ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 5, May 2015

Mechanical Properties of ECAP-Biomedical Titanium Materials: A Review

Mohsin Talib Mohammed [1], Shahir Hussain [2]

Ph.D Scholar, Department of Mechanical Engineering, Jamia Millia Islamia, New Delhi-110025, India[1]

Lecturer, Department of Electrical Engineering, College of Engineering, Jazan University, Jazan City, KSA[2]

ABSTRACT: Wide use of titanium (Ti) materials in medicine gives impetus to a search for development new techniques with elevated properties such as strength, corrosion resistance and Young's modulus close to that of bone tissue. This article presents most recent state of the art on the use of equal channel angular pressing (ECAP) technique in evolving mechanical characteristics of the ultrafine-grained bio-grade Ti materials. Over past few decades, research activities in this area have grown enormously and have produced interesting results, including achieving combination of conflicting properties that are desirable for biomedical applications by severe plastic deformation (SPD) processing. A review of the most recent work in this area is systematically presented. The challenges in processing ultrafine-grained Ti materials are identified and discussed. An overview of the biomedical Ti alloys processed with ECAP technique is given in this review, along with a summary of their effect on the important mechanical properties that can be achieved by SPD processing. The paper also offers insights in the mechanisms underlying SPD.

KEYWORDS: Mechanical properties, ECAP, SPD, Titanium, Biomedical applications.

I. INTRODUCTION

Titanium based materials are well recognized as promising functional materials for biomedical applications due to their good mechanical properties, superior corrosion behaviour and excellent biocompatibility. Demands of divest combination of requisite properties encourage the researchers to develop new materials and processing techniques for materials in the field of implantlogy. Micro-structural features, including grain size, phase combinations, the state of grain boundaries, and lattice defects, play a major role in improving the desired biomedical properties. However conventional processes such as solution treatment, cold rolling, aging treatment do not suffice to achieve a balance among all the required properties such as high strength while maintaining low Young's modulus etc. During past two decades, SPD techniques have been developed increasingly with the objective of creating ultrafine-grained (UFG) materials. SPD is regarded as one of the most efficient metalworking technologies for fabrication of bulk nano-crystalline materials [1]. Typically, the SPD techniques transform the micro-grained aggregates to the nano-size by accumulation and rearrangement/annihilation of the crystal lattice defects and mainly the dislocations. Several researches have revealed, that the micro to nano scale transformations take place due to a heavy of plastic deformation which usually exceeds the maximum equivalent strain achievable in simple plastic formation [2-3]. Therefore, the microstructure can be refined and tailored to have grain sizes of sub-micrometre (between 100 nm and 1000 nm) or nanometre (less than 100 nm) [4-5]. The UFG structure can be produced by several SPD techniques, such as equal channel angular pressing (ECAP), accumulative roll-bonding (ARB), high pressure torsion (HPT) and others [6-8]. The properties of nano-materials produced by these techniques essentially depend upon processing method applied and the technical parameters. It is also established that the use of SPD techniques allow formulating materials with excellent properties like hardness, strength, plasticity, corrosion and biocompatibility. Currently, these UFG materials have an a huge potential for use in highly advanced applications especially in the field of medicine [9].

A large number of studies in terms of extensive experimentation and focus have been dedicated by engineers and materials scientists in the development of Ti materials for various biomedical applications with superior required mechanical properties. There exists ever increasing importance of the subject, growing research interest and a vast unexplored potential which is the main driving force behind this literature review. There are only a limited number of

ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 5, May 2015

reports currently available on the mechanical characteristics of ECAP-Ti materials for medical applications. Hence, the present review study has been done with a view to provide the researchers to have a bird's eye view on the hotspots on research in the field of biomedical Ti alloys.

II. EFFECT OF ECAP ON MECHANICAL PROPERTIES

Investigations on the behaviour of severe plastically deformed Ti materials have been greatly motivated by the expectations that the processed parts possess unique combination of properties as also to understand the fundamental mechanisms underlying the specific properties associated with extreme grain refinement. Hardness, strength, ductility, fatigue strength and Young's modulus 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 ductility, along with low Young's modulus (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:

$$\sigma_y = \sigma_0 + K_{HP} d^{-1/2} \quad (1)$$

In Eqn-1, σ_0 is the known as friction stress and K_{HP} is a material constant. The Hall-Petch relationship establishes that the mechanical properties directly relate to the grain size [10]. The UFG and nano-crystalline (NC) materials are found to exhibit exceptionally good mechanical properties, such as high strength, toughness, and superelasticity at ambient temperatures compared with their coarse grained counterparts [11, 12]. 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 behaviour 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 [13]. From the practical point of view, it is important to mention that the ECAP (Fig. 1) is the prominent technique and recent studies have shown great potential for its use in biomedical applications. The ECAP enables to impart extremely high simple shear deformations on large billet of metals and alloys [8, 14]. The main advantage of ECAP over other SPD techniques is its ability to maintain the net dimensions of billets [15]. In SPD the billet is pressed through special die with equal channels that make an angle in the range of 90-120°. The billet can be subjected to several ECAP passes in order to increase the total strain introduced into the billet. Several factors influence the workability and the micro-structural characteristics of ECAPed materials such as the specification of ECAP facility, the angles within the die between the two parts of the channel and the outer arc of curvature where the channels intercept. Moreover, experimental factors like the speed and temperature of pressing, processing route and the number of passes influence the grain refinement and the homogeneity of the microstructures and textures of the pressed material [16]. Subsequent thermo-mechanical processing (TMP) enables to produce billets with various shapes and dimensions and also contributes to additional strengthening of the alloy [17]. It can be inferred from the literature that after multiple passes of ECAP under different conditions, very fine grain size of the order of 200–300 nm can be obtained which results in a 1.2-2.6-fold increase in yield stress and tensile ultimate strength. The yield strength of ECAPed pure Ti is found to be in the range of 450–800 MPa whereas the ultimate strength in the range of 660–820 MPa. Moreover, the ductility after ECAP was in the 10–20% range, which is sufficient for structural application. The ECAP alone may not result is requisite refine as reported in the latest literature [18]. Jian et al. (2015) reported that the strength of ECAPed pure Ti was lower and merely at par with that of Ti–6Al–4V alloy. The limited enhancement in strength related to the limited grain size refining capability of the ECAP alone [18]. A large number of investigations are being carried out to further enhance the strength by superposing cold working or SPD processes after the ECAP processing on CP Ti. These hybrid processes result in further grain refinement and/or introduce more dislocations which improve the strength. With these strategies strengths above 1 GPa is reported to be obtained, which are typically higher than that of Ti–6Al–4V [18]. Xu et al. [19] utilized back pressure equal channel angular pressing (BP-ECAP) to process a Ti-29Nb-13Ta-2Zr β alloy at different temperatures. The revealed that the temperature range for ECAP in order to control the resulting microstructure as a variety of phase transformations might occur in this metastable β-Ti alloy at elevated temperatures. They found that the ECAP at 673 K led to the occurrence of omega (ω) phase in alloy and resulted in higher strength and hardness but less ductility compared with those in the alloy processed at a a higher temperature of 903 K.

ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 5, May 2015

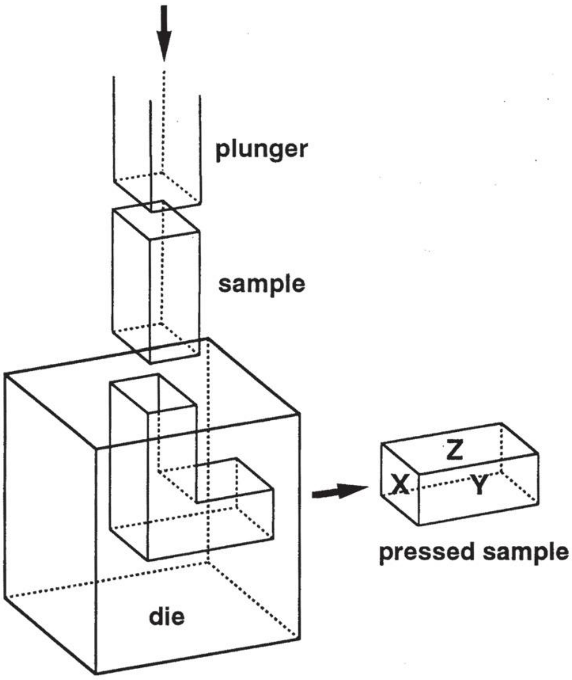

Fig. 1 – Schematic of ECAP using a die with a channel angle of 90^{o} [20]

Miroslav et al. [21] investigated the effect of ECAP on the microstructure and the mechanical properties of CP-Ti and demonstrated that the main mechanism of significant strengthening of Ti increases was grain refinement. The UFG-Ti has higher specific strength than ordinary Ti. Zhao et al. [22] employed multi-pass ECAP technique to extrude CP-Ti. After the first and second pass, deformation twins appeared in the CP-Ti specimen. The grains were refined to 200 nm after the eighth pass and the micro-hardness and UTS increased sharply to 2640 MPa and 790 MPa respectively with maintaining ductility to 16.8%. Byeli et al. [23] studied the influence of ECAP and nitrogen ion implantation on the structure and mechanical properties of BT1-00 Ti alloy to ensure the optimum combination of the bulk material and surface layer properties. The plastic deformation of Ti alloy results in modification of its structure and properties. In particular, transition from the random to the correlated distribution of dislocations, the appearance of dislocation walls, and the formation of nano-scale (100–300 nm) grains have been observed in Ti alloy. Moreover, the hardness of the alloy bulk dramatically increased from 1630 MPa to 2500 and 1700 MPa after ECAP and ECAP plus ion implantation respectively. The surface micro-hardness for Ti alloy increased up to 3700 MPa. The authors demonstrated that combined mechanical and nitrogen ion beam processing makes Ti alloy very promising for the manufacture of biocompatible implants for heavily loaded applications. Juno et al. [24] investigated the microstructure and the strengthening mechanisms of the Ti-6Al-7Nb (ASTM F1295) alloy after ECAP followed by TMP. The analyses have shown that the microstructure of ECAP treated alloy was composed by UFG with sizes ranging from 200 to 400 nm. The authors explained the co-existence of low- and high-angle grain boundaries with the presence of grains with unfavourable orientation to the plastic deformation. These grains can act as rigid bodies and concentrate the deformation in its surrounding areas, as an "open-die grain" mechanism. Such deformation mechanism could be attributed to the differences in the plastic behaviour between the alpha and beta Ti phases. Petr et al. [25] studied the mechanical properties of UFG CP-Ti by ECAP. A series of specimens was prepared after 1 to 10 passes through ECAP die with the channel angle of 120° at 350°C. The authors found that the microstructure is only partly refined after pass-1 and completely refined microstructure divided by high-angle grain boundaries after passes-6. Furthermore, the Micro-hardness of UFG CP Ti has been improved as it is much higher than that of coarse grained counterpart. Recently, Ján et al. [26] investigated the influence of traditional cold drawing of 20% and four-pass-ECAP performed at 460^{o}C on the strength of CP-Ti. They witnessed the presence of Widmanstätten morphology in the microstructure of the cold drawn specimens, while the ECAPed specimens possessed a super fine grained microstructure. They demonstrated that the ultimate tensile strength and hardness increased significantly after applying both the processes with the reduction in

ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 5, May 2015

ductility. The ECAPed specimens showed significantly high YS and UTS as compared to the cold drawn specimens. However, the ductility was found to be considerably lower as compared to the cold drawn specimens. The authors also showed significant increase in strength and sufficient ductility can be obtained in pure Ti that by the ECAP.

Of all the mechanical properties, resistance against fatigue failure is of utmost importance as "secondary" property in safe designing of biomedical components. The fatigue properties of Ti alloys can greatly enhance by replacing the conventional processing schemes by new more productive ones. Grain size can be regarded as a key structural factor affecting nearly all aspects of the mechanical behavior of metals, including fatigue behavior. The newer processes are reported to enhance the fatigue limit of CP-Ti to make it at par or even in excess to that of conventionally manufactured Ti alloys. This makes new processes, that produce UFG in the Ti, extremely attractive for biomedical applications and as a substitute for less bio-compatible Ti alloys in implant surgery [27]. A group from Korean Researchers [28] investigated the fatigue properties of UFG pure Ti produced by ECAP at 683 K to determine its suitability for medical applications. The tensile strength, fatigue strength and especially fatigue notch sensitivity of the treated ECAP Ti were measured and compared with Ti before ECAP. It is found that significant grain refinement of nearly equiaxed grains with an average size of about 0.3 µm is apparent when the microstructure is compared with the initial microstructure. After ECAP, the UTS was increased by 60%, while the elongation was decreased by 31%. Also, the fatigue limit of the pure Ti was improved by 67% after ECAP. Unfortunately, the treated ECAP pure-Ti showed a high notch sensitivity (K_f/K_t = 0.96) due to a large decrease in the characteristic micro-structural length which governs crack growth. The authors supported the notion that fatigue notch sensitivity will increase with decreasing grain size. Gabitova et al. [29] investigated the influence of UFG structure on fatigue properties and fracture mode in the Ti-6Al-7Nb ELI alloy. The authors processed the alloy by ECAP followed by extrusion processing. The ECAP was performed at 600^0C in 4 passes, while the extrusion process was done with 4 passes at 300^0C and 1 pass at room temperature. They demonstrated that the formation of a UFG structure with the average grain size of 250 ± 50 nm led to significant increase in strength and, consequently, enhancement of fatigue endurance limit by 40% as compared to the coarse-grained state. The presented micro-structural investigations demonstrated that the UFG microstructure significantly influences the crack paths and is very stable in the course of cyclic deformation. According to Ján et al. [26] the fatigue behaviour of CP-Ti after subjecting it to cold drawing and four-pass-ECAPed CP-Ti has been studied. The authors found that the specimens subjected to hybrid processing exhibited that the fatigue limit decreased significantly for higher numbers of cycles of torsional loading. The specimens after ECAP alone showed a higher degree of fatigue properties degradation.

III. CONCLUSION

A large number of different mechanical properties, often a contradicting combination thereof, is required in biomaterials for them to perform successfully. The foregoing literature review indicates that the UFG and NC structures generated by SPD techniques produce exotic combination of desirable mechanical properties that usher direction to the further developments of Ti-based biomaterial. The ECAP technique of different Ti-based materials is an effective way to promote various mechanical properties for biomedical applications. The review presented here also shows that the ECAP technique is recently applied on biomedical Ti materials and has not been exploited fully. There exists immense potential in this technique when used in the production of medical implants. The interest in UFG or NC-Ti materials in biomedical applications has focused renewed attention on the use of ECAP as a mean for achieving significantly enhanced mechanical properties. Many efforts have been dedicated to improve the mechanical performance of implantable Ti materials. An overview of advanced ECAP fabrication technology of UFG and NC-Ti materials with various mechanical properties is given in this paper. The mechanisms of ECAP critical process parameters thereof, and the possibilities of controlling these properties during the synthesis and subsequent processing procedures are also introduced and discussed.

REFERENCES

[1] Valiev, R. Z., "Nanostructuring of Metals by Severe Plastic Deformation for Advanced Properties", Nature Materials, vol. 3, pp. 511–516, 2004.
[2] Valiev, R. Z., Islamgaliev R. K., and Alexandrov, I. V., "Bulk Nanostructured Materials from Severe Plastic Deformation", Prog Mater Sci, vol. 45, pp. 103-189, 2000.
[3] Hughes, D. A., and Hansen, N., "Microstructure and Strength of Nickel at Large Strains", Acta Mater, vol. 48, pp. 2985-3004, 2000.

ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 5, May 2015

[4] Lowe, T. C., and Valiev, R. Z., Eds, "Investigations and Applications of Severe Plastic Deformation", Kluwer Academic Publishing: Dordrecht, Netherlands, 2000.
[5] Valiev, R. Z., and Alexandrov, I. V., "Nanostructured Materials Obtained by Severe Plastic Deformation", Logos, Moscow, 2000.
[6] Azushima, A., Kopp, R., , Korhonen, A., Yang, D. Y., Micari, F., Lahoti, G. D., Groche, P., Yanagimoto, J., Tsuji, N., Rosochowski A., and Yanagida, A., "Severe Plastic Deformation (SPD) Processes for Metals", CIRP Annal Manuf Technol, vol. 57, pp. 716-735, 2008.
[7] Valiev, R. Z., Estrin, Y., Horita, Z., Langdon, T. G., Zehetbauer, M. J., and Zhu, Y. T., "Producing Bulk Ultrafine-Grained Materials by Severe Plastic Deformation", JOM, vol. 58, pp. 33-39, 2006.
[8] Segal, V. M., Beyerlein, I. J., Tome, G. N., Chuvil'deev, V. N., and Kopylov, V. I., "Fundamental and Engineering of Severe Plastic Deformation", Nova Science Publishers Inc., New York, 2010.
[9] 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., "The Innovation Potential of Bulk Nanostructured Materials", Adv. Eng. Mater, vol. 9, pp. 527–533, 2007.
[10] Petchn, J., "The Cleavage Strength of Polycrystals", J. Iron Steel Inst., vol. 174, pp. 25-28, 1953.
[11] Tsuji, N., Ito, Y., Saito, Y., and Minamino, Y., "Strength and Ductility of Ultrafine Grained Aluminum and Iron Produced by ARB and Annealing", Scripta Mater, vol. 47, pp. 893-899, 2002.
[12] Horita, Z., Furukawa, M., Nemoto, N., Barnes, A. J., and Langdon T.G., "Superplastic Forming at High Strain Rates after Severe Plastic Deformation," Acta Mater, vol. 48, pp. 3633-3640, 2000.
[13] Niinomi, M., "Mechanical Properties of Biomedical Titanium Alloys", Mater. Sci. Eng. A, vol. 243, pp. 231–236, 1998.
[14] Valiev, R. Z., and Langdon, T. G., "Principles of Equal-Channel Angular Pressing as a Processing Tool for Grain Refinement", Prog. Mater. Sci, vol. 51, pp. 881–981, 2006.
[15] Valiev, R. Z., and Alexandrov I. V., "Bulk Nanostructured Metallic Materials: Processing, Structure and Properties", Academkniga, 2007.
[16] Kazeem, O., Oluwole, D., and Graeme, J., "Equal Channel Angular Pressing Technique for the Formation of Ultra-Fine Grained Structures", S Afr J Sci, vol. 108, 2012.
[17] Saitova, L. R., Semnova, I. P., Raab, G. I. and Valiev, R. Z., "Improvement of Mechanical Properties of Ti-6Al-4V Alloy by Methods of Equal-Channel Angular Pressing Followed by Plastic Deformation", Physics and technology of high pressures, vol. 14, pp. 19-23, 2004.
[18] Jian Liu, S. Akhtar Khan, Laszlo Takacs, S. Christopher Meredith, " Mechanical behavior of ultrafine-grained/nanocrystalline titanium synthesized by mechanical milling plus consolidation: Experiments, modeling and simulation," *International Journal of Plasticity*, vol. 64, pp. 151–163, 2015.
[19] W. Xu, X. Wu1, D. Sadedin and K. Xia, "Equal channel angular consolidation and deformation of titanium based alloys," *Materials Forum* vol. 32, 2008.
[20] Berbon, P. B., Furukawa, Horita, M., Z., Nemoto, M., and Langdon, T. G., "Influence of Pressing Speed on Microstructural Development in Equal Channel Angular Pressing", Metall Mater Trans A, vol. 30, pp. 1989–1997, 1999.
[21] Miroslav G., Radim K., Ladislav K., and Petr J., "Nanostructured Titanium for Dental Applications", Metal, vol. 2010, 2010.
[22] Zhao, X., Yang, X., Liu, X., Wang, X. , and Langdon, T., "The Processing of Pure Titanium through Multiple Passes of ECAP at Room Temperature", Materials Science and Engineering A, vol. 527, pp. 6335– 6339, 2010.
[23] Byelia, A.V., Kukareko, V. A., and Kononov, A. G., "Titanium and Zirconium Based Alloys Modified by Intensive Plastic Deformation and Nitrogen Ion Implantation for Biocompatible Implants", Journal of Mechanical Behavior of Biomedical Materials, vol. 6, pp. 89–94, 2012.
[24] Juno G., Tiago S., Ruslan Z., Veronika P., Claudemiro B., Cláudio S., Alberto M., and Walter J., "Microstructural Characterization of Ti-6Al-7Nb Alloy after Severe Plastic Deformation", Materials Research, vol. 15, pp. 786-791, 2012.
[25] Petr H., Michal H., and Pavel Z., "Mechanical Properties and Microstructure Evolution of Ultra-Fine Grained cp Ti", Metal, vol. 2013, 2013.
[26] Ján M., Marián B., and Petra L., "Influence of Deformation Technology on Fatigue Properties of Titanium", Materials Engineering, vol. 21, pp. 88-93, 2014.
[27] Yuri E. and Alexei V., "Fatigue Behaviour of Ultrafine Grained Light Alloys", in Proc. 17th European Conference on Fracture, Czech Republic, pp. 169-182, 2008.
[28] Woo-Jin K., Chang-Young H., and Ho-Kyung K., "Fatigue Strength of Ultrafine-Grained Pure Ti after Severe Plastic Deformation", Scripta Materialia, vol. 54, pp. 1745–1750, 2006.
[29] Gabitova, S. A., Polyakova, V. V. and Semenova I. P., "Enhanced Fatigue Strength of Ultrafine-Grained Ti-6Al-7Nb ELI Alloy: Microstructural Aspects and Failure Peculiarities", Rev. Adv. Mater. Sci, vol. 31, pp. 123-128, 2012.