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Compressibility of a Ti-based alloy with varying amounts of surfactant prepared by high-energy ball milling

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ABSTRACT

The combined effects of varying amounts of surfactant (ethylene bis-stearamide; EBS) and milling time on the compressibility of ball-milled Ti–10Nb–3Mo (wt.%) alloy were investigated. Ball milling process was performed on the elemental powders with different amounts of EBS (0–3 wt.%) for 5 and 10 h, and the ball-milled powders were consolidated by a uniaxial cold pressing in the range of 500–1100 MPa. Results indicated that the addition of surfactant in ball milling process lead to significant changes in particle packing density. The relative density was higher for powders ball milled with larger amounts of EBS and for the shorter milling time. The compressibility of powders was examined by the compaction equation developed by Panelli and Ambrosio Filho. The densification parameter (A) increased with the increasing amount of EBS, and decreased with increasing milling time.

1. Introduction

The essential condition to ensure complete mechanical alloying of elemental powders in a high-energy ball milling process is the balance between cold welding and fracturing of powders. Introducing a suitable organic material, termed a surfactant, plays a critical role to minimize excessive cold-welding of powder particles and attain this balance. The surfactant is adsorbed onto the surface of powder and impedes clean metal-to-metal contact, leading to the suppression of cold welding and an increase in fracturing rate [1].

Besides the important role of surfactants in mechanical alloying, they have also been used as a lubricating agent in elementally blended powders and between powders and the die wall [2–6]. Surfactants can reduce interparticle friction and die wall friction by lubricating surfaces via short range repulsive forces [7]. Powder lubrication not only improves powder flow during the die filling stage and pressing but also can significantly reduce the ejection force needed to remove a powder compact, and therefore, leads to a longer die life and improvement on the surface of the compacts [8,9]. By mixing various amounts (0–0.8 wt.%) of ethylene bis-stearamide (EBS) with Fe–Cu–C powders, Enneti et al. [4] reported an improvement in the flowability of powders, extension of die life and reduction of friction during ejection. Despite these reported positive effects of surfactants in reducing friction between powder particles and between powders and die wall during compaction and the ejection process, surfactants can adversely affect the green strength of the powder compacts [4,10–12]. Given the importance of high green strength in minimizing scrap during part production and handling, an optimal amount of surfactant should be utilized to meet the combined requirements for ease of processing as well as suitable green strength.

Compressibility is defined as the ability of a powder to decrease in volume and form a green compact under pressure, and is quantified as the value of the compacting pressure required to attain a specified green density of the part [2,13]. The compressibility of the powder is a crucial factor in the effective production of powder metallurgical parts and design of pressing tools. Compressibility is mediated by several factors including particle shape, particle hardness, lubrication, compacting pressure, temperature, and to a lesser extent, particle size [13]. Over the past few decades, a substantial body of research has been conducted on compressibility of metal powders. Some of these studies were primarily involved in the effect of ball milling process on the compressibility of powder particles [14–19], whereas many other studies investigated the influence of surfactants, also referred to as lubricants, on compressibility of elementally blended or prealloyed powders [4–6,10,12,20]. Considering the dynamic nature of the ball milling process, the added surfactant in powder mixture is subjected to high energetic impact by the balls in a milling container. Thus, it is plausible to envisage that the combined effects of ball milling process and the presence of surfactant can considerably affect the compressibility of the ball-milled powders.

The present study attempts to investigate the combined effects of ball milling process and the addition of surfactant on compressibility of Ti–10Nb–3Mo powders prepared by mechanical alloying. The choice

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of alloy was determined from our experimental results by considering the non-toxic elements of Nb and Mo as well as a suitable combination of mechanical properties required for orthopedic and dental applications including mechanical strength, ductility and Young's modulus. In order to measure the compressibility, the relative density of the green compacts made from the ball-milled powders with different amounts of surfactants and milling time was measured as a function of pressure. Subsequently, the compressibility behavior of the green compacts was examined by the compaction equation proposed by Panelli and Ambrozio Filho [21].

2. Materials and methods

Elemental metal powders of Ti (purity 99.7%, ≤45 μm), Nb (purity 99.99%, ≤45 μm) and Mo (purity 99.99%, ≤10 μm) were purchased from Atlantic Equipment Engineers (NJ, USA) and weighed according to the predetermined stoichiometric composition of Ti–10Nb–3Mo (wt.%, hereafter) alloy. Four batches of Ti–10Nb–3Mo powders were mixed with varying amounts of 0, 1, 2, and 3 wt.% EBS $[CONHCH_2CH_3(CH_2)_{16}]_2$ as a surfactant and added in milling containers. Note that the terms "surfactant" and "lubricant" are interchangeably used in the present study. The powders were handled in a glove box chamber under argon gas to prevent atmospheric contamination. The mechanical alloying process was performed at room temperature in a planetary ball mill (Vacon®) using stainless steel containers and 10 mm steel balls. The ball-to-powder weight ratio was maintained at 20:1 and the ball milling was carried out at rotation speed of 300 rpm for 5 and 10 h.

Cylindrical samples were prepared with 10 mm diameter and 3.5 mm length using a uniaxial cold press under four compaction pressures of 500, 700, 900, and 1100 MPa. All samples were sintered in a high vacuum furnace (10^{-6} bar) at 1150 °C for 3 h at a heating/cooling rate of 10 °C min^{-1}. The morphology of the powder particles and cross section of the green and sintered compacts were characterized by scanning electron microscopy combined with secondary electron imaging (SEM–SEI) (Zeiss Supra 55VP). The distribution of elements within the particles was examined using backscattered electron imaging (SEM–BEI). Samples of each powder were characterized by PANalytical X-ray diffraction (XRD) using Cu Kα radiation (40 kV, 30 mA) at a scanning rate of 2° min^{-1} over a 2θ angular range of 30–90°. The mean particle size and particle size distribution were measured using a Malvern Instruments Mastersizer 2000 with a Hydro 2000S side feeder. Thermogravimetric analysis (TGA) was performed using thermal analyzer STA 409 PC Luxx (Netzsch) to study the thermal decomposition of the surfactant during the ball milling process via measuring the mass change of the samples as a function of temperature. For this analysis, 100 mg of ball-milled powder were heated up to 800 °C in an alumina crucible under an argon flow with a heating rate of 10 k min^{-1}. The TGA data were analyzed using Proteus software (ver. 5.2.0) from Netzsch. To measure the level of oxygen, carbon and nitrogen, chemical analysis was carried out on the powder via the Leco combustion method.

3. Results and discussion

3.1. XRD analysis and particle size-distribution of ball-milled powders

Fig. 1 shows the XRD patterns of Ti–10Nb–3Mo powder particles ball-milled with varying amounts of EBS at two different milling times of 5 and 10 h. For the milling times investigated, the diffraction patterns of the ball-milled powders without the addition of EBS were slightly broader and of less intensity than those of powders ball milled with the addition of EBS. The lower intensity peak for the sample without surfactant is more noticeable for the powders ball milled for 10 h, as seen in Fig. 1B (a), due to the increased number of crystal defects (i.e. dislocations, vacancies, stacking faults, grain boundaries, etc.)

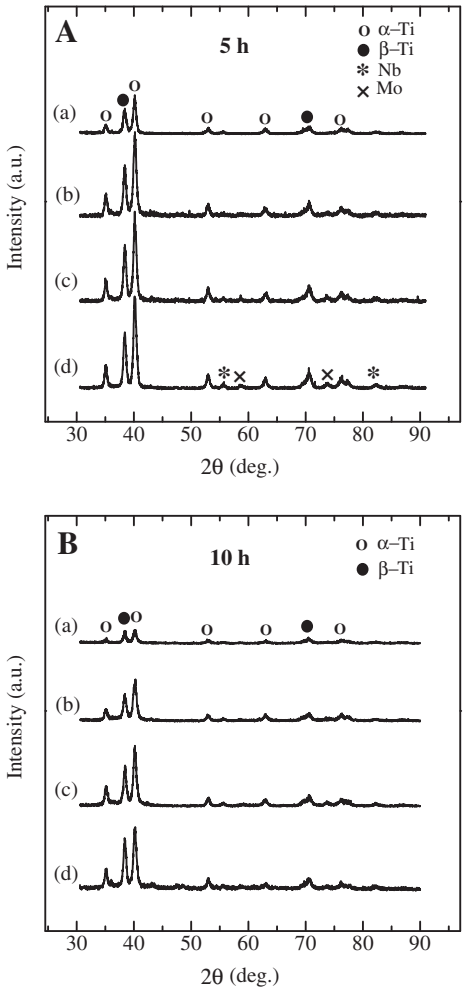

Fig. 1. XRD patterns of Ti–10Nb–3Mo powder particles ball milled for (A) 5 h and (B) 10 h at rotation speed of 300 rpm (a) without EBS; and with the addition of (b) 1 wt.%, (c) 2 wt.%, and (d) 3 wt.% EBS.

introduced after long milling times. The lower intensity peak implies the occurrence of mechanical alloying and the partial formation of a Ti-based solid solution in the absence of surfactant.

With the addition of EBS, the diffraction peaks became sharper and more intense. However, there was no significant difference between peak intensities of the powders ball-milled with the addition of different amounts of EBS for 5 h, as indicated in Fig. 1A (b,c,d). Meanwhile, the diffraction peaks belonging to Nb and Mo were still detectable in the powders ball-milled with 3 wt.% EBS, suggesting that Nb and Mo remained unalloyed after ball milling for 5 h, shown in Fig. 1A (d). The increasing intensities of α– and β–Ti peaks as well as the solute diffraction peaks with the addition of surfactant can indicate the absence of a solid solution formation. Nevertheless, after ball milling for 10 h, there was no noticeable presence of solute peaks in the powders ball milled with 3 wt.% EBS.

The particle-size distribution of the Ti–10Nb–3Mo powders ball milled with varying amounts of EBS for 5 and 10 h is shown in Fig. 2. Ball milling of powders without the addition of EBS resulted in excessive agglomeration of powders with the median size diameter (D50) of 86 μm and 159 μm for 5 and 10 h ball milling, respectively. This suggests the dominance of cold welding over fracturing during ball milling in the absence of EBS. The addition of EBS to powder particles led to a significant displacement of the curves towards the smaller particle size, due to dominance of fracturing over cold welding during ball milling process. For 5 h milling and with increasing EBS content from 1 to 3 wt.%, the mean particle size decreased from 42 μm to 36 μm, respectively. As