Biomaterials and Biomechanics in Bioengineering, *Vol. 2, No. 3 (2015) 185-196*
(Formerly, Biomaterials and Biomedical Engineering)
DOI: http://dx.doi.org/10.12989/bme.2015.2.3.185

Mechanical and wear properties of HPT-biomedical titanium: A review

Mohsin Talib Mohammed*

Mechanical Engineering Department, Faculty of Engineering, Kufa University, Najaf, Iraq

(Received June 25, 2015, Revised November 3, 2015, Accepted November 23, 2015)

Abstract. Titanium (Ti) based alloys are widely used in biomedical implants due to their low density, excellent corrosion resistance and good biocompatibilities. In recent years, growing interest in sever plastic deformation (SPD) has stimulated research and development on the techniques to attain refining of the grain size to the submicrometer or even nanometer level. The mechanical and wear properties determining the application of Ti in medicine may be improved via SPD. High pressure torsion (HPT) technique is one of the approaches available for improving the mechanical and wear properties of biomedical Ti materials. Accordingly, this article is designed to examine most recent state of the art scientific works related to the developments in mechanical properties and wear resistance of biomedical Ti materials processed by HPT. A comprehensive review in this area is systematically presented.

Keywords: titanium; biomedical applications; mechanical properties; wear; HPT

1. Introduction

The good mechanical properties, outstanding corrosion resistance, and excellent biocompatibility of Ti materials have led to increase their use in various biomedical applications especially in orthopedic and dental implants. The mechanical properties of Ti alloys are strongly affected by micro-structural features such as grain size, constituent phases, lattice defects and so on. SPD is one of the effective processing routes to enhance the mechanical strength and wear resistance of Ti alloys by ultrafine-grained (UFG) structures (Figueiredo and Langdom 2012) or specific nanostructural features (Valiev *et al*. 2014). To obtain the UFG structure and bulk nanostructured materials (BNMs), several SPD techniques have been suggested as follows; high pressure torsion (HPT) (Zhilyaev and Langdon 2008, Wang *et al*. 2012, Wang *et al*. 2013a, Wang *et al*. 2013b, Wang and Langdon 2013), equal channel angular pressing (ECAP) (Meredith and Khan 2012, Lin *et al*. 2013), accumulative roll-bonding (ARB) (Raducanu *et al*. 2011, Kent *et al*. 2011, Cojocaru *et al*. 2013), hydrostatic extrusion (HE) (Topolski *et al*. 2012, Ozaltin *et al*. 2014), multi-directional forging (Yang *et al*. 2012), friction-stir processing (FSP) (Bo *et al*. 2014) and others. The properties of BNMs produced by SPD techniques essentially depend upon processing method applied and the technical parameters. These techniques have a huge potential for use in

*Corresponding author, Ph.D., E-mail: mohsent123@yahoo.com

http://www.techno-press.org/journals/bme&subpage=7 ISSN: 2288-3738 (Print), 2288-3746 (Online)

highly advanced applications particularly in the field of medicine (Valiev *et al*. 2007). Among SPD processes, HPT merits particular attention due to the possibility of obtaining extremely small grains and high strength (Zhilyaev and Langdon 2008). Currently, HPT is one of the most popular SPD techniques for producing grain refining with superior properties and high performance. There exists ever increasing importance of the subject, growing research interest and a vast unexplored potential which is the main driving force behind this review. There is a limited number of reviews presently available on the characteristics of HPT-processed Ti materials for medical applications. Therefore, the aim of this paper reviews the research status of HPT-processed Ti for biomedical applications.

2. Background of high pressure torsion technique

HPT is a valuable technique for inducing an extremely intense plastic strain to produce a microstructure composed of UFG (100-500 nm) or nanostructured (<100 nm) grain in metallic materials (Zhilyaev and Langdon 2008). In HPT processing (Fig. 1) (Xu *et al*. 2008), a small disk is placed between two massive anvils and one of them is able to rotate under sever compressive pressure of several GPa. The HPT processing is performed by imposing torsional straining through rotation of the lower anvil. Subsequently, the disk is plastically deformed by pure shear due to surface frictional forces (Zhilyaev and Langdon 2008). Therefore, the bulk material is deformed by a very intense torsional strain without introducing any significant changes in the overall dimensions of the work-piece. Such metallic materials show great mechanical strength as compared to that of the coarse grained (CG) materials (Valiev *et al*. 2000, Kim *et al*. 2006).

At different position of the disk, the equivalent shear strain (γ) in HPT can be estimated via the following relationship (Valiev *et al*. 1996)

$$\gamma = 2\pi NR/h \tag{1}$$

where R and h are the radius and height (or thickness) of the processed sample, respectively, and N is the number of HPT turns. In practice, Eq. (1) includes only the effect of the torsional straining regardless of the presence of an additional strain imposed by the compressive pressure (P). It is readily apparent from Eq. (1) that the imposed shear strain is a maximum around the edge of the

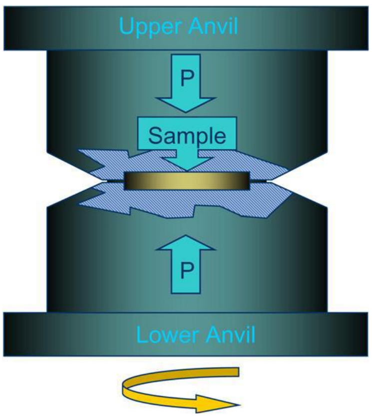

Fig. 1 The Schematic illustration of HPT processing

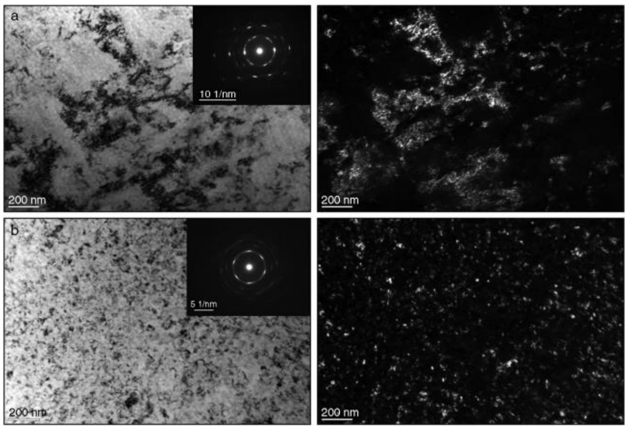

Fig. 2 The microstructure of Ti-24Nb-4Zr-8Sn alloy after HPT with 1 turn (a) and 50 turns (b)

sample but it becomes equal to zero in the center of the sample where R=0. Thus, it is predicted that the microstructure and the micro-hardness will be inhomogeneous and will depend on the precise position within the sample. However, some recent literature showed that the microstructure and the hardness become reasonably homogeneous at high pressures after sufficiently increasing the applied pressure and/or the total number of turns (Cho *et al*. 2014, Shahmir *et al*. 2014). Generally, in HPT processing, the factors influencing the grain size as well as the homogeneity of the microstructure are the numbers of turns in torsion, the applied pressure, the frictional effects and temperature (Fu *et al*. 2015). For example, Fig. 2 (Sharman 2015) shows the effect of the numbers of turns on the microstructure of Ti-24Nb-4Zr-8Sn alloy after 1 turns and 50 turns respectively. A non-homogenous microstructure with relatively large subgrains and a small fraction of fine grains can be observed after 1 turn (Fig. 2a), while a highly refined and homogenous microstructure can be achieved after 50 turns (Fig. 2b).

It is found that the HPT can generate strains as high as 15 in one single twisting operation. However, the sizes and geometries of samples prepared by this technique are limited (Eliasa *et al*. 2013).

3. Effect of HPT on the mechanical properties

Investigations on the behavior of SPD-Ti materials have been greatly motivated by the expectations that the processed parts possess unique combination of properties. Moreover, there is an imperative necessity to understand the fundamental mechanisms underlying the specific properties associated with extreme grain refinement. Hardness, strength, ductility (e%), Young's modulus (E) and fatigue are primary grain-size-dependent characteristics of implantable Ti materials, which determine virtually all facets of material's in service response. A suitable Ti biomaterial should ideally combine high ultimate strength (UTS) and yield strength (YS), with sufficient e%, along with low E. However, high strength and good ductility are often mutually contradicting to have them simultaneously. The grain size (d) dependence of mechanical strength (yield stress σ_y) is directly related by the Hall-Petch equation as

$$\boldsymbol{\sigma_y=\sigma_0+Kd^{-1/2}} \qquad (2)$$

In Eq. (2), σ_0 is the known as friction stress and K is a material constant. It is accounted that **K**=6 MPa mm$^{1/2}$ for Ti (Elias *et al*. 2013). The Hall-Petch relationship establishes that the mechanical properties directly relate to the grain size. The UFG and BNMs are found to exhibit exceptionally good mechanical properties, such as strength, toughness, and superelasticity at ambient temperatures compared with their CG counterparts (Horita *et al*. 2000, Tsuji *et al*. 2002). The Hall-Petch strengthening depends mainly on the grain boundaries which act as pinning points to impede further dislocation propagation. In addition, the nature of grain boundaries is much more disordered than inside the grain which leads to prevent the dislocations from moving and consequently enhance the mechanical properties of the material (Wadood *et al*. 2013). Extensive studies and serious attempts were made in recent past to achieve better mechanical performance in terms of biomechanics of Ti materials via changing grain size using HPT as given in Table 1.

It is well known that commercially pure Ti (CP-Ti) is regarded as a desirable material for biomedical applications due to its low weight, excellent corrosion behavior and high biocompatibility. However, the mechanical strength of CP-Ti is relatively low compared to other biomedical metals due to which its application to heavy load conditions is limited (Niinomi 1998). It is reported that nanostructured CP-Ti deformed by SPD improves the mechanical performance of CP-Ti compared to conventionally processed pure Ti (Valiev *et al*. 2008). According to Sergueeva *et al*. (2001), the refinement of microstructure of CP-Ti leads to significant increase in hardness and/or strength. The authors performed a combination of SPD such as HPT followed by short annealing at low temperatures which enhanced the strength to more than 1200 MPa with sufficient ductility (more than 20%). Valiev and Alexandrov (2002) pointed out that the microstructure of pure Ti can be tailored using HPT technique to produce UFG structure that has a combination of high strength and high ductility. The authors suggested the presence of the combination of UFG size and high-density dislocations to enable the plastic deformation as new mechanisms. It has been reported that UFG structure with mean grain size of about 120 nm forms in CP-Ti after processing it using HPT at room temperature. In addition, a rearrangement of

Table 1 Different mechanical properties of medical Ti materials subjected to HPT process

Ti material	Processing	Grain size nm	UTS MPa	e %	E GPa	Ref.
Grade 2	ECAP	-	750	7	-	Zhang *et al*. (2011)
Grade 4	ECAP	300	947	25	-	Purcek *et al*. (2011)
Grade 3	ECAP+HPT (1.5 GPa)	200	730	25	-	Stolyarov *et al*. (1999)
Grade 3	HPT (5 GPa) HPT+Annealing	~120 ~120	950 >1200	14 >20	- -	Sergueeva *et al*. (2001)
Grade 4	HPT (6 GPa)	-	1600	-	-	Islamgaliev *et al*. (2008)
Grade 2	HPT (3 GPa)	130	940	23	-	Wang *et al*. (2013a)
Ti-6Al-4V	HPT	80	1750	-	-	Stolyarov *et al*. (2011)
Ti–29Nb–13Ta–4.6Zr	HPT (1.25 GPa, N=60)	-	1100	6.7	~60	Yilmazer *et al*. (2013)
Ti–24Nb–4Zr–8Sn	HPT (3 GPa, N=50)	20-50	1050	0.025	<60	Sharman *et al*. (2015)

defects promotes high angle grain boundaries with very high dislocation densities after annealing above 200°C, which indicates the non-equilibrium character of such grain boundaries after annealing (Valiev *et al*. 2003). It is important to mention here that the results available to date (Table 1) show that processing by HPT generally produces greater grain refinement and improved strengthening by comparison with ECAP (Zhao *et al*. 2008, Edalati *et al*. 2009, Sabirov *et al*. 2010, Sabirov *et al*. 2011, Zhang *et al*. 2011). For example, a high UTS of 1600 MPa was achieved in pure Ti (Grade 4) using HPT but with a corresponding significant loss in ductility (Islamgaliev *et al*. 2008). Moreover, a group from Japanese researchers (Wang *et al*. 2013a) reported that HPT at room temperature can reduce the grain size of CP-Ti from ~8.6 μm in the as-received state to UFG of ~130 nm. The results showed a good combination of high UTS (940 MPa) and a reasonable elongation to failure (23%) as a result of reducing grain size after performing HPT. Fig. 3 shows the microstructures and tensile curves of the CP Ti before and after processing by HPT.

β-type and metastable β-type Ti alloys containing β-stabilizers such as niobium (Nb), zirconium (Zr), tantalum (Ta) and others have attracted considerable attention for orthopedic implants applications owing to their unique combination of high mechanical properties, low Young's modulus (E), superior corrosion behavior, and excellent biocompatibility (Mohsin *et al*. 2014). Yilmazer *et al*. (2012) investigated the micro-structural refinement of Ti–29Nb–13Ta–4.6Zr (TNTZ) alloy using HPT processing in order to get the preferred mechanical properties. They found that HPT is an effective process for producing UFG in a single β structure of TNTZ. Further, Yilmazer *et al*. (2013) investigated the effect of HPT, under different rotation numbers (N=1-6), on the mechanical biocompatibility of TNTZ alloy. The authors found that the alloy which subjected to HPT processing at low N exhibits a heterogeneous microstructure in micro-scale and nano-scale. The grains show high dislocation densities with nonequilibrium grain boundaries and non-uniform subgrains. However, at high N (N>20), the microstructure and hardness distribution became more homogeneous with increasing equivalent strain. The results proved the outstanding mechanical biocompatibility of TNTZ subjected to HPT with high UTS (around 1100 MPa) and low E (around 60 GPa). On the other hand, the ductility of the alloy showed a reverse trend and a low-level elongation, at around 7%. The authors concluded that the mechanical strength of TNTZ can be improved while maintaining a low E in single β grain structures through HPT. However, the mechanisms of dislocation generation and micro-structural refinement are unclear for β-type Ti

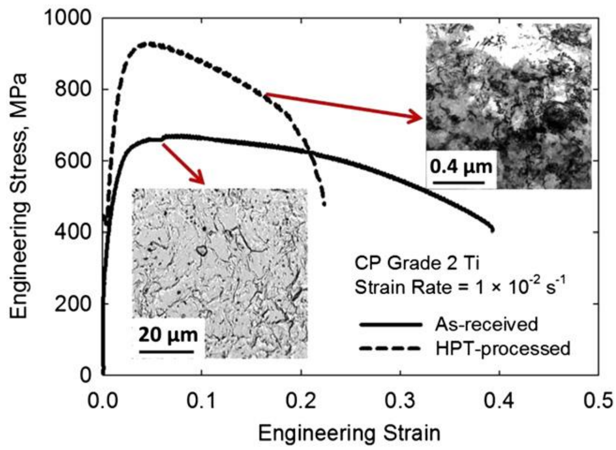

Fig. 3 Microstructure and tensile curves of Ti substrates

alloys having body centre cubic (BCC) structure during HPT processing. Later, Yilmazer *et al*. (2014) studied the micro-structural changes of TNTZ through HPT processing using X-ray diffraction analysis and transmission electron microscopy. The most important findings were that the intense *β* {110} peak revealed that the preferred orientation is *β* <110> for TNTZ alloy due to HPT. The HPT processing led to a drastic increase in dislocation density (5.3×10^{16} m^{-2}) due to a very large accumulation of dislocations.

The formation of metastable phases due to the deformation through HPT process is an important aspect which can have an effect on the tenacity of Ti alloys and in turn on their mechanical properties. It is developed an attracted UFG microstructure for Ti–20Mo alloy by significantly changing the aging response of the alloy through HPT. The process led to entire ultrafine-duplex (*α*+*β*) structure in which equiaxed *α* phase precipitated in equiaxed *β* grains. This exceptional microstructure may outcome from three key sources: nanoscale *β* grains, abundant grain boundaries and enhanced atomic transport (Xu *et al*. 2013). Svetlana *et al*. (2014) studied the effect of HPT on the aging kinetics of metastable *β* Ti-15Mo alloy and its mechanical properties. The alloy was solution treated at 810°C for 20 minutes, water quenched and then subjected to 5 HPT rotations at 200°C with an applied pressure of 6 GPa. The solid solution treated samples aged at different temperatures and time. The authors showed that SPD at 200°C allows producing an UFG (*β*+*ω*) structure of the Ti-15Mo alloy which led to the strengthening of the alloy by 1.5 times. It is found that during aging the α-particles in the UFG alloy mostly have an equiaxed morphology, as compared to the needle-like and lens shape ones in the CG-alloy. Moreover, the micro-hardness in an UFG-alloy after aging is significantly higher than in a CG-alloy.

Thermal treatment process and its effect on the micro-structural constituents of Ti alloys are another essential factors which can affect directly the HPT progress. Janecˇek *et al*. (2013) produced biocompatible UFG-Ti-6Al-7Nb alloy by HPT up to 15 revolutions. They performed solution and annealing treatments below *β*-transus temperature before HPT. It is found that the thermal treatment before HPT results in duplex microstructure with chemically heterogeneous primary α grains having 18 pct volume fraction. The primary *α* grains provided sufficient ductility for SPD, whereas the major (*α*+*β*) lamellar microstructure became more easily fragmented and hardened. The authors revealed heavily deformed primary *α* grains surrounded by significantly fragmented (*α*+*β*) microstructure after 5 HPT revolutions. Furthermore, the micro-hardness significantly increased with increasing strain, but it is heterogenous due to heterogenous microstructure. It is established that the heterogeneity of micro-hardness increases with increasing strain, suggesting that the (*α*+*β*) lamellar microstructure is more hardened than primary *α* grains. Recently, Fu *et al*. (2015) performed three different heat treatments on a cold-rolled Ti-6Al-4V alloy prior to processing by HPT at room temperature. The authors produced microstructures with various volume fractions of equiaxed and lamellar *α* and *β* phases. The authors suggested that the noticeable grain refining effect in Ti-6Al-4V (Fig. 4) is coupled with the prevalence of the lamellar structure which includes many boundaries. It is also noted that the area of low micro-hardness decreases as the volume fraction of the lamellar structure increases. The results showed that the micro-hardness increases with increasing numbers of turns in HPT and for all heat treatment conditions stable micro-hardness values are reached after about 20 turns. It is found that the grain size decreases as the volume fraction of α phase decreases and the lamellar (*α*+*β*) increases.

Sharman *et al*. (2015) presented an analysis of the micro-structural and mechanical properties of theTi–24Nb–4Zr–8Sn *β*-alloy processed by HPT at room temperature with various processing parameters. It is found that HPT leads to a significant micro-structural refinement, gradually progressing with increasing numbers of turns which induces major improvements in the

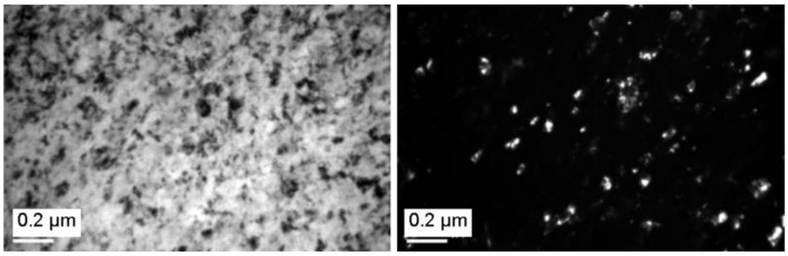

Fig. 4 The microstructure of Ti-6Al-4V alloy after HPT in bright field (left side) and dark field (right side)

mechanical properties. The authors established that the tensile strength increases from about 700 MPa in the initial state to over 1000 MPa after HPT while the E remains at a relatively low level (less than 60 GPa). Moreover, the micro-hardness measurements revealed that HPT leads to a significant growth of this value. However, the elongation to failure decreases drastically in comparison to the as-annealed alloy. The combination of the mechanical properties (high strength and low E) obtained via HPT formulates the Ti–24Nb–4Zr–8Sn alloy promising for biomedical applications as an implant material.

4. Effect of HPT on wear resistance

In general, engineering materials should demonstrate good wear resistance for use in structural applications because most of machine components involve relative motion between two surfaces (Valiev 2004).Wear behavior of implantable Ti materials is an imperative issue which requires a comprehensive and deep understanding of wear reasons, mechanisms, and techniques that can be used to improve it (Mohsin *et al*. 2013). It is well known that the wear failure is one of the main reasons of degradation (Yildiz *et al*. 2009), and thus, great difference of tribocharacteristcs between bone and implant must be limited to increase the service life of the surgical implants and to avoid bone adsorption. In practice, the influence of friction and wear in the bio-system of human body will generate metallic debris that may cause pain in the patient and lead ultimately to mechanical failure of the device (Diomidis *et al*. 2012). Unfortunately, Ti is an extremely reactive metal and has poor tribological properties reputation compared with another implantable materials (Capitanu *et al*. 2008, Mohsin *et al*. 2013). Hence, the application of Ti and its alloys under severe wear and friction conditions is severely restricted (Dai *et al*. 1997). It is important to mention here that the wear resistance of Ti materials must be taken into account in the design of biomedical parts, especially for UFG structures. It is reported that the UFG-Ti has a lower coefficient of friction than the CG-Ti under different conditions (Stolyarov *et al*. 2004). Moreover, it is pointed out the relation between the grain size and wear behavior of Ti at very low temperature (Jain *et al*. 2010). The authors proved that Ti with the smallest grain size reveals the best wear resistance owing to a higher strength and lower ductility at above condition. Therefore, a great concern has been directed to researches and studies which related to the tribological behaviour of SPD-Ti materials.

HPT is one of the main crucial techniques that is used to enhance the tribological behaviour of biomedical Ti materials. It is found that the HPT-processed nanostructured Ti gained a grain size of 5-10 nm compared to 10 and 50 µm from untreated and annealed samples, respectively. The

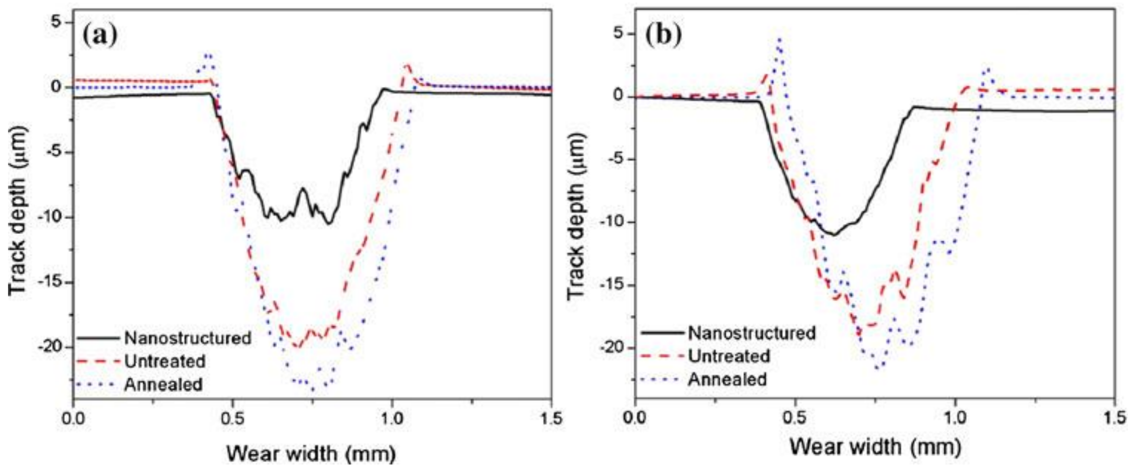

Fig. 5 Wear track cross-sectional profile of titanium with different grain sizes (a) Dry and (b) tribocorrosion experiment

HPT-processed Ti samples showed a better tribocorrosion performance either in dry or wet wear tests compared to CG-Ti samples. Fig. 5 shows a cross-section profile of the wear tracks measured by the surface profilemeter for both dry and tribocorrosion wear tests. The figure evidently reveals that the nanostructured Ti samples have the lowest wear track volume, compared with untreated and annealed Ti samples (Faghihi *et al*. 2010).

Recently, Wang et al. (2012) investigated the micro-wear behaviour of CP-Ti before and after processing by HPT to provide comparisons over a range of grain sizes. It is found that all Ti samples have a similar dynamic coefficient of friction but different wear mechanisms. Wear of the CG-Ti showed extensive plastic deformation and wedge formation which produced large wear debris whereas wear of the UFG-Ti was dominated by abrasive wear mechanisms and produced small wear debris. In addition, the UFG-Ti showed a more homogenous wear grooving and a lower wear rate than CG-Ti which suggests that UFG-Ti is more suitable for wear applications.

Recently, some researchers used new technique to improve the wear resistance of Ti materials by fabricating a coated UFG-Ti using SPD processing and surface coating technology. It comes into view that the use of a surface coating may provide a significant enhancement in the wear resistance. Accordingly, Wang *et al*. (2013a) used physical vapor deposition (PVD) to deposit TiN coatings, with a thickness of 2.5 μm, on CP-Ti samples both with and without HPT processing. It was found that the wear resistance improves if TiN coating was deposited using UFG-Ti as the substrate comparing with CG-Ti. The authors suggested that CP-Ti processed by HPT and subsequently coated with TiN provides a potentially important material for use in bio-implants. Diamond-like carbon (DLC) coatings are regarded as good candidates for the purpose of wear protection owing to their excellent mechanical and tribological properties such as high hardness and good wear behaviour. Subsequently, Wang *et al*. (2013b) deposited several DLC coatings with thicknesses of ~1.4 mm on as-received CP-Ti, HPT processed CP-Ti and Ti–6Al–4V samples via PVD. It is found that a much improved adhesion of DLC coatings with HPT processed Ti as the substrate by comparison with the same coatings on CG-Ti. The results suggested that CP-Ti processed by HPT and coated with a DLC provides a potential candidate material for bio-implant applications.

5. Conclusions

UFG and BNMs-Ti obtained by several different techniques of SPD presents a bright potential for biomedical applications. HPT is an efficient method for achieving diverse desired properties for biomedical Ti materials especially mechanical and wear properties. HPT has been increasingly investigated and recognized as valuable method of obtaining high required properties. The foregoing literature review indicates that the UFG and BNMs generated by HPT technique promote various functional mechanical and wear properties for biomedical applications that usher direction to the further developments of Ti-based materials. Many efforts from the researchers have been dedicated to improve the performance of implantable Ti materials. An overview of advanced HPT fabrication technology of UFG and BNMs-Ti materials with various mechanical and wear properties is given in this review. The critical parameters of HPT process and the possibilities of controlling the properties during the synthesis and subsequent processing procedures are also introduced and discussed.

Acknowledgments

The Author would like to sincerely acknowledge the financial assistance provided by the Government of Iraq, Ministry of High Education and Scientific Research and University of Kufa, Faculty of Engineering, Mechanical Engineering Department, Najaf, Iraq.

References

Bo, L., Yifu, S., Weiye, H. and Lei, L. (2014), "Surface modification of Ti–6Al–4V alloy via friction-stir processing: microstructure evolution and dry sliding wear performance", *Surf. Coat. Technol.*, **239**, 160-170.

Capitanu, L., Onisoru, J., Iarovici, A. and Tiganesteanu, C. (2008), "Scratching mechanisms of hip artificial joints", *Tribol. Industry*, **30**(1-2), 23-32.

Cho, T., Lee, H., Ahn, B., Kawasaki, M. and Langdon, T.G. (2014), "Microstructural evolution and mechanical properties in a Zn–Al eutectoid alloy processed by high-pressure torsion", *Acta Materialia*, **72**, 67-79.

Cojocaru, V., Raducanu, D., Gordin, D.M. and Cinca, I. (2013), "Texture evolution during ARB (Accumulative Roll Bonding) processing of Ti-10Zr-5Nb-5Ta alloy", *J. Alloy. Compounds*, **546**(5), 260-269.

Dai, Z.D., Pan, S.C., Wang, M., Yang, S.R., Zhang, X.S. and Xue, Q.J. (1997), "Improving the fretting wear resistance of titanium alloy by laser beam quenching", *Wear*, **213**(1-2), 135-139.

Diomidis, N., Mischler, S., More, N.S. and Roy, M. (2012), "Tribo- electrochemical characterization of metallic biomaterials for total joint replacement", *Acta Biomaterialia*, **8**(2), 852-859.

Edalati, K., Matsubara, E. and Horita, Z. (2009), "Processing pure Ti by high-pressure torsion in wide ranges of pressures and strain", *Metallurgic. Mater. Trans. A*, **40**(9), 2079-2086.

Eliasa, C.N, Meyers, M.A, Valiev, R.Z. and Monteiro, S.N. (2013), "Ultrafine grained titanium for biomedical applications: An overview of performance", *J. Mater. Res. Technol.*, **2**(4), 340-350.

Faghihi, S., Li, D. and Szpunar, J.A. (2010), "Tribocorrosion behaviour of nanostructured titanium substrates processed by high-pressure torsion", *Nanotechnol.*, **21**(48), 485703.

Figueiredo, R.B. and Langdom, T.G. (2012), "Fabricating ultrafine-grained materials through the application of severe plastic deformation: A review of developments in Brazil", *J. Mater. Res. Technol.*, **1**(1), 55-62.

Fu, Jie, Ding Hua, Huang, Yi, Zhang, Wenjing and Langdon T.G. (2015), “Influence of phase volume fraction on the grain refining of a Ti-6Al-4V alloy by high-pressure torsion”, *J. Mater. Res. Technol.*, **4**(1), 2-7.

Horita, Z., Furukawa, M., Nemoto, N., Barnes, A.J. and Langdon, T.G. (2000), “Superplastic forming at high strain rates after sever plastic deformation”, *Acta Mater.*, **48**(14), 3633-3640.

Islamgaliev, R.K., Kazyhanov, V.U., Shestakova, L.O., Sharafutdinov, A.V. and Valiev, R.Z. (2008), “Microstructure and mechanical proper- ties of titanium (Grade 4) processed by high-pressure torsion”, *Mater. Sci. Eng. A.*, **493**(1), 190-194.

Jain, A., Basu, B., Manoj Kumar, B.V. and Harshavardhan, Sarkar, J. (2010) “Grain size-wear rate relationship for titanium in liquid nitrogen environment”, *Acta Mater*., **58**(7), 2313-2323.

Janecˇek, M., Stra´sky´, J., Cˇi´zˇek, J., Harcuba, P., Va´clavova´, K., Polyakova, V. and Semenova, P. (2013), “Mechanical properties and dislocation structure evolution in Ti6Al7Nb alloy processed by high pressure torsion”, *Metallurgic. Mater. Trans. A*, **45**(1), 7-15.

Kent, D., Wang, G., Yu, Z., Ma, X. and Dargusch, M. (2011), “Strength enhancement of a biomedical titanium alloy through a modified accumulative roll bonding technique”, *J. Mech. Behav. Biomed. Mater.*, **4**(3), 405-416.

Kim, H.Y., Sasaki, T., Okutsu, K., Kim, J.I., Inamura, T., Hosoda, H. and Miyazaki, S. (2006), “Texture and shape memory behavior of Ti-22Nb-6Ta alloy”, *Acta Mater.*, **54**(2), 423-433.

Lin, Z., Wang, L., Xue, X., Lu, W., Qin, J. and Zhang, D. (2013), “Microstructure evolution and mechanical properties of a Ti–35Nb– 3Zr–2Ta biomedical alloy processed by equal channel angular pressing (ECAP)”, *Mater. Sci. Eng. C*, **33**(8), 4551-4561.

Meredith, C.S. and Khan, A.S. (2012), “Texture evolution and anisotropy in the thermo-mechanical response of UFG Ti processed via equal channel angular pressing”, *Int. J. Plast.*, **30-31**, 202-217.

Mohsin, T.M., Zahid A.K. and Arshad N.S. (2013), “Influence of microstructural features on wear resistance of biomedical titanium materials”, *Int. J. Chem., Nuclear, Metallurgic. Mater. Eng*., 7(1), 52-56.

Mohsin, T.M., Zahid, A.K. and Arshad N.S. (2014), “Beta Titanium Alloys: The lowest elastic modulus for biomedical applications: A review”, *Int. J. Chem., Nuclear, Metallurgic. Mater. Eng*., **8**(8), 726-731.

Niinomi, M. (1998), “Mechanical properties of biomedical titanium alloys”, *Mater. Sci. Eng. A*, **243**(1-2), 231-236.

Ozaltin, K., Chrominski, W., Kulczyk, M., Panigrahi, A., Horky, J., Zehetbauer, M. and Lewandowska, M. (2014), “Enhancement of mechanical properties of biocompatible Ti–45Nb alloy by hydrostatic extrusion”, *J. Mater. Sci.*, **49**(20), 6930-6936.

Purcek, G., Yapici, G.G., Karaman, I. and Maier, H.J. (2011), “Effect of commercial purity levels on the mechanical properties of ultrafine-grained titanium”, *Mater. Sci. Eng*., **528**(6), 2303-2308.

Raducanu, D., Vasilescu, E., Cojocaru, V.D., Cinca, I., Drob, P., Vasilescu, C. and Drob, S.I. (2011), “Mechanical and corrosion resistance of a new nanostructured Ti–Zr–Ta–Nb alloy”, *J. Mech. Behav. Biomed. Mater.*, **4**(7), 1421-1430.

Sabirov, I., Perez-Prado, M.T., Molina-Aldareguia, J.M., Semenova, I.P., Salimgareeva, G.K. and Valiev, R.Z. (2011), “Anisotropy of mechanical properties in high-strength ultra-fine-grained pure Ti processed via a complex severe plastic deformation route”, *Scripta Materialia*, **64**(1), 69-72.

Sabirov, I., Valiev, R.Z., Semenova, I.P. and Pippan, R. (2010), “Effect of equal channel angular pressing on the fracture behavior of commercially pure titanium”, *Metallurgic. Mater. Trans. A*, **41**(3), 727-733.

Sergueeva, A.V., Stolyarov, V.V., Valiev, R.Z. and Mukherjee, A.K. (2001), “Advanced mechanical properties of pure titanium with ultrafine grained structure”, *Scripta Materialia*, **45**(7), 747-752.

Shahmir H., Nili-Ahmadabadi M., Huang Y. and Langdon T.G. (2014), “Evolution of microstructure and hardness in NiTi shape memory alloys processed by high-pressure torsion”, *J. Mater. Sci.*, **49**, 2998-3009.

Sharman, K., Bazarnik, P., Brynk, T., Bulutsuz, A.G., Lewandowska, M., Huang, Y. and Langdon, T.G. (2015), “Enhancement in mechanical properties of a β-titanium alloy by high-pressure torsion”, *J. Mater. Res. Technol.*, **4**(1), 79-83.

Stolyarov, V.V., Shuster, L.S., Migranov, M.S., Valiev, R.Z. and Zhu, Y.T. (2004), “Reduction of friction coefficient of ultrafine-grained CP titanium”, *Mater. Sci. Eng. A*, **371**(1), 313-317.

Stolyarov, V.V. (2011), "Mechanical and functional properties of titanium alloys processed by severe plastic deformation", *Materials Science Forum*.

Svetlana, G., Irina, S., Milos, J. and Josef, S. (2014), "Effect of high pressure torsion on the aging kinetics of *β*-titanium Ti-15Mo alloy", *6th International Conference on Nanomaterials by Sever Plastic Deformation, Materials Science and Engineering*, Metz.

Topolski, K., Garbacz, H., Wiecinski, P., Pachla, W. and Kurzydłowski, K.J. (2012), "Mechanical properties of titanium processed by hydrostatic extrusion", *Archiv. Metallurgy Mater.*, **57**(3), 863-867.

Tsuji, N., Ito, Y., Saito, Y. and Minamino, Y. (2002), "Strength and ductility of ultrafine grained aluminum and iron produced by ARB and annealing", *Scripta Mater*., **47**(12), 893-899.

Valiev, R. (2004), "Nanostructuring of metals by SPD for advanced properties", *Nat. Mater.*, **3**, 511-516.

Valiev, R.Z. and Alexandrov, I.V. (2002), "Paradox of strength and ductility in metals processed by severe plastic deformation", *J. Mater. Res.*, **17**(1), 5-8.

Valiev, R.Z., Islamgaliev, R.K. and Alexandrov, I.V. (2000), "Bulk nanostructured materials from severe plastic deformation", *Prog. Mater. Sci.*, **45**(2), 103-189.

Valiev, R.Z., Ivanisenko, V., Rauch, E.F. and Baudelet, B. (1996), "Structure and deformation behaviour of Armco iron subjected to severe plastic deformation", *Acta Materialia*, **44**(12), 4705-4712.

Valiev, R.Z., Semenova, I.P., Jakushina, E., Latysh, V.V., Rack, H., Lowe, T.C., Petruˇzelka, J., Dluhoˇs, L., Hruˇsak, D. and Sochova, J. (2008), "Nanostructured SPD processed titanium for medical implants", *Mater. Sci. Forum*, **584-586**, 49-54.

Valiev R.Z., Sergueeva A.V. and Mukheijee A.K. (2003), "The effect of annealing on tensile deformation behavior of nanostructured SPD titanium", *Scripta Mater.*, **49**(7), 669-674.

Valiev, R.Z., Zehetbauer, M.J., Estrin, Y., Ho¨ppel, H.W., Ivanisenko, Y., Hahn, H., Wilde, G., Roven, H.J., Sauvage, X. and Langdon, T.G. (2007), "The innovation potential of bulk nanostructured materials", *Adv. Eng. Mater*., **9**(7), 527-533.

Valiev, R.Z., Zhilyaev, A.P. and Langdon, T.G. (2014), *Bulk Nanostructured Materials: Fundamentals and Applications*, Wiley & Sons, New Jersey, USA.

Wadood, A., Inamura, T., Yamabe-Mitarai, Y. and Hosoda, H. (2013), "Strengtheningof b Ti–6Cr–3Sn alloy through *β* grain refinement, a phase precipitation and resulting effects on shape memory properties", *Mater. Sci. Eng. A*, **559**, 829-835.

Wang, C.T., Escudeiro, A., Polcar, T., Cavaleiro, A., Wood, R.J, Gao, N. and Langdon, T.G. (2013b), "Indentation and scratch testing of DLC-Zr coatings on ultrafine-grained titanium processed by high-pressure torsion", *Wear*, **306**(1), 304-310.

Wang, C.T., Gao, N., Gee, M.G., Wood, R.J. and Langdon, T.G. (2012), "Effect of grain size on the micro-tribological behavior of pure titanium processed by high-pressure torsion", *Wear*, **280-281**, 28-35.

Wang, C.T., Gao, N., Gee, M.G., Wood, R.J. and Langdon, T.G. (2013a), "Processing of an ultrafine-grained titanium by high-pressure torsion: an evaluation of the wear properties with and without a TiN coating", *J. Mech. Behav. Biomed. Mater.*, **17**, 166-175.

Wang, Y.C. and Langdon, T.G. (2013c), "Influence of phase volume fractions on the processing of a Ti-6Al-4V alloy by high-pressure torsion", *Mater. Sci. Eng. A*, **559**, 861-867.

Xu, C., Horita, Z. and Langdon, T.G. (2008), "The evolution of homogeneity in an aluminum alloy processed using high-pressure torsion", *Acta Materialia*, **56**(18), 5168-5176.

Xu, W., Edwards, D.P., Wu, X., Stoica, M., Calin, M., Kühn, U. and Xia, K. (2013), "Promoting nano/ultrafine-duplex structure via accelerated *α* precipitation in a *β*-type titanium alloy severely deformed by high-pressure torsion", *Scripta Materialia*, **68**(1), 67-70.

Yang, X., Okabe, Y., Miura, H. and Sakai, T. (2012), "Effect of pass strain and temperature on recrystallisation in magnesium alloy AZ31 after interrupted cold deformation", *J. Mater. Sci.*, **47**(6), 2823-2830.

Yildiz, F., Yetim, A.F., Alsaran, A. and Efeoglu, I. (2009), "Wear and corrosion behaviour of various surface treated medical grade titanium alloy in biosimulated environment", *Wear*, **267**(5), 695-701.

Yilmazer, H., Niinomi, M., Cho, K., Nakai, M., Hieda, J., Sato, S. and Todaka, Y. (2014), "Nanostructure of *β*-type titanium alloys through severe plastic deformation", *Adv. Mat. Lett.*, **5**(7), 378-383.

Yilmazer, H., Niinomi, M., Nakai, M., Cho, K., Hieda, J., Todaka, Y. and Miyazaki,T. (2013), "Mechanical properties of a medical β-type titanium alloy with specific microstructural evolution through high-pressure torsion", *Mater. Sci. Eng. C*, **33**(5), 2499-2507.

Yilmazer, H., Niinomi, M., Nakai, M., Hieda, J., Todaka, Y., Akahori, T. and Miyazaki, T.J. (2012), "Heterogeneous structure and mechanical hardness of biomedical β-type Ti-29Nb-13Ta-4.6Zr subjected to high-pressure torsion", *Mech. Behav. Biomed. Mater.*, **10**, 235-245.

Zhang, Y., Figueiredo, R.B., Alhajeri, S.N., Wang, J.T., Gao, N. and Langdon, T.G. (2011), "Structure and mechanical properties of commercial purity titanium processed by ECAP at room temperature", *Mater. Sci. Eng. A*, **528**(25), 7708-7714.

Zhao, X., Fu, W., Yang, X. and Langdon, T.G., (2008), "Microstructure and properties of pure titanium processed by equal-channel angular pressing at room temperature", *Scripta Materialia*, **59**(5), 542-545.

Zhilyaev, A.P. and Langdon, T.G. (2008), "Using high-pressure torsion for metal processing: fundamentals and applications", *Prog. Mater. Sci.*, **53**(6), 893-979.

DH