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Original Research Paper

Effect of ball milling on the physical and mechanical properties of the nanostructured Co–Cr–Mo powders

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ABSTRACT

In the present study, Co-based machining chips (P1) and Co-based atomized alloy (P2) has been processed through planetary ball mill in order to obtain nanostructured materials and also to comprise some their physical and mechanical properties. The processed powders were investigated by X-ray diffraction technique in order to determine several microstructure parameters including phase fractions, the crystallite size and dislocation density. In addition, hardness and morphological changes of the powders were investigated by scanning electron microscopy and microhardness measurements. The results revealed that with increasing milling time, the FCC phase peaks gradually disappeared indicating the FCC to HCP phase transformation. The P1 powder has a lower value of the crystallite size and higher degree of dislocation density and microhardness than that of the P2 powder. The morphological and particle size investigation showed the role of initial HCP phase and chemical composition on the final processed powders. In addition results showed that in the first step of milling the crystallite size for two powders reach to a nanometer size and after 12 h of milling the crystallite size decreases to approximately 27 and 33 nm for P1 and P2 powders, respectively.

1. Introduction

Metallic biomaterials are widely used for load-bearing implants because they have higher mechanical properties compared with polymer materials and ceramics [1–3]. The most important metals used for orthopedic implants include: commercially pure titanium and its alloys, cobalt-based alloys and stainless steel [4]. Among these metallic alloys, Co–Cr–Mo alloys, namely vitallium, have been widely used both as surface replacement such as hip and knee joint replacement and for total hip replacements [5]. The application of Co–Cr–Mo for surgical implants has been due to their excellent mechanical properties, biocompatibility and also their corrosion and wear resistance [6].

The microstructure and above-mentioned properties, in particular the mechanical properties, depend not only on the chemical composition but also on the manufacturing process, include: precise casting (lost wax), forging and powder metallurgy [7]. The presence of inherent defects such as shrinkage, chemical heterogeneity and large grain size, in the precise casting method, leads to low mechanical properties compared to other manufacturing process [8]. On the other hand, powder metallurgy exhibit superior mechanical and chemical properties compared with the two other methods, due to chemical homogeneity, finer grain size, fabrication of complex shaped parts, and control of percent and geometry of porosity in order to improve bone tissue in-growth [5,9].

Preparing of the powder and knowing the changes in microstructure, morphology, and grain size during the milling of powders prior to sintering step, is one of the key stages of powder metallurgy. Although there are a few researches on the manufacturing composite through powder metallurgy [7,10–12], there is a need for further information about the effect of milling on the properties of prepared powders.

Milling is one of the most effective and economical methods to produce powders with nano sized structure [11,13]. In nanostructure materials with extra fine grains, a high fraction of atoms are in grain boundaries which result in interesting physical and mechanical properties such as high strength and good fracture toughness [4]. During milling, powder particles are undergone high-energy impacts by balls [11]. The high energy impacts result in a high amount of defects such as vacancies, dislocations, grain boundaries and stacking faults in particles which in turn give rise to nanometer crystallite size and phase transformations [4].

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The aim of this work was to produce nanostructured Co-based alloy by means of milling from machining chips and gas atomized powders. The current work, focused mainly on the characterization and comparison of some physical and mechanical properties between two kinds of powders before and during milling.

2. Materials and experimental procedure

2.1. Materials and milling conditions

Machining chips (P1) of Co-based alloy bars and gas atomized powders were supplied by Nobilium (USA) and Carpenter Co. (Sweden), respectively. Fig. 1(a) and (b) shows images of as-received chips and gas atomized powder respectively. As can be seen, the machining chips have a spring-like morphology and alloy powder particles have a spherical shape with about 150 μm in diameter. The chemical compositions of these as-received materials According to the measured by aforementioned companies, are given in Table. 1. The milling process was performed in a PM 4000 Retsch planetary ball mill, using a rotational speed of 300 rpm at a constant rotation direction for 3, 6, 9 and 12 h. As the use of balls with different size improve accident collisions during the milling operation, minimize the surface layers on the balls, and also provide maximum impact energy [14,15], it was used three different stainless steel balls in size; the whole number of balls was eight: four balls with 10 mm, two balls with 19 mm, and two balls with 21.3 mm in diameter. The 150 ml stainless steel container was charged with a 12.7 g of the powders. The milling experiments were carried out with a ball to powder weight ratio of 12:1 with 3 cc ethanol as process control agent (PCA). In order to prevent oxidation of the powders the container was filled with Argon gas.

2.2. Scanning electron microscopy

The morphologies, particle size and particle size distribution of the milled powders after predetermined milling times were investigated using a scanning electron microscopic (SEM; Philips XL 30). An Image analyzer software (Image Tool, the University of Texas Health Science Center in San Antonio, 2002) was utilized to determine the particle size. In order to determine particle size distribution a number of 500 particles were chosen [16].

2.3. X-ray diffraction analysis

The structure and phase composition of the mechanically milled powders were characterized by means of a Philips X'pert-MPD X-ray diffraction instrument with Cu kα radiation (($\lambda_{Cu\ K\alpha}$ = 0.154186 nm, radiation at 30 mA and 40 kV) in the range of $35° < 2\theta < 100°$ (step size: 0.05 and time per step: 1 s).

Diffraction data were adjusted by a Gaussion-4 parameter function according to:

Table 1
Chemical composition (wt%) of as-received materials.

Code	Co	Cr	Mo	Others (C, Si, Fe, Mn)
P1	65	28	6	1
P2	61	28	5.3	5.7

$$I = I_0 + A\exp(-0.5(\theta - \theta_m/b)^2) \quad (1)$$

where I and I_0 are intensities, θ is the diffraction angle, θ_m is the diffraction angle in maximum intensity, b is equal to 24.3β that β is the width (rad) at half maximum intensity and A is a constant.

The following equation, proposed by Williamson–Hall, was used to determine the average crystallite size and internal strain in the milled powders from the XRD peak broadening [17].

$$\beta_r \cos\theta = k\lambda/l + 2A\sqrt{(\varepsilon)^2}\sin\theta \quad (2)$$

where λ represents the wavelength of the incident copper X-ray radiation (λ = 1.5404 A°), θ is the Bragg diffraction angle, l the average crystallite size, k a constant with a value of 0.89, ε the average internal strain, and A is the coefficient which depends on the distribution of strain.

The relative volume fractions of transformed ε-martensite were calculated using the expression proposed by Sage and Guilaud [18].

$$\mathrm{HCP(wt\%)} = I(10\bar{1}1)_{HCP}/(I(10\bar{1}1)_{HCP} + 1.5I(200)_{FCC}) \quad (3)$$

where $I(200)_{FCC}$ and $I(10\bar{1}1)_{HCP}$ are the integrated intensities of the (200) and $(10\bar{1}1)$ XRD peaks of the FCC and HCP phase, respectively.In order to determine dislocation density, the internal strain values ($\langle\varepsilon^2\rangle^{1/2}$) that conform to the slope of the Williamson–Hall plots, and grain sizes (l) related to each sample after predetermined time were inserted into the equation [19]:

$$\rho = 2\sqrt{3}\langle\varepsilon^2\rangle^{1/2}/l\beta \quad (4)$$

Using the value of lattice parameter (a), Burgers vector (β), which is equal to $a\sqrt{6}/3$ for the $\langle 11\bar{2}0\rangle$ direction, was calculated. Value of lattice parameter has been calculated using the following equation [16] for HCP system.

$$a = (\lambda/\sqrt{3}\sin\theta)\sqrt{h^2 + hk + k^2} \quad (5)$$

where h and k are Miller–Bravais indices in the hexagonal system of the planes that l equals to zero.

2.4. Microhardness evaluation

Vickers microhardness was measured at the load of 25 gr and the dwell time of 5 S with a microhardness tester (Micromet, Buehler, Japan). The samples were prepared by mixing a small amount of powder in glue followed by grinding and polishing

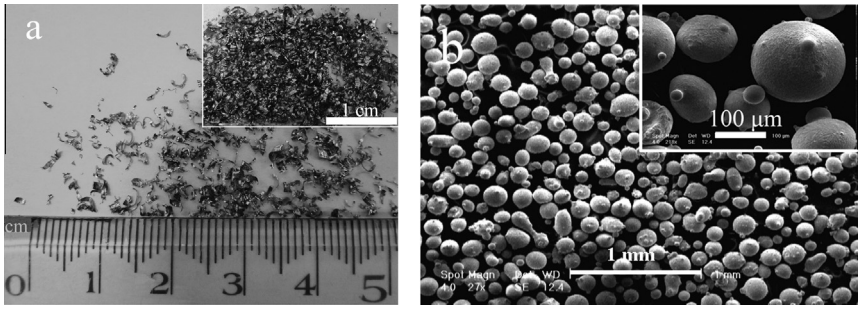

Fig. 1. The as-received materials after (a) machining (P1) and (b) gas atomizing (P2).