Influence of Ball Milling Time on Mechanical Properties of Porous Ti-20Nb-5Ag Alloy

M. J. Shivaram, Shashi Bhushan Arya, Jagannath Nayak, Bharat Bhooshan Panigrahi

***Abstract*—**Titanium and its alloys have become more significant implant materials due to their mechanical properties, excellent biocompatibility and high corrosion resistance. Biomaterials can be produce by using the powder metallurgy (PM) methods and required properties can tailored by varying the processing parameters, such as ball milling time, space holder particles, and sintering temperature. The desired properties such as, structural and mechanical properties can be obtained by powder metallurgy method. In the present study, deals with fabrication of solid and porous Ti-20Nb-5Ag alloy using high energy ball milling for different times (5 and 20 h). The resultant powder particles were used to fabricate solid and porous Ti-20Nb-5Ag alloy by adding space holder particles (NH_4HCO_3). The resultant powder particles, fabricated solid and porous samples were characterized by scanning electron microscopy (SEM). The compressive strength, elastic modulus and microhardness properties were investigated. Solid and porous Ti-20Nb-5Ag alloy samples showed good mechanical properties for 20 h ball milling time as compare to 5 h ball milling.

***Keywords*—**Ball Milling, compressive strengths, microstructure, porous Titanium alloy.

I. Introduction

POROUS titanium and its alloys are attractive materials for orthopedic applications because of their superior biocompatibility, excellent mechanical properties and high corrosion resistance [1], [2]. Porous implants are ideal materials to replace the hard tissues like bone, because it helps to restore the functions of native tissues. Introduction of porous structures into biomaterials are enabled to fabricate porous titanium and its alloys with producing suitable elastic modulus, which is tailored to that of human bone with the lowest stress-shielding effect [3].

In porous implant materials, it not only focuses on producing high mechanical property, but also focused on suitable porosity and pore morphology. It is suggested that the pore size variation must be wide range such as larger than 100-600 µm for facilitating the new bone ingrowth and to transport optimum body fluids [4]-[6]. Metallic biomaterials provide a wide range of mechanical properties and found more suitable for orthopedic applications over ceramic and polymers type biomaterials [7]. Presently, titanium based alloys such as Ti-Sn-Nb Ti-10Nb-3Mo, Ti-Nb-Zr, Ti-7.5Mo alloys [8]-[11], are very attractive biomaterials in implantation (orthopedic and dental) because they possessed lower elastic modulus and higher resistance to corrosion.

M. J. Shivaram, Shashi Bhushan Arya and Jagannath Nayak are with the Department of Metallurgical and Materials Engineering, National Institute of Technology Karnataka, Surathkal, India-575025 (e-mail: shivumj135@gmail.com, sbarya@nitk.ac.in, jagan@nitk.ac.in).

Bharat Bhooshan Panigrahi, Department of Materials science and Metallurgical Engineering, Indian Institute of Technology Hyderabad (email: bharat@iith.ac.in).

Porous titanium alloys can be produced by using different fabrication methods such as liquid metallurgy and depositions techniques. However, powder metallurgy (PM) technique has been widely used for processing porous titanium due to low cast fabrications for small size components. PM process provides adjustable pore size distribution with different level of porosities which allows to obtain a optimum mechanical properties of implant materials [12]-[14]. Previous studies have been explained the role of powder particles size, range of distributions with the shape on the sintering behavior and it directly effects on mechanical and microstructural properties of the porous titanium implants which is produced by mechanical alloying [15].

The present paper focused to examine the effect of ball milling time on microstructural and mechanical properties of solid and porous Ti-Nb-Ag alloy for biomedical application. The Ti-20Nb-5Ag (wt.%) alloys were successfully fabricated by mechanical alloying using high energy planetary ball milling.

II. Raw Materials and Powder Metallurgy Processing

A. Preparation of Porous Ti-20Nb-5Ag alloy

In this study, commercial grades powders of Titanium (<45 µm), Niobium (<45 µm), and Silver (<5 µm) were used as initial materials. Elemental powders with a composition of Ti-20Nb-5Ag (in wt.%) were milled together using high energy planetary ball milling machine (PM100). The ball to powder ratio was maintained at 10:1 and performed at room temperature with steel container and hard stainless steel balls with 250 rpm for 5 h (BM5) and 20 h (BM20). There are two different Ti-20Nb-5Ag alloy powders were obtained after ball milling for different time with the average powder particle sizes of 20-22 µm and 1-3 µm for ball milling time of 5 h (BM5) and 20 h (BM20) respectively. The morphologies of ball milled powders of BM5 and BM20 are shown in Figs. 1 (a) and (b). The two different ball milled powders mixed with 30 (wt.%) ammonium bicarbonate (NH_4HCO_3) used as space holder particles (Fig. 1 (c)). The space holder particles size ranging from 50-250 µm. The mixed powders were cold compressed into cylindrical green compacts under the load of 500 MPa. The green compacts were sintered in two steps, in the first step of the sintering process carried out at 200 ºC for 2 h to remove the space holder particles and further, temperature was raised to 1200 ºC for 3 h under the high vacuum level of 10^{-5} mbar. The fabricated porous Ti-20Nb-5Ag samples were ultrasonically cleaned and dried for further study of microstructural and mechanical properties.

The microstructural properties of solid and porous Ti-20Nb-5Ag alloy were studied using scanning electron microscope (SEM) (JEOL JSM-6380LA analytical scanning electron microscope).

III. Results And Discussion

A. Microstructural Characterization

Fig. 2 shows the morphologies of sintered solid and porous Ti-20Nb-5Ag alloys with space holder particles (NH_4HCO_3). The two different samples were produced without and with (30 wt.%) using space holder particles for milled powders BM5 and BM20. Figs. 2 (a) and (b) exhibited the solid Ti-20Nb-5Ag alloys (without space holders) for BM5 and BM20 respectively, after sintering at 1200 ºC for 3h. Both the microstrctures indicated that an excellent sintering had occurred between the ball milled powders of Ti-20Nb-5Ag alloy and high densed alloy after sintering at 1200 ºC for 3h. It did not shown any other oxides formation due to controlled atmosphere. However, it also exhibited the presence of some micro porosity which depend on time of ball milling. The morpholgy of the sintered porous Ti-20Nb-5Ag alloy fabricated by using the powders of BM5 and BM20 are shown in Figs. 2 (c) and (d) respectively. The average pore size obtained about 138 µm and distribution of pore sizes from 50 µm to larger than 400 µm are obtained after sintering.

B. Evaluation of Porosity and Mechanical Properties

The porosity was measured using the Archimedes's principle. The micro-Vickers hardness of solid and porous Ti-20Nb-5Ag alloy was measured by microhardness test instrument (Shimadzu HMV-G 20ST). The average hardness values of Ti-20Nb-5Ag alloys were obtained from five indents using a load of 1.5 kg for 30 s. A cylindrical sintered sample with a 10-mm diameter and 15 mm length was used for compression test. The compressive strength was measured using a universal testing machine (Shimadzu AG-X plus, Japan) at the strain rate of 0.001 mm/s.

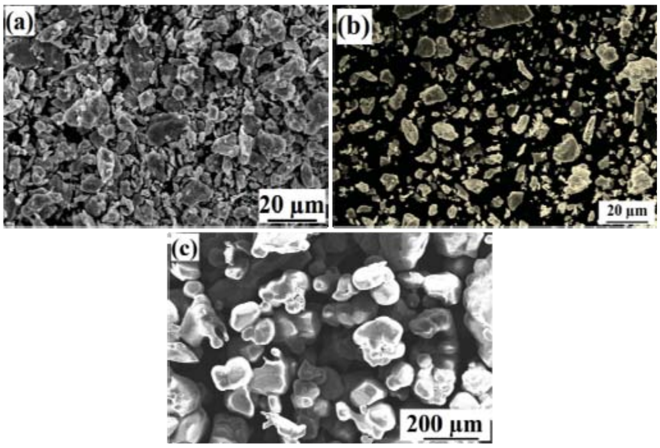

Fig. 1 SEM micrographs of (a) BM5 (b) BM20, and (c) space holder particles

Thus, the fabricated sintered porous Ti-20Nb-5Ag alloys after addition of 30 (wt.%) NH_4HCO_3 showed the preliminary requirement for porous implant materials. The pore size larger than 100 µm can be allowed for new bone tissue ingrowth [5], [6]. Finally, alloy particles were partially visible after sintering, which is connected by the sintering neck for 5 h of ball milling, however, it did not reveal any sintering neck for 20 h of ball milling (BM20). The effect of ball milling time and amount of NH_4HCO_3 particle (without and with 30 wt.%) on the porosity Ti-20Nb-5Ag sample after fabricated by using BM5 and BM20 powders as shown in Fig. 3. Fig. 3 depicts the porosity of both without and with 30 wt.% NH_4HCO_3 added samples for ball milled powders as BM5 and BM20. Sintered porous Ti-20Nb-5Ag sample produced by BM5 showed higher porosity than BM20.

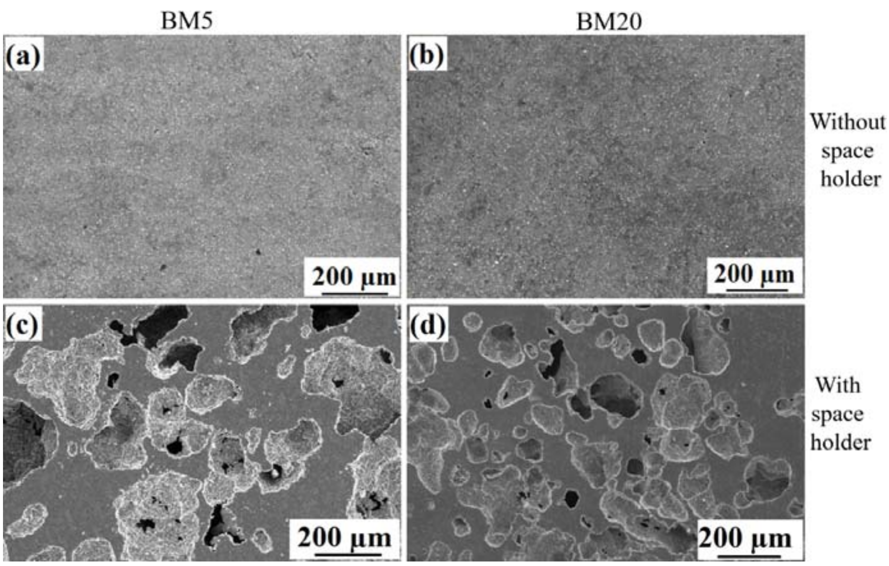

Fig. 2 Morphology of solid and porous Ti-20Nb-5Ag alloys without and with 30 (wt.%) of NH_4HCO_3 using a BM5 and BM20 powders

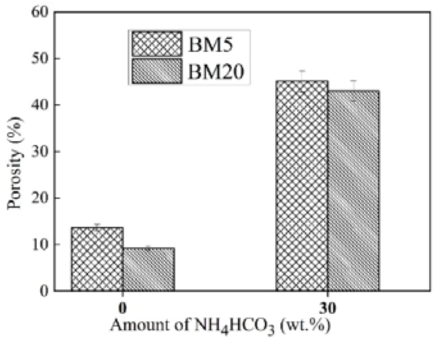

Fig. 3 Effect of NH_4HCO_3 of NH_4HCO_3 on porosity of sintered Ti-20Nb-5Ag alloys for BM5 and BM20

C. Evaluation of Mechanical Properties

The determined micro-Vickers hardness values of solid and porous Ti-20Nb-5Ag alloys are presented in the Fig. 4. The hardness values are obtained under the two different conditions namely, milled powders (BM5 and BM20) without space holder materials and with addition of 30 wt.% NH_4HCO_3.

The hardness of sintered solid Ti-20Nb-5Ag alloys (without space holder) showed significant influence on particle size and milling time. Hardness obtained for milled powder of 20h (BM20) is about 40% and 12% higher over hardness of sintered solid and porous samples obtained by BM5 (Fig. 4). The porous sintered alloys containing 30 wt.% of NH_4HCO_3 have shown lower values as compared to sintered without space holder materials.

It also clearly showed that the trivial variation of hardness values for two different time of ball milling, the hardness values is observed about 276 HV and 310 HV for ball milling of BM5 and BM20 respectively. The hardness values clearly indicated that the possible hardness changes due to reduction of particle size [7].

The mechanical properties of solid and porous Ti-20Nb-5Ag alloys were investigated by compression test on the cylindrical samples. The compressive stress-strain curve of Ti-20Nb-5Ag alloys for BM5 and BM20 powders with and without addition of NH_4HCO_3 are shown in Fig. 5. It is clearly exhibited that, the compressive strength increased with increasing time of ball milling (BM5 to BM20) for sintered solid and porous alloy.

A linear elastic deformation and followed to plateau stress region is obtained (shown in Fig. 5) from a typical compressive stress-strain plots of sintered solid and porous Ti-20Nb-5Ag samples by using ball milled powders of 5 h (BM5) and 20 h (BM20) [7].

No significant deformation of plateau stresses is found in the stress-strain curve. When the compressive stresses reached maximum and deformation continues at low stress, but brittle failure did not occure immediately due to the presence of porous structure in sintered Ti-20Nb-5Ag samples prepared by using BM5 and BM20.

The compressive strength and elastic modulus of solid and porous Ti-20Nb-5Ag samples of BM5 and BM20 are calculated from compressive stress-strain curves are shown in Figs. 6 and 7. To compare compressive strength and elastic modulus of BM5 with respect to BM20, it showed that a relatively higher compressive strength for BM20 due to small the particles size which increases the contactvity amongs the fine powders and developed a high densification during sintering. The elastic modulus of solid and porous Ti-20Nb-5Ag alloys prepared by BM5 and BM20 was determined from the initial linear slope of the stress-strain curve. The compressive strength and elastic modulus of solid and porous Ti-20Nb-5Ag alloys prepared by BM5 was observed about 1068 MPa and 45 GPa for without addition of NH_4HCO_3 and 115 MPa and 4.6 GPa (for 30 wt.% NH_4HCO_3) respectively.

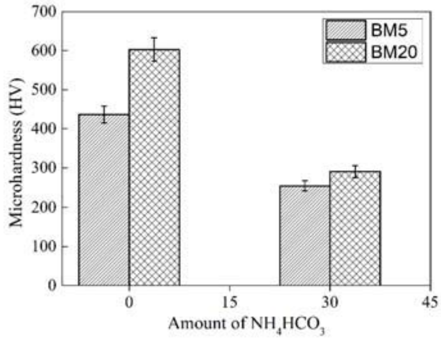

Fig. 4 Microhardness of sintered Ti-20Nb-5Ag alloy without and with addition of 30 (wt.%) NH_4HCO_3 for BM5 and BM20

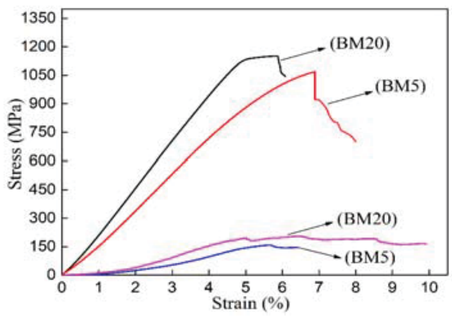

Fig. 5 Stress-strain curve of sintered Ti-20Nb-5Ag alloy without and with addition of 30 (wt.%) NH_4HCO_3

Further, an increment of compressive strength and elastic modulus of solid and porous Ti-20Nb-5Ag alloys for milled powders of 20 h (BM20) obtained about 1150 MPa and 47 GPa for without addition of NH_4HCO_3 and 205 MPa and 5.2 GPa for (with addition of 30 wt.%) respectively. The compressive strength obtained for milled powder of BM20 is about 7.6% higher over compression strength obtained by BM5.

Results indicated that, the compressive strength and elastic modulus of porous Ti-20Nb-5Ag alloys prepared by BM5 and BM20 are close to that of natural bone [8], [9] and obtained lower than cp. Ti and its alloys (90-110 GPa) [10].

Notably, the compressive strength and elastic modulus of porous Ti-20Nb-5Ag alloys synthesised by using powders of 5 h (BM5) and 20 h (BM20) are found be very closed to the cortical bone [11]. Study also revealed that the BM20 podwers have given better mechanical properties as compared to BM5 powders.

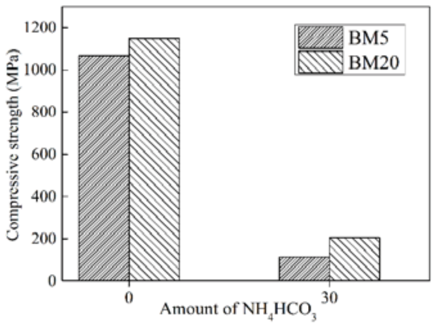

Fig. 6 Compressive strength of sintered solid and porous Ti-20Nb-5Ag of BM5 and BM20

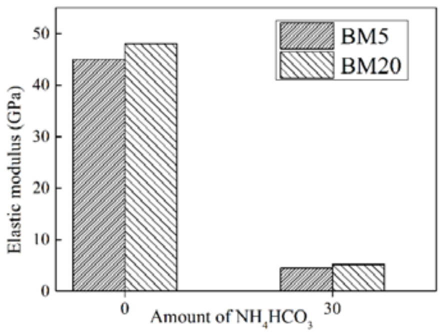

Fig. 7 Elastic modulus of sintered solid and porous Ti-20Nb-5Ag of BM5 and BM20

IV. Conclusions

Powder metallurgy (PM) method is used for fabrication of solid and porous Ti-20Nb-5Ag alloy using high energy ball milling for different times (BM5 and BM20 h). Porous structure was developed by using 30 wt.% of NH_4HCO_3 as space holder materials. A sound sintered solid products were obtained with higher hardness, compressive strength and elastic modulus for ball milled powder at 20 h (BM20) over 5 h BM5.

Porous sintered Ti-20Nb-5Ag alloy exhibited a high hardness, compressive strength and elastic modulus for milled powder at 20 h (BM20).

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