Severe Plastic Deformation and Its Application on Processing Titanium: A Review

Zahid Husaain, Awais Ahmed, Osama M. Irfan, and Fahad Al-Mufadi

Abstract—**Severe Plastic Deformation (SPD) is a widely technique used to produce Ultrafine Grained (UFG) structure that has superior mechanical properties. In this review a large family of different SPD processes like Equal Channel Angular Pressing (ECAP), High-Pressure Torsion (HPT), Multi-Axial Forging (MAF) and others has been presented. Also application of SPD techniques on some metals and alloys have been discussed. Titanium with different grades is one of the most important materials that is subjected to SPD due to its distinguished mechanical properties like low density, high strength, high corrosion resistance etc. This makes Titanium useful in many industrial and biomedical applications. Ultrafine grained (UFG) titanium has even more applications rather than coarse grained (CGS) material. In this review, the recent development in mechanical characterization and applications of nanostructure Titanium has been dictated. Moreover, the potential future work has also been discussed.**

Index Terms—**Nano-structured, SPD, Titanium, Ultrafine Grained.**

Abbreviations

ARB	Accumulative roll bonding	MAF	Multi axial forging
CGS	Coarse grained structure	NS	Nano-structured
ECAE	Equal channel angular extrusion	SMAT	Surface mechanical attrition treatment
ECAP	Equal channel angular pressing	SPD	Sever plastic deformation
HAGB	High angle grain boundary	SRC	Straightening & repetitive corrugation
HE	Hydrostatic extrusion	TE	Twist extrusion

I. Introduction

Grain size is considered as a central microstructural parameter to control the physical, chemical, mechanical and biochemical properties of polycrystalline materials. At grain size of the level of microns to nanometer scale, the material exhibits extraordinary characteristics. Hence to get desired functional properties, grain size control is one of the most important factors to be considered. As the need for better material performance ever persists, viable techniques for manufacturing of ultrafine grained structure have been introduced. These techniques are broadly classified into consolidation processes and severe plastic deformation (SPD). The SPD can be defined as a metal forming process applied on bulk solid material under sever hydrostatic pressure to attain very high strain. This occurs without significant change in the overall dimensions of the sample [1].

Sub-micrometers sized grains are formed in initially coarse-grained (CG) material by application of SPD. Consequently, an enhanced mechanical performance (strength, tribological etc.) was observed. It is, however, thought that SPD results in formation of shear bands which causes subdivision of grains due to deformation process [2 and 3]. It is well established that enormous deformation of metals results in a distinctive structure of dislocations and extremely fine grains. The important parameters in defining a submicron grain structure are average spacing of high angle grain boundaries (HAGB) and proportion of HAGB area [4], [5]. SPD causes major changes of the materials structure which are reflected in enhanced mechanical and physical properties of metals such as hardness and yield stress. However, the disadvantage of the material deformed by SPD is limited ductility [6]. On the other hand, many researches showed increase in ductility and toughness as well as enhancement of the physical properties of other materials. The fine grained structure of materials attained by SPD results in superplastic behavior at lower temperatures [7].

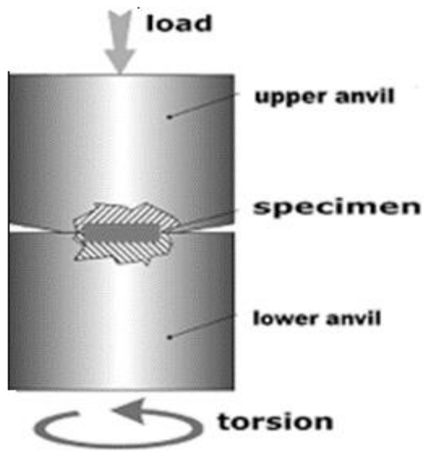

Fig. 1. Torsion at high pressure [13].

Titanium is considered as very suitable candidate for applications in areas of implants, marine environment, leisure and sports, transportation, power generation and aerospace disciplines [8], [9]. Commercial pure titanium and its alloys possess low modulus of elasticity, high biocompatibility, relatively high strength, high erosion and

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corrosion resistance, formability and good machinability. However, titanium has certain limitations as well [10]. Poor tribological properties, toxicity of some of its alloys susceptibility to galling and seizure have limited its use in certain technological devices [11]. The purpose of this article review is to have a bird's eye view on different SPD techniques available and to study their effect on the properties of commercially pure titanium. It is hoped that this would prove a mild stone in this field to understand what has been done and what is expected in future.

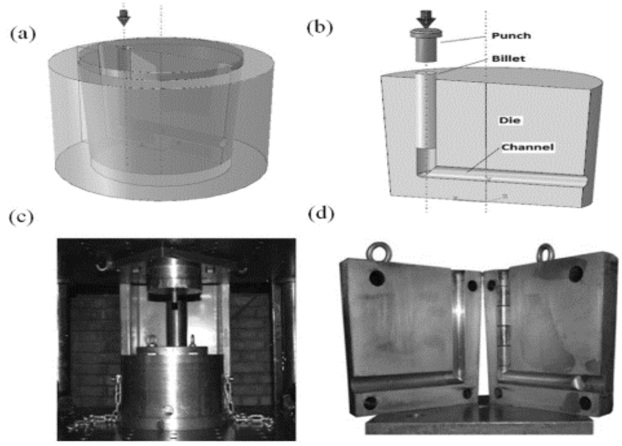

Fig. 2. ECAP Process (a) Dies in assembled form (b) Cross-section of die with specimen and plunger (c) Pressing (d) Specimen in the channel [72].

II. Severe Plastic Deformation (SPD)

Severe plastic deformation technique can serve the purpose only when certain requirements have been fulfilled. Firstly, severe deformation of bulk samples and billets should result into predominantly high angle grain boundaries to ensure qualitative changes. Second condition is concerned with homogeneity of processed materials to have uniform and stable properties through the material.

A. High-Pressure Torsion (HPT)

Grain size of 100 nm or less can be obtained by application of HPT [12]. This technique has been successfully applied to a variety of materials such as metals, alloys, composites and more recently on semiconductors [13]. Fig.1 shows the schematic of HPT technique. A circular specimen of suitable dimensions is placed between two anvils; one fixed and other rotatable. High pressure is applied on fixed anvil and rotatable anvil is rotated with high speed. The friction between sample and fixed anvil results into large strains. The pressing force (high pressure) prevents the specimen from breaking despite application of severe deformation [14]. The importance of high pressure has been investigated in case of nickel [15]. A significant grain refinement may be achieved by half or one rotation. However, homogeneous structure essentially requires deformation by several complete turns. On the advantage side, a clearer understanding can be developed about the influence of different parameters like applied pressure, strain rate, accumulative stain on structure of the sample because of the fact that these parameters are independently control the process. However, this technique is limited to small scale (laboratory) and cannot be applied for mass production which is a disadvantage.

B. Equal-Channel Angular Pressing (ECAP)

The idea that ECAP can be used as a SPD technique has been provided in literatures [17]-[18]. Later on, applying ECAP to produce UFG structure material was advised by R. Z. Valiev and his colleagues [19]-[20].

A variety of metals and alloys have been successfully processed by this method, schematic of which is shown in Fig.2. Here a specimen of suitable diameter and length is pressed into the channels of die. High pressure causes the material to flow within the channel. Die channel angle may vary from 75 degrees to 120 degrees. A substantial grain refinement can be obtained by single pass, however, it four to six passes are required to make homogeneous structure. To date, many materials and alloys such as copper, Al-Mg alloys, pure titanium has been processed [21]-[23].

Soft materials are easily processed but hard to deform materials put some challenge to die design because mechanics of this method has revealed that contact stresses in die are of paramount importance. Experimental and numerical computation are required iteratively for design optimization of die to get uniform ultrafine grains. In spite of recent progress, many problems remain unresolved to obtain homogenous ultrafine grain size especially in the range below nano size for very hard materials of large size.

ECAP is considered as an effective method to control both microstructure and texture of materials. Through ECAP many researchers studied the texture and microstructure of metals and alloys. For example, ECAP process through a die angle of 90° was applied on Al-7075 alloy. X-ray diffractometer and imaging microscopy were implemented. The results revealed that different routes of ECAP leads to dissimilar textures, enhanced significantly after first pass and weaken after subsequent passes [72].

C. Straightening and Repetitive Corrugation (SRC)

Schematic of straightening and repetitive corrugation method has been shown in Fig.3 where a specimen is alternatively subjected to shear and bending stresses when it is passed between two gears like rollers under constraining pressure [24]-[25]. By using straightening and repetitive corrugation method many of the limitations of SPD were overcome. The drawbacks of this process is non-uniformity in structure along the length of the sample while by increasing the number of passes a homogenous structure can be obtained. This can be done at the price of reduction in thickness because of frequent passes between the rollers.

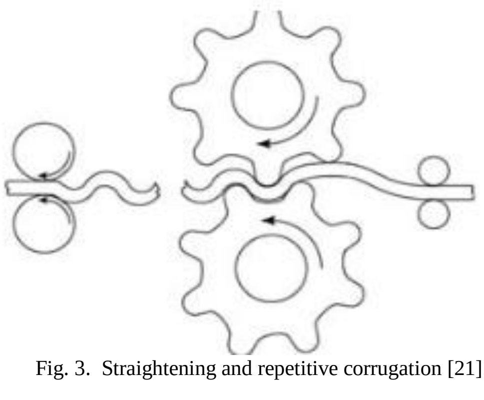

Fig. 3. Straightening and repetitive corrugation [21].

D. Multi-axial Forging (MAF)

Multi Axial Forging (MAF) is one of the most effective and easiest ways to have large strain on bulk materials [26-28]. No specific devices are needed to apply MAF as shown in Fig. 4. The appropriate strain rate and temperature are the key parameters that control this process. It should be kept in mind these parameters are specific to the material and need to be determined experimentally or computationally. Besides the other SPD methods, MAF can be used in mass production as it has great capability for producing large work pieces [27]-[32]. So far, MAF technology has been limited to produce some UFG materials such as copper, titanium and its alloys, steel, aluminum and magnesium [33]-[37]. These studies mainly focus on the function of MAF process on grain refinement and the subsequent mechanical properties of the materials [38], [39]. However, this kind of processing and the microstructure development is not clear yet and much research work needed to be done to clarify the process.

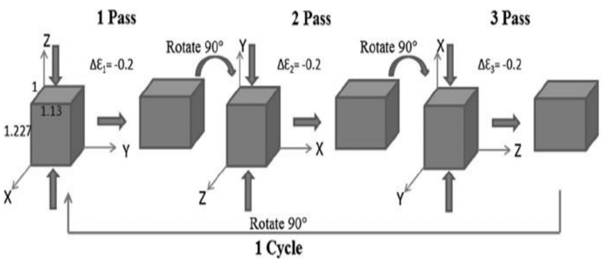

Fig. 4. Schematic representation of MAF for 1 cycle [35].

E. Twist Extrusion (TE)

The principle of twist extrusion (TE) is shown in Fig. 5. A billet of suitable dimension is twisted in die and thus intense severe deformation is obtained [40], [41]. The form and the cross-sectional area remains the same along the extrusion axis.

Twist extrusion method can be used to process metallic materials and alloys [42], [43]. One of the attractiveness of TE is that it can be performed in any extrusion facility by replacing existing die with a twist die. Bhandari *et al.* [44] reported that selection of loading path is an important factor that controls not only mechanical properties but also heterogeneity. Very hard materials require high temperature to undergo plastic deformation. For instance, Ti 6Al 4V is very hard to deform and hence requires to be done at high temperature [46]. It has been reported that application of ECAP and TE followed by one another is an effective method to control and get uniform properties in the material [45], [46].

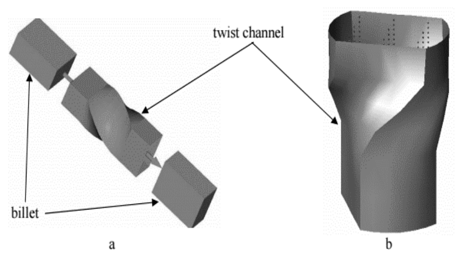

Fig. 5. Twist extrusion process and (b) twist channel inside a die [43]

F. Accumulative Roll Bonding (ARB)

Accumulative roll bonding (ARB) is a deformation method in which two metal sheets of the same thickness are simultaneously passed between two rollers. In ARB the two sheets join together to form one solid body and can be halved once again. The rolling process can be repeated many times. In most cases, the process is repeated up to 10 times [47]-[48]. The bonding of sheets is accompanied by microstructural refinement. The strength of the processed material is comparable with other methods. This process is, however, experimental only and still not have been employed on mass production of UFG materials. Much of the research work has been done for other SPD methods such as cyclic extrusion compression and combined ECAP-HPT methods [49]-[50]. A large work remains regarding ARB to investigate its effectiveness and potential to be used as SPD method.

III. TITANIUM PROCESSED BY SPD

High yield strength, low density and high wear and corrosion resistances are the properties which make titanium a distinguished material in its family. Due to low elastic modulus, titanium is increasingly being used in medical implants. It also has applications in aerospace industries, chemical process equipment and others. It has been established that application of SPD technique results in superior mechanical properties such as strength and hardness. The enhancement of the properties is due to refinement of the grain size of the material [51]. The mechanical properties of different grades commercial pure titanium processed by equal channel angular extrusion (ECAE) have been examined [52]. The findings of the study regarding tensile properties are summarized in Table I. The minimum processing temperature without shear-localization to withstand 8 ECEAE passes was 300 ℃ for grade 2 Ti and 450 ℃ for grades 4. Coarse-grained microstructures with average grain size 110 μm to 70 μm was refined down to 300 nm. Fig.6 shows true stress-strain plot which clearly indicates the improvement in strength with UFG materials. Grade 2 Ti is commonly processed by SPD to refine grain size but method and specific temperatures was proposed for grade 4 Ti and was recommended to use it as dental implants instead of Ti6Al4V alloy. Antonialli et al. [53] examined the machinability of ECAP processed Ti and found a significant decrease in machinability as compared with coarse grained (CG) titanium.

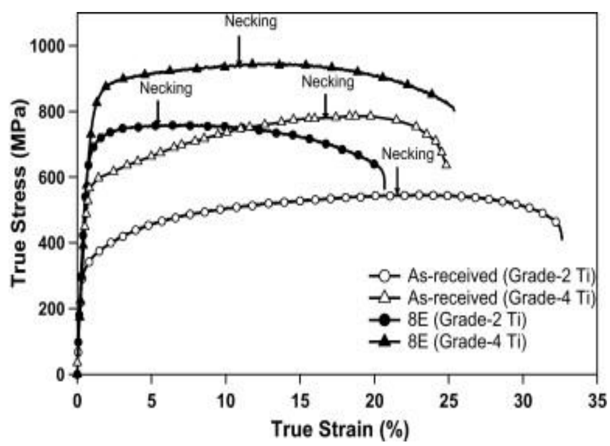

Fig. 6. True stress–true strain response of as-received, grade-2 and grade-4 CP-Ti. Subjected to ECAE process (8E) [52].

TABLE I: TENSILE PROPERTIES OF AS-RECEIVED AND ECAE-PROCESSED GRADE2 AND GRADE4 CP-TI. [53]

Materials	Condition	Grain size (μm)	σ_y (MPa)	σ_{UTS} (MPa)	ε_f (%)	ε_u (%)
Grade-2 CP-Ti	As-received	110	307	532	33	22
	8 ECAE	0.30	620	760	21	6
Grade-4 CP-Ti	As-received	70	531	792	25	16
	8 ECAE	0.30	758	947	25	11
Ti–6Al–4V (ASTM Grade-5)	Annealed	–	880	950	14.0	–

Titanium and its alloys are also being used in biomedical implants rather than dental applications. For instance, Ti-6Al-4V alloy has enough strength to be employed for tooth implant. But this alloy is toxic and has potential harmful effect on human health. It may result in severe pain in human body due to presence of aluminum and vanadium. On the other hand, commercially pure titanium is not strong enough to withstand real environment despite of its non-toxic nature and high corrosion resistance properties. It has been advised that UFG titanium should be used for orthodontic mini implants instead of commercially pure Ti and Ti6AL4V alloy. Nano-Ti was obtained by Equal Channel Angular pressing (ECAP) from commercially pure Ti. Mechanical properties were compared by Torque Test and surface properties by scanning electron microscope SEM. It was observed that UFG Ti exhibited superior mechanical properties (advantage over commercial pure Ti) and good corrosion resistance and biomedical compatibility (advantage over Ti alloy) [54].

Moreover, biomedical applications require high fatigue strength because of repetitive loading on the implants. Fatigue strength of ultrafine grained titanium has been observed to increase and make it valid candidate for biomedical applications. Kim *et al.* [55] found that ultrafine grain titanium produced by ECAP showed increasing fatigue strength of up to the factor of 1.67.

Another factor to be considered is the compatibility of an implant material with bones and tissues. In this regard, production of ultrafine grained materials possesses additional advantages for their beneficial effects. They show improved ability of implant surface to integrate with bones and tissues and also on fatigue strength [56], [57].

Excessive wear of prosthetic components has adverse effects on performance. It generates debris and fragments, which cause severe cellular response and inflammation thereby leading to implant loosening and pain in the body. Eventually, this may lead to bone loss [57]. Molinari et al. [58] stated two reasons of poor wear properties (1) low strength (2) low protection due to surface oxide resulting in high temperature between dry surfaces. UFG show excellent tribological properties because of high strength and minimized friction between the surfaces. Pei Qing La et al. [59] examined the dry-sliding tribological properties of UFG Ti (produced by ECAP route B_c) against AISI52100 steel. The study was carried out under varying loads (5-35N) and sliding speed (0.02 to 0.08 ms^{-1}) under ambient conditions. Scanning Electron Microscope (SEM) and X-ray photoelectron microscope were used to examine worn surfaces. Fig. 7 depicts that wear rate is smaller for UFG Ti as compared to Ti at a given load and increases with increasing load. It has been also found that the UFG structures enhanced the wear properties and the behavior of material was significantly affected by micro ploughing and delamination [60], [61]. The higher mechanical resistance of UFG Ti is considered to be the most important property that enable it to be used in biomedical applications [62]-[65].

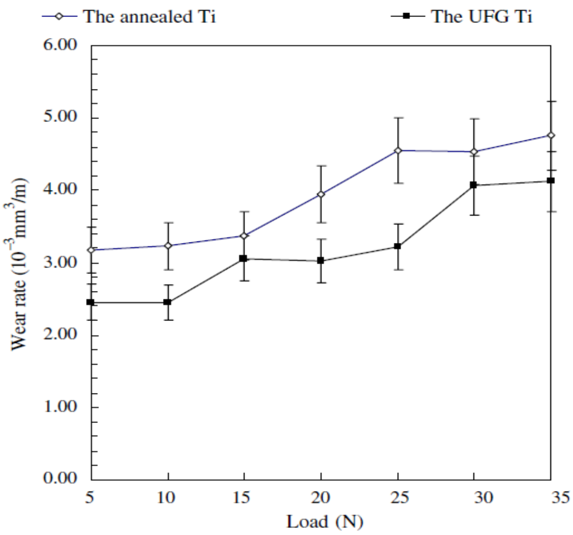

Fig. 7. Wear rate at different loads [60].

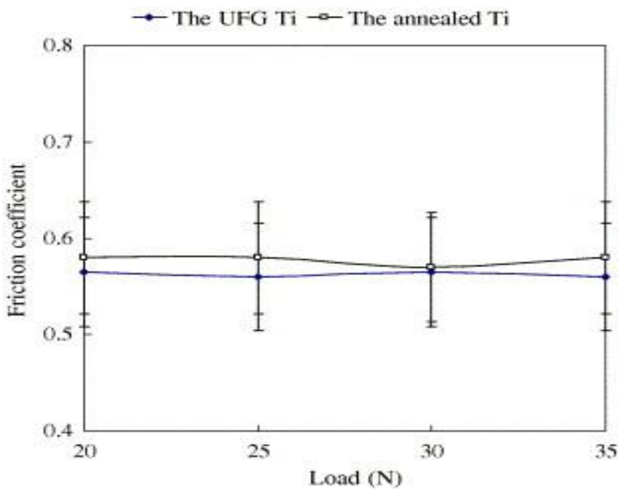

Fig. 8. Friction coefficients at different loads [60].

The coefficients of friction of UFG and CGS titanium are comparable as shown in Fig.8. However, Fig. 9 indicates that wear rate of UFG titanium is inversely related with speed.

Garbacz *et al.* [66] compared the wear properties of NS titanium obtained from hydrostatic extrusion (HE) method and CGS Ti-grade2. The experiments were carried out under various lubricant conditions included dry sliding contact, normal saline water, and paraffin oil. Tribological examination was made with pin-on-disk tribometer. Table II & Table III give the changes in coefficient of friction and linear wear investigated during the test. Poor wear resistance was observed in both cases. Tribological reactions occurring at interface are mainly responsible for variation in tribological characteristics. Additional experiments conducted under dry sliding revealed the favorable effect of HE process on titanium and proved excellent tribological properties of this frictional pair.

TABLE II: RESULTS OF WEAR TEST OF TITANIUM UNDER SLIDING CONDITIONS AGAINST STEEL DISC [61]

Lubricant condition	specimen material	$\Delta/(\mathbf{mm})$		$\Delta/(\mathbf{mg})$		μ	
		Ti- Grade		Ti Grade		Ti-Grade	
		2	HE	2	HE	2	HE
NaCl	Steel	752	850	21.53	23.86	0.29	0.32
Paraffin	Steel	1600	2010	39.5	58.3	0.38	0.28

TABLE III: RESULTS OF WEAR TESTS UNDER SLIDING AGAINST STEEL DISC AT DIFFERENT PRESSURES [61]

Normal Pressure, (MPa)	$\Delta/(\mathbf{mm})$		$\Delta/(\mathbf{mg})$		μ	
0.28	185	195	4.76	5.45	0.71	0.71
0.7	250	310	8.85	10.21	0.33	0.45
2.0	750	935	20.94	26.05	0.29	0.27

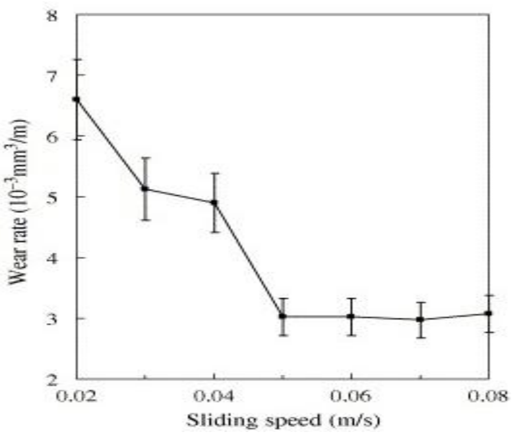

Fig. 9. Wear rate of UFG Ti.

Recently, a relatively new surface severe plastic deformation method called surface mechanical attrition treatment (SMAT) was developed. Wen M. et al. [67] carried out SMAT for Ti and investigated the tribological properties of titanium. Ball-on-disc tribometer was used to investigate the tribological behavior. SMAT exhibited better wear resistance as compared to CG titanium. High wear has been observed in case of CG titanium as shown in Fig.10.

Metals and alloys with an UFGS are known to have lower corrosion resistance compared to those with a coarse-grained structure (CGS) [68], [69]. Therefore, it is necessary to investigate the corrosive behavior of plastically deformed materials. Mirkhanova *et al.* [70] investigated the corrosion behavior of UFGS titanium. It showed relatively larger corrosion potential as compared to CGS but interesting results were found with chemical or electrochemical polishing.

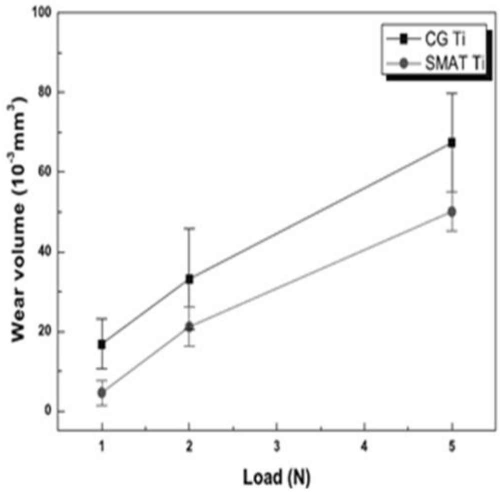

Fig. 10. Wear volume of SMAT and CG Ti versus different loads [67].

The corrosion resistance of UFGS Ti increased to greater degree as compared to CGS. Table shows the effect of chemical polishing and a substantial increase in corrosion resistance where ε is the corrosion potential, and j is the corrosion current density. Also no significant effect of ECAP was observed on corrosion properties of titanium [70].

TABLE IV: EFFECT OF ECAP ON ELECTROCHEMICAL CHARACTERISTICS OF TI (GRADE 2) IN VARIOUS STRUCTURAL STATES [70]

State of Titanium	$\varepsilon, \mathbf{V}$		$\mathbf{j}.\,\mu\mathbf{A/cm^2}$	
	Before ECAP	After ECAP	Before ECAP	After ECAP
CGS	-0.280	-0.393	0.0015	0.12
UFGS	-0.373	-0.146	0.0032	0.16

IV. CONCLUSION

Nano structured titanium has many applications in industry. One important recent application is in the biomedical discipline. Fatigue strength, tensile strength, corrosion resistance and nontoxic nature make nano structure titanium a sole option in biomedical implants. However, implants face impingement of blood with certain ingredients which may cause erosion and perhaps erosion-corrosion. To our knowledge very limited work has been done to study the erosion of UFG titanium. Moreover, tribological properties of nano Ti are not fully investigated. We did not find any published data to ascertain thermal properties of UFG Ti in spite of applications of Ti at high temperature environment.

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