samples identification and ECAP deformation modes.

Sample	Condition
0X	Annealed
1X	Annealed, one pass room temperature
1XH	Annealed, one pass at 300 °C.
4XH	Annealed, four passes at 300 °C.

3. Results and Discussion

Table 3 summarizes the results of the tensile and hardness tests. For each condition differences between maximum and minimum σ_u are less than 5%. Comparison of samples 1X and 1XH shows that, although yield and maximum tensile strength are almost identical, the hot deformed sample exhibits a much higher ductility, viz 22% strain to fracture against 11%. This suggests that room temperature ECAP may not be ideal when compared with warm ECAP, provided that in this latter case deformation temperature is kept below levels which would promote softening mechanisms. On this respect 300 °C seems to be ideal since analysis of the tensile behavior of the present samples suggests only a small amount of recovery[6], but the residual stresses appear to be reduced and as a result strength is unchanged whilst elongation is considerably increased. Studies employing cold rolling as a deformation mode showed that below 600 °C recrystallization is negligible[16] besides, 300 °C corresponds to a homologous temperature close to 0.3, which is insufficient for static recrystallization.

Figure 1 shows ultimate stress (σ_u) and elongation at rupture (ε_t) for each experimental condition. When compared with the coarse-grained Grade 2 Ti, a harmful effect on ductility is observed in all samples subjected to SPD, although values are still above the minimum that ASTM F136[17]specifies for the alloy Ti6-4. Summarizing: when compared with the Ti6-4 alloy all SPD conditions give a higher ductility, and one of them, namely the 4XHCR(70) sample, a higher strength Another important observation is that ductility losses are much higher when DPS does not include ECAP.

Comparison of the present results with literature data is shown in Figure 2. It can be seen that, although the strength of the Ti6-4 alloy has not yet been reached, the combination of properties here obtained is very good. Indeed; whilst the tensile strength obtained by Stolyarov et al.[6,18] is higher, the corresponding ductility is quite low, with total elongation below 10%.

In order to use DPS processed Grade 2 Ti in the manufacture of the cortical screws type HA 3.5, as identified by the ASTM F543 Standard[20] it is necessary to comply with a number of requirements regarding mechanical properties, see the above mentioned Standard. It must be recalled here that F543[20] is a document that, among other guidelines, provides performance considerations and a test method for measuring mechanical properties in torsion of metallic bone screws that are implanted into bone. Thus, the method is intended to measure the uniformity of the product tested, or to compare the mechanical properties of different, yet similarly sized products. These considerations make clear that the present results can only apply to the material; in other words, they intend to be a measure of the effects of SPD techniques on Ti Grade 2, which, eventually can be employed in the manufacture of bone screws. In order to associate the present results with the cortical screws performance requirements, two important parameters of said products must be defined:

- Maximum torque (T_{max})-largest value of torque before screw failure in torsional shear, whose minimum value is equal to 2.3 Nm;
- Breaking angle (θ_f)-angle of rotation when the screw fails in tension; it is related to the material ductility; minimum angle is 180°.

From tensile tests data it is possible to calculate torsional properties related to strength and ductility, making use of the following Equations 1 and 2[21]:

$$\sigma_u = \frac{3\sqrt{3}\ T_u}{2\pi\ (D/2)^3} \tag{1}$$

$$\varepsilon_{eq} = \frac{D\ \theta}{2\ L\ \sqrt{3}} \tag{2}$$

where σ_u is the equivalent maximum tensile stress, T_u the maximum torque, ε_{eq} the von Mises strain, D the torsion test cylindrical sample diameter and L its gauge length. When $\varepsilon_{eq} = \varepsilon_{uniform}$, the angle θ is equal to the angle corresponding to T_u, and here will be named twist angle (θ_u). It must be pointed out that Equation 1 is valid only when $\sigma = \sigma_u$. Since the above equations require the introduction of values for D and L, the

Table 3. Tensile data of Grade 2 Ti under different processing conditions.

Sample	σ_y (MPa)	σ_u (MPa)	$\varepsilon_{uniform}$ (%)	ε_{total} (%)	HV 20
0X	337	481	15	30	140
1X	561	589	3.1	11	196
1XH	544	573	3.9	22	205
4XH	651	682	3.7	22	269
1XH+CR(70)	747	769	3.4	19	196
4XH+CR(70)	779	877	3.3	18	205
CR(70)	605	702	2.4	12	229
CR(90)	697	797	3.0	12	271
Ti6-4 (F136)	795*	860*	5**	10*	-

*Minimum value specified by ASTM F136; **estimated value-not specified by the standard.

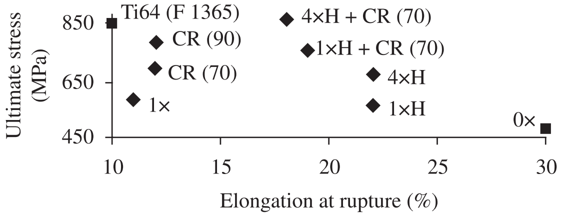

Figure 1. Ultimate stress and elongation at rupture for Grade 2 Ti submitted to different DPS conditions, compared to Grade 2 Ti (annealed) and Ti6Al4V.

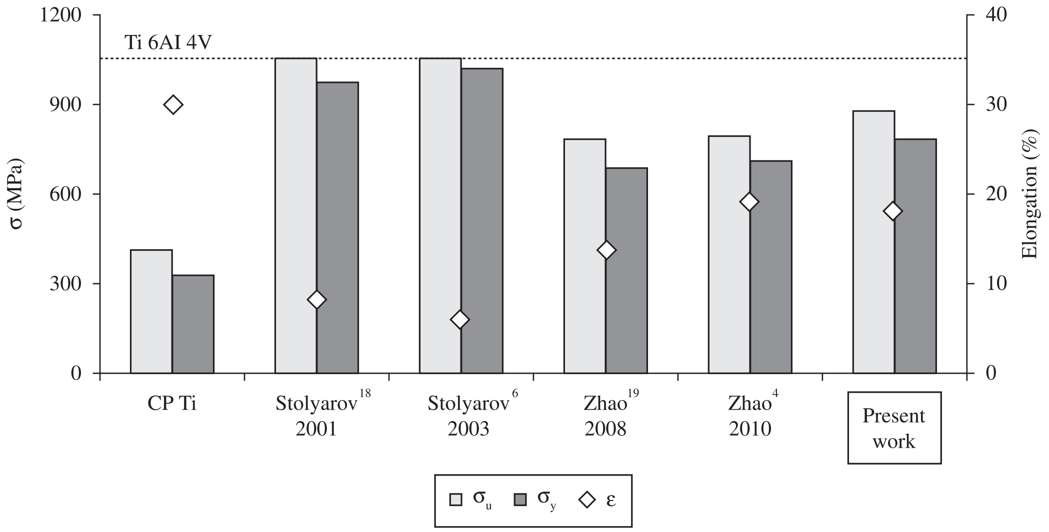

Figure 2. Comparison of the tensile behavior here obtained with those of other investigators. Dashed line indicates the ultimate strength of the Ti6-4 alloy[4,6,18,19].

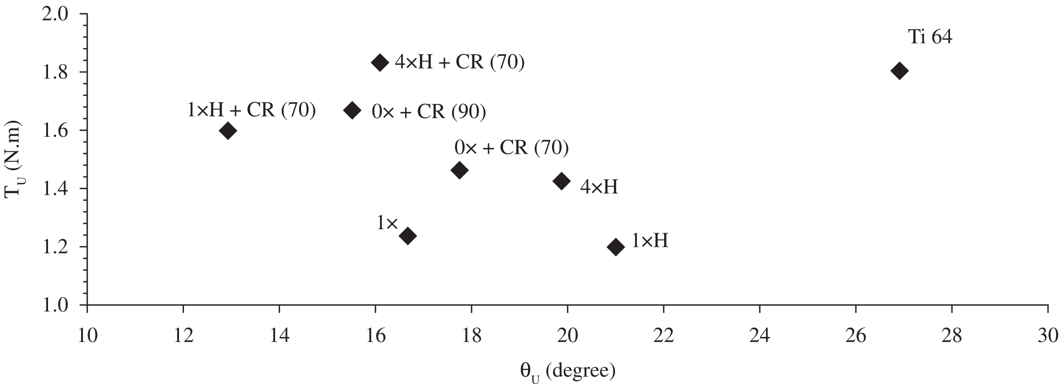

Figure 3. Torque and twist angle corresponding to $\sigma = \sigma_u$ for Grade 2 Ti, under different processing condition and compared with the alloy torsional properties.

present data can be compared with the values of maximum torque and breaking angle prescribed by the ASTM F543 Standard[20] by employing the relevant dimensions of type HA 3.5 cortical screws.

Figure 3 relates data on maximum torque with the correspondent twist angle θ_u, calculated by Equations 1,2, in which the σ_u and $\varepsilon_{uniform}$ values (Table 3) were introduced. D and L were respectively taken as 2.4 mm (the screw core diameter of cortical screws type HA 3.5) and 6.5 mm (about five times the thread pitch). It must be stressed that the resulting T_u and θ_u refer to a smooth torsion specimen made of Grade 2 Ti, having dimensions equal to the cortical screw type HA 3.5 but not to the screw itself.

Figure 3 shows that the 4XH+CR(70) sample has slightly higher T_u than that of Ti6-4 alloy, but it must be taken into account that in the present calculation D was considered equal to the core diameter of the screw. In fact, the screw thread is a helical groove on a cylindrical surface, so that the screw cross section is about 22% higher than the circular core, thus the resulting torque may have been underestimated. On the other hand, when dealing with real cortical screws, stress concentration originated by the threads must be considered; therefore, the actual torque may be lowered, thus counteracting the above mentioned underestimation. However, inspection of the ASTM F543 Standard[21] shows that in most types the screw profiles are relatively smooth, a feature devised to reduce stress concentration as much as possible. At any rate, precise data on maximum torque and breaking angle of real screws can only be obtaining by following the test method provided by the Standard, and this is outside the scope of the present paper.

Furthermore, although a requirement regarding the twist angle calculated by Equation 2 (valid within the uniform deformation range), is absent in the ASTM Standard[20] the present value was compared to an estimated θ_u for Ti6-4 (using Equation 2). Figure 3 shows that values of all twist angles for Ti are lower than that estimated for the alloy, but Table 3 shows that the non-uniform elongation at rupture in tension for Ti, that is, ε_{total} - $\varepsilon_{uniform}$, is much higher than that estimated for the alloy, namely 14 against 5%, an indication of lower strain rate sensitivity of the former material. That this behavior is advantageous for the SPD Ti in terms of the breaking angle θ_f specified by the Standard was confirmed by some recent experimental data obtained on smooth torsion samples[9], in which the breaking angle for Ti4X and the Ti6-4 alloy were equal to 253° and 230°, respectively.

4. Conclusions

1. The tensile strength difference between Grade 2 Ti and the alloy Ti6-4, was reduced from 379 to –17 MPa by four ECAP passes performed at 300 °C and a subsequent 70% thickness reduction by cold rolling;
2. In terms of strength enhancement, a thickness reduction of 70% by cold rolling is similar to four ECAP passes at 300 °C. Although all modes of deformation show ductility losses with respect to the non-deformed sample, losses are more severe when only cold rolling was employed;
3. Sample 4XH + CR(70) has the same maximum torque than the 6-4 alloy, but its twist angle is ≈40% lower. Suggestions can be made that the low strain rate sensitivity of Grade 2 Ti may benefit the breaking angle; and
4. Results show that on a material-to-material comparison, ECAP processed Grade 2 Ti can replace the Ti6-4 alloy as a construction material for medical bone screws.

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