Effect of Milling Time on the Morphological Evolution of Titanium Alloy Powder

O.S. Ogbonna¹, S.A. Akinlabi²-³, N. Madushele¹, P.M. Mashinini², A.S. Afolalu³

¹Mechanical Engineering Department, University of Johannesburg, South Africa
²Department of Mechanical and Industrial Engineering, University of Johannesburg, South Africa
³Department of Mechanical Engineering, Covenant University Ota, Nigeria

Abstract

This work examines the influence of disc milling duration on the morphological transformation and crystal reorientation of titanium alloy powder with a particle size below 90 μm. The disc milling time was varied from 2 mins to 10 mins, the morphological features of the powders were characterized through the scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffractometer (XRD). From the results, milling time had a significant effect on the morphology and the orientation of phases in the titanium alloy powder. The SEM images revealed a plate-like shape compared with the un-milled powder with a spheroidal shape. It was also observed that the flattening of the particles increased with milling time. This suggests that the powder is ductile. The oxygen content of the particles increased from 3.4 wt. % before milling to above 10 wt. %. XRD results showed that the milling time did not bring about a new phase and in the position of maximum diffraction intensity, which occurred at 2θ equal to approximately 40.6°. However, there was a decrease in the crystallite size while the lattice strain became higher as milling time increased.

Keywords: Titanium, Milling, Morphology

1. Introduction

Titanium and titanium alloys due to their improved physical, mechanical and chemical behaviour have made them be potential materials in the automotive, marine, aerospace and biomedical for various applications. However, these properties are substantially influenced by the processing parameters due to changes in the microstructure and mechanical behavior [1]. The titanium alloy powder used in this study, (45-90 μm) is a Titanium alloy with chemical composition Ti-6Al-2Sn-2Mo-2Cr-0.25Si. It is α+β type titanium alloy, composed mainly of hexagonal α crystal-structured grains with dispersed β phase. Among this group of titanium alloy, Ti-6Al-4V is the most popular and most intensively developed titanium alloy. The α+β type titanium alloys possess high ductility, high strength, high density, high corrosion resistance with reduced cold formability [2]. Mechanical milling is a powder processing technique widely employed in the production of fine metal powders from metal scraps and also in the reduction of metal powders into the desired particle size.

Disc mill, on the other hand, is a type of milling equipment often utilized for this purpose of mechanical milling of metal scraps. The samples are ground through the mechanical actions of the discs (a rotating disc and a fixed disc). The sample, when charged into the grinding compartment, is first subjected to preliminary crushing by the teeth of the grinding disc. The samples are then
transferred to the exterior part of the grinding discs via centrifugal force where they are finely ground. The ground particles fall into the sample collector through the opening between the grinding discs. Characterizing these samples regarding microstructural changes is a significant step that provides notable insight in the area of powder processing and successful technological applications [3]. This study seeks to examine the effects of milling time on the microstructural behavior of Ti-6Al-2Sn-2Mo-2Cr-0.25Si.

Several researches have investigated the influence of milling and milling variables on the properties of materials. Dikici and Sutcu [1] observed that the average particle size of Ti-6Al-4V flake powders was notably reduced by increasing the milling time and speed in a disc milling operation. However, XRD conducted in the study revealed that there is no alteration in the crystal structure of the alloy. Garroni et al. [3] in their study of the refinement of microstructure induced in the ball milling in Cu-based mixtures and individual metals, are of the opinion that the decrease in grain size induced in metals is determined by the processing technique, relative amount of metals present in the mixture and as well as the disparity between their respective hardness values. Singh et al. [4] reported an increase in the microhardness and compressive strength of ball-milled aluminium in Al-MWCNT nanocomposite compared with nanocomposite without milled aluminium powders. Milling has a notable influence on the properties of components produced via powder metallurgy due to the increased specific surface area of powders and increased diffusivity path created [4]. For example, in spark plasma sintering process, the surface area per unit mass is proportional to the thermodynamic force for sintering due to the increased number and area of contact points as well as heat generation as a result of influence on local pressure and current density [5],[6].

Chen et al. [7] realized that milling increased the rate of microstructural evolution in cold-pressing of atomized alloy powders. Li et al. [8] observed that the average particle sizes, morphology and microstructure of milled tungsten-copper alloy powders are significantly affected when milled below 48 h. However, upon the increase of milling duration from 48 h to 96 h, there were no significant alteration other than the reduction of the lattice parameter of tungsten. Several other types of research are in agreement of these microstructural evolutions induced by milling and their variation with milling time [9]-[10]. Milling and variation of milling time also influence the properties of metallic powders such as magnetic properties [11]-[12] and mechanical properties such as microhardness, fracture toughness, yield strength, tensile strength, compressive strength and ductility [13],[14],[15],[16]-[17]. The transformations in the microstructures and properties of metal powders induced by milling also depend on the type of milling employed [18].

2. Experimental Procedure

The material used for this study is titanium alloy powder with a particle size of below 90 μm but more than 45 μm manufactured and supplied by TLS Technik GmbH & Co company, South Africa. The milling of the powder was carried out on a vibratory disc-milling machine at 2 minutes intervals from 2 to 10 minutes.
The morphological analysis was carried out with scanning electron microscopy, Tescan VEGA 3 LMH type that is equipped with energy dispersive spectroscopy operated by Oxford software. A voltage of 20 kV was selected with a vacuum distance of 15 mm from the specimen to the detector. The X-ray diffraction spectroscopy was carried out on X-ray diffraction machine at 30 mA, 40 kV, with a scan speed of 0.5 deg./min and scan range from 10-90 degrees.

3. Results and Discussion

3.1 Scanning Electron Microscopy (SEM)

Representative morphological features of the titanium alloy powder milled under varying time are as shown in Figure 1. The result shows that the un-milled powder was spherical in shape. However, as the milling progressed, their spheroidal shapes became irregular with rough edges due to the repeated vibratory compression imposed on the powder by the milling disc. From the results obtained, it is evident that the powder is milled through the process of plastic deformation [1],[7]. After 10 mins of the milling process, all the particles have completely been flattened into irregular plate-like shape and observed to be significantly larger than the particles obtained after milling for 2 mins. The flattening of the powder with an increase in milling duration indicates the ductile nature of the titanium powder [25].

Figure 1: Scanning electron microscopy of titanium alloy powder between 0-10 mins milling time: (a) 0 min; (b) 2 mins; (c) 4 mins; (d) 6 mins; (e) 8 mins; and (f) 10 mins respectively.
3.2 Energy Dispersive Spectroscopy (EDX)

The elemental compositions of the titanium alloy powder obtained from the energy dispersive spectroscopy are shown in Figure 2 (a)-(f). From the graphs, on average, it was observed that milling increases the oxygen content of the powder. The oxygen content of the powder after milling increased from 3.4 % to above 10 %. Reduction in oxygen content with increased milling time was also observed in another study [1]. However, the percentage of titanium decreases with milling from 88.8 % to 66.3 %. The increase in the oxygen content imposes a challenge in certain applications such as in medical implants.

Figure 2(a-f): Representative EDX spectrum of titanium alloy powder with a milling time of 0 – 10 mins: (a) 0 min (b) 2 mins; (c) 4 mins; (d) 6 mins; (e) 8 mins; and (f) 10 mins respectively.
3.3 X-ray diffraction (XRD)

The x-ray diffraction graph of the milled powder is displayed in Figure 3. It was observed that there was no formation of new phases other than alpha titanium and aluminium titanium phases and in the position of maximum diffraction intensity, which occurred at a 20 value of 40.6° as the milling progresses from 2 mins to 10 mins. Similar observation was made in milling of Ti6Al4V powders [1]. However, for the un-milled powder at position ‘a’ as indicated in Figure 3; the formation of two peaks (Aluminium titanium and alpha titanium) at planes (200) and (100) respectively were identified.

![Figure 3: X-ray diffraction graph of the milled powder.](image)

The peak at position ‘a’ for the milled powder is an alpha-titanium phase at (100) plane. Also at the position ‘c’, the un-milled powder has two peaks aluminium-titanium phase at (201) plane and alpha-titanium phase at (101) plane at 2θ value equal to 40.65 and 41.04° respectively. For the milled powder, there is only one peak, alpha-titanium phase at (101) plane. A similar trend was observed at peak positions identified as d, e, f, g and h respectively. A reduction in the crystallite size and a rise in the lattice strain were however observed as milling duration progressed. Zhao et al [16] also noted this trend.
4.0 Conclusions

The scanning electron micrograph shows that the titanium powder is ductile and was milled by severe plastic deformation, which is a process of mechanical deformation. Milling has a notable influence on the oxygen content in the titanium powder, and this should be considered in cases whereby a certain amount of oxygen is required for certain applications. Lastly, this study confirms that mechanical milling does not result in the formation of new phases. However, it increases lattice strain and increases the crystallite sizes.
References

[1] Dikici, T. & Sutcu, M. (2017). Effects of disc milling parameters on the physical properties and microstructural characteristics of Ti6Al4V powders. Journal of Alloys and Compounds, 723, 395–400. https://doi.org/10.1016/j.jallcom.2017.06.202

[2] Peters, M. Titanium and Titanium Alloys Edited by Leyens, C and Peters, J. Weinheim: WILEY-VCH Verlag GmbH & Co. KGaA (2003)1, 1-35.

[3] Garroni, S., Soru, S. & Delogu, F. (2014). Reduction of grain size in metals and metal mixtures processed by ball milling. Scripta Materialia, Acta Materialia Inc. 88, 9–12. https://doi.org/10.1016/j.scriptamat.2014.06.012

[4] Singh, L.K., Bhadauria, A. & Laha, T. (2018). Al-MWCNT nanocomposite synthesized via spark plasma sintering : effect of powder milling and reinforcement addition on sintering kinetics and mechanical properties. Integrative Medicine Research, 1–10. https://doi.org/10.1016/j.jmrt.2018.03.005

[5] Diouf, S., Menapace, C. & Molinari, A. (2012). Study of effect of particle size on densification of copper during spark plasma sintering. Powder Metallurgy, 55, 228–34. https://doi.org/10.1179/1743290111Y.0000000019

[6] Diouf, S. & Molinari, A. (2012). Densification mechanisms in spark plasma sintering: Effect of particle size and pressure. Powder Technology, 221, 220–7. https://doi.org/10.1016/j.powtec.2012.01.005

[7] Chen, Y., Chen, T., Zhang, S. & Li, P. (2015). Effect of ball milling on microstructural evolution during partial remelting of 6061 aluminum alloy prepared by cold-pressing of alloy powders. 25, 2113–21. https://doi.org/10.1016/S1003-6326(15)63822-5

[8] Li, C., Zhou, Y., Xie, Y., Zhou, D. & Zhang, D. (2018). Effects of milling time and sintering temperature on structural evolution , densification behavior and properties of a W-20wt.% Cu alloy. Journal of Alloys and Compounds, 731, 537–45. https://doi.org/10.1016/j.jallcom.2017.10.081

[9] Yang, M., Guo, Z., Xiong, J., Liu, F. & Qi, K. (2017). Microstructural changes of ( Ti , W ) C solid solution induced by ball milling. International Journal of Refractory Metals and Hard Materials, 66, 83–7. https://doi.org/10.1016/j.ijrmhm.2017.03.008

[10] Kiran, U.R., Kumar, M.P., Sankaranarayana, M., Singh, A.K. & Nandy, T.K. (2015). High energy milling on tungsten powders. RMHM, 48, 74–81. https://doi.org/10.1016/j.ijrmhm.2014.06.025

[11] Meka, V.M., Chirantana, K. & Jayaraman, T. V. (2018) Influence on the structural and magnetic properties of pre-alloyed gas-atomized Fe 49 Co 49 V 2 powders during mechanical milling. Powder Technology, 332, 33–40. https://doi.org/10.1016/j.powtec.2018.03.038

[12] Dekhil, L., Alleg, S., Bououdina, M., Suñol, J.J. & Grenèche, J.M. (2015). Phase transformations and magnetic properties of ball-milled. Advanced Powder Technology,
[13] Ozkaya, S. and Canakci, A. (2016). Effect of the B 4 C content and the milling time on the synthesis, consolidation and mechanical properties of AlCuMg-B 4 C nanocomposites synthesized by mechanical milling. Powder Technology, 297, 8–16. https://doi.org/10.1016/j.powtec.2016.04.004

[14] Zhao, M., Zhou, Z., Tan, J., Ding, Q. and Zhong, M. (2015). Effects of ball milling parameters on microstructural evolution and mechanical properties of W-3 % Y composites. Journal of Nuclear Materials, 465, 6–12. https://doi.org/10.1016/j.jnucmat.2015.05.018

[15] Yang, C., Huang, H., Zhou, X., Li, Z., Zhou, X. and Xia, T. (2019). High-temperature stability of Ni-3 wt. % SiC NP composite and the effect of milling time. Journal of Nuclear Materials, Elsevier B.V. 467, 635–43. https://doi.org/10.1016/j.jnucmat.2015.10.044

[16] Materials, H., Debata, M., Acharya, T.S., Sengupta, P., Acharya, P.P., Bajpai, S. et al. (2017) International Journal of Refractory Metals & Hard Materials, 69, 170–9. https://doi.org/10.1016/j.ijrmhm.2017.08.007

[17] (2014) Effect of process parameters on micro and macro-properties of an Al-based nanocomposite prepared by means of mechanical milling. Journal of Alloys and Compounds, 586, S85–9. https://doi.org/10.1016/j.jallcom.2013.02.084

[18] Ağaoğulları, D., Balci, Ö. and Öveçoğlu, M.L. (2017) Effect of milling type on the microstructural and mechanical properties of W-Ni-ZrC-Y 2 O 3 composites. Ceramics International, 43, 7106–14. https://doi.org/10.1016/j.ceramint.2017.02.142

[19] Chen, M., Tang, A. & Xiao, X. (2015). Effect of milling time on carbothermic reduction of ilmenite. Transactions of Nonferrous Metals Society China, 25, 4201–4206.

[20] Rogachev, A.S., Moskovskikh, D.O., Nepapushhev, A.A., Sviridova, T.A. & Vadchenko, S.G. (2015). Experimental investigation of milling regimes in planetary ball mill and their influence on structure and reactivity of gasless powder exothermic mixtures. Powder Technology, 274, 44–52.

[21] Meka, V.M., Chirantana, K. & Jayaraman, T.V. (2018). Influence on the structural and magnetic properties of pre-alloyed gas-atomized Fe 49 Co 49 V 2 powders during mechanical milling. Powder Technology, 332, 33–40.

[22] Albaaji, A.J., Castle, E.G., Reece, M.J., Hall, J.P. & Evans, S.L. (2017). Effect of ball-milling time on mechanical and magnetic properties of carbon nanotube reinforced FeCo alloy composites. Material Design, 122, 296–306.

[23] Dekhil, L., Alleg, S., Bououdina, M., Suñol, J.J. & Grenèche, J.M. “Phase transformations and magnetic properties of ball-milled,” Advanced Powder Technology, vol. 26, no. 2, pp. 519–526, 2015.
[24] Leonel, E.C., Nassar, E.J., Ciuffi, K.J., Dos Reis, M.J. & Calefi, P.S. (2017). Effect of high energy ball milling on structure and properties of 95W-3.5Ni-1.5Fe heavy alloys. International Journal of Refractory Metals and Hard Material, 69, 170–179.

[25] Yang, C., Wei, T., Muránsky, O., Carr, D., Huang, H. & Zhou, X. (2018). The effect of ball-milling time and annealing temperature on fracture toughness of Ni-3 wt.% SiC using small punch testing. Material Characterization, 138, 289–295.

[26] Shukla, A.K., Murty, S.N., Kumar, R.S. & Mondal, K. (2013). Effect of powder milling on mechanical properties of hot-pressed and hot-rolled Cu – Cr – Nb alloy. Journal of Alloys and Compounds, 580, 427–434.

[27] Casati, R., Fiocchi, J., Fabrizi, A., Lecis, N., Bonollo, F. & Vedani, M. (2017). Effect of ball milling on the ageing response of Al2618 composites reinforced with SiC and oxide nanoparticles. Journal Alloys and Compounds, 693, 909–920.

[28] Liang, J.M., Jia, M.T., Guo, X.Q. & Zhang, D.L. (2014). Microstructural evolution and microhardness change of Al – 7wt % Si – 0.3wt % Mg alloy granules / powder particles during high energy ball milling. Material Science Engineering A, 590, 307–313.

[29] Real, C., Fombella, I., Trigo, I. & J. M. C. (2018). Effects of milling time, sintering temperature, Al content on the chemical nature, microhardness and microstructure of mechanochemically synthesized FeCoNiCrMn high entropy alloy. Journal of Alloys and Compounds, 749, 834–843.

[30] Guel, E., Gallardo, C.C., Porras, C.L. & Sanchez, R.M. (2014). Effect of process parameters on micro and macro-properties of an Al-based nanocomposite prepared using mechanical milling. Journal of Alloys and Compounds, 586, 85–89.

[31] Ağaoğulları, D., Balci, Ö. & Öveçoğlu, M.L. (2017). Effect of milling type on the microstructural and mechanical properties of W-Ni-ZrC-Y 2 O 3 composites. Ceramics International, 43(9) 7106–7114.