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# Mechanical Properties of A β–Ti -Xnb-3.5sn Alloy Synthesized by Mechanical Alloying and Cold Isostatic Pressure

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*Abstract:* -- **A developed □-Ti-xNb-3.5%Sn (wt%) alloy was synthesized by mechanical alloying of high energy ball milled powders and powder consolidation with cold isostatic pressure (CIP). The starting powder materials were as mixed powders and powders were produced by high energy ball milling (HEBM) for 1 hr, 2 hr and 4 hr respectively. The bulk solid samples were fabricated by the (CIP at temperature of 900-1050 oC for 2 hrs. It was found that the Ti was completely transformed from □ to □ phase after milling for 4 hr in powder state, and almost transformed to □-Ti phase with the sintered specimen at 1000 oC. The homogeneity of the sintered specimen increases as the milling time, Nb contents and sintering temperature increase. Also the hardness of the sintered alloys increases as increase of sintering temperature, Nb contents and milling time, reached to a value of 400 HV with 4 hr milling time. The Young's modulus is almost constant for all sintered Ti-x%Nb-3.5%Sn specimens at different milling time. Young's modulus is law (62.-66Gpa) compared to the standard alloy of Ti-6Al-4V about (110 Gpa).**

**Index Terms: - Biomedical materials, Ti-Nb-Sn alloys, □ titanium, mechanical alloying, mechanical properties, Young' Modulus.**

## I. INTRODUCTION

Ti has a lustrous finishing and characterized with silver color, low density and high strength. It has a high ability to resist corrosion in various media such as sea water, aqua regia and chlorine [1]. Ti is also claimed to be biocompatible since it is nontoxic nor rejected by the human body. Thus, Ti and its alloys can be used in various medical usages, e.g. surgical implements and implants, and in dentistry So that these materials and their alloys could be widely used as permanent implant materials in the replacement of damaged hard tissues such as those in artificial hip joints and teeth due to their excellent corrosion resistance, high strength and biocompatibility [2- 4]. Some of these materials have been registered as standard materials for biomedical applications, but there have been some problems for using these materials [5-6]. For example, the vanadium in the standard Ti-6Al-4V alloy which is a standard registered material for making implants is known to be a toxic element, both in the elemental state and in its oxide form. Also the applications of Ti–Ni shape memory alloys are limited by the toxicity and hypersensitivity of nickel in spite of having been applied successfully in many medical areas [7,8]. In order to overcome these problems, new Ti-based alloys with non-cytotoxic and biocompatible elements such as Nb, Ta, Sn and Mo have been developed, as a result, Nb, Ta, Mo, Zr and Sn are selected as the safest alloying elements to titanium. [9, 10]. A lower elastic modulus between the bones and implant materials is preferable for an orthopedic alloy [11]. Recently, □-titanium alloys, and Ti–Nb, and Ti–Ta based alloy systems have been studied to achieve both lower elastic modules and higher tensile strengths than commonly possible with metals and alloys. These studies mostly utilized casting processes to make the test samples of the alloys [5, 12]. In terms of the effects of alloying elements on the phase transformation of titanium alloys, some elements such as Mn, Cr, Fe, Mo, V and Nb are the □□phase stabilizing alloying elements, and some alloying elements such as Zr and Sn have principally no effect on the □-to-□ transformation temperature [13,14]. The favored titanium alloy for biomedical applications should have low elastic modulus, excellent mechanical strength, corrosion resistance, formability, and no potential toxic elements [17,18]. When the clinical durability of a renewal is considered, its wear resistance is one of the important characteristics that must be studied. However, wear testing procedures are not standardized, and many types of wear testing have been used [19,20]. In general, metals with low theoretical tensile and shear strengths exhibit higher coefficients of friction than high-strength materials [19, 21]. Therefore, in order to select a suitable

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dental material, it is the most important to understand wear behavior of experimental materials [22, 23]. The aims of this study are to characterize the microstructure, mechanical properties as well as hardness for a new biomedical material. The new alloys are prepared from Ti , Nb and Sn at different niobium contents using mechanical alloying of elemental powders and consolidation of the powders using cold isostatic pressure (CIP) at different temperatures.

## II. EXPERIMENTAL PROCEDURE

Elemental powders of high purity Ti (99.9%), 100 mesh, Nb 99.9%, and Sn 99.8% 325 mesh were used as the starting reactant materials. The powders were blended for 4 hours using a low speed milling machine in a plastic jar, with a ball to powder ratio of 10:1. The other experiments were carried out using a high-energy Ball Mill (HEBM) model TH1080. The materials were placed in a stainless steel vial with a stainless steel ball. Isopropyl alcohol (7 drops) was placed in the vial before the vial was sealed in a glove box under Ar gas atmosphere. The desired ball-to-powder weight ratio was 6:1. The milling procedure was conducted at room temperature for three different milling times (1, 2 and 4 hours) using HEBM.

The milled powder was tested by X-ray diffraction (XRD) with CuK radiation and transmission electron microscopy (TEM) operated at 200 kV for observation of the structural changes of the powders. The morphological (shape and size) changes of the produced powders were analyzed using scanning electron microscopy (SEM) operated at 15 to 25 kV. Energy dispersive spectroscopy (EDS), via SEM techniques, was used to analyze the concentration of the constituent elements. The produced powders were placed in a cylindrical stainless steel die 15 mm diameter and pressed using a uniaxial pressure of 60 MPa for 5 min was applied to the powder compact. The perfumes were sintered in a muffle furnace under Ar atmosphere at 1173, 1273 and 1373 K (900, 1000 and 1050oC). After sintering, the samples were ground and polished for subsequent indentation and microscopy studies. Density is determined by Archimedes' principle, using an accurate balance with 0.1 mg accuracy. The microstructure of the sintered samples was characterized using X-ray diffractometry (XRD) with Cu-K radiation and scanning electron microscopy (SEM) operated at 15 to 25 kV. Energy dispersive spectroscopy (EDS) via SEM was used to analyze the concentration of the constituent elements. After sintering, the samples were ground and polished for subsequent indentation and microscopy studies. Density is determined by Archimedes' principle, using an accurate balance with 0.1 mg accuracy. The microstructure of the sintered samples was characterized using X-ray diffractometry (XRD) with Cu-K radiation and scanning electron microscopy (SEM) operated at 15 to 25 kV. Energy dispersive spectroscopy (EDS) via SEM was used to analyze the concentration of the constituent elements. The hardness test was carried out using Vickers hardness (load 0.5 Kgf). The compressive strength was carried out using compression tensile test equipment on the sintered specimen at 1000℃, the Young' Modulus was estimated and compare with Ti-6Al-4V.

## III. RESULTS AND DISCUSSION

### 3.1 Sintering behavior of the powder compacts using CIP

XRD analysis was carried out to observe the phase changes during milling times as shown in Fig 1. This figure shows the XRD patterns for Ti-36%Nb-3.5%Sn powders produced by 4 hrs. From this figure, it can be seen that the intensity of the -Ti peaks is completely disappeared after 4 hr milling. Also, the intensity of the Sn peaks also is completely disappeared after 4 hr milling. The as-mixed powder contain only the -Ti peaks, Nb and Sn peaks as starting materials, thereby during mixing the reaction between the elemental phases was started. The Nb and Sn may have been dissolved in Ti phase to form a solid solution and the presence of Nb act as -Ti stabilizer as shown in Figure 1.

Figure 2 shows the SEM micrographs of the (a) as-mixed and (b) 1 hs, (c) 2 hrs, and (d) 4 hrs HBEM powders. The figure indicates that the particle size decreased with an increasing milling time. The as-mixed and 1 hr HEBM particles were plate shaped, as shown in Figures 2(a) and (b), while the particles shown in Figs. 2(c) and (d) were smaller and more rounded. From Fig. 2, the average particle sizes were 15 µm, 12 µm, 9 µm, and 7 µm for the as-mixed, 1 hr, 2 hrs., and 4 hrs. HBEM powders, respectively.

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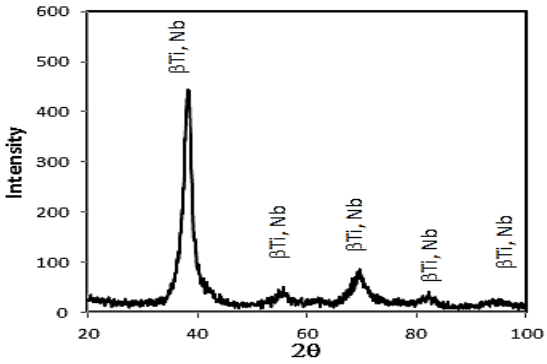


***Fig. 1 XRD diffraction patterns for Ti-36Nb-3.5Sn alloys produced by mechanical alloying at milling time 4hr using HEBM.***

Fig 3 shows the XRD patterns of the sintered samples produced by CIP using as mixed and milled powders for 1, 2 and 4 hrs. The XRD analysis shows that there are 3 phases: □-Ti, □-Ti and FeO in all sintered samples. The intensity of FeO was appeared due to Fe formed by collision between vial and steel balls. From the comparison of the intensity of the (101) plane of □-Ti phase in the XRD patterns, it is clear that the fraction of □-Ti phase in the sintered samples clearly decreased in the order of as-mixed powder, and 1 hr, 4 hr and 12 hr HEBM powders. In the meantime, the fraction of the new phase increased in the same order. Decreasing particle size made a stabilizing of □-Ti phase by Nb and made high surface area between Ti and Nb. It encourages creating good homogeneity of solid solution. It can be concluded that the sintered sample by PCAS using 4 hr HEBM powder was almost the □-Ti phase [23].

Fig. 4 shows the SEM micrographs for the sintered samples produced by CIP of different powders at 1000 oC. From this figure, the sintered samples using as mixed and 1 hr HEBM powders showed large Ti rich □-Ti phase regions as shown in Figures 4 (a) and (b). The size of the Ti rich □-Ti phase regions decreased with increasing milling time, and in the meantime, the homogeneity of the □-Ti (or new phase) particles also increased dramatically. The chemical compositions of the dark and light phases in the as mix sample sintered at 1000 oC were analyzed using EDS and marked as points 1, 2 as shown in Fig. 5(a). As shown by the EDS spectra in Figure 5, a composition of the light phase (Point 1) almost niobium while a composition of the dark or black phase (Point 2) was much closer to that of Ti. From this result, it is clear that the dark phase is □ metastable phase containing Nb, while the light phase is Nb rich □-Ti phase. This is confirmed by Ti-Nb phase diagram[24] the □-Ti phase increased with increasing Nb content at temperature lower □-Ti to □-Ti transformation temperature (882℃).

Fig 6 shows the backscattered electron SEM micrographs for the sintered samples at 1000℃ at different milling time using CIP of as mixed and milled powders for 1, 2 and 4hrs.

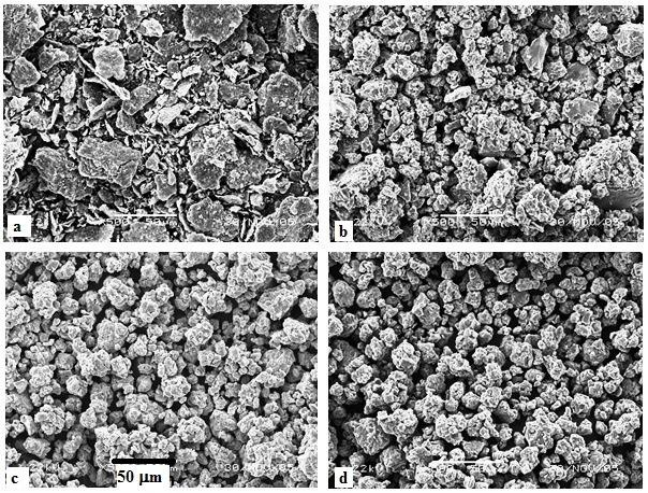


***Fig.2 Shows the SEM micrographs for Ti-36Nb-3.5Sn alloys produced by mechanical alloying at (a) as-mixed and (b) 1 h, (c) 2 h, and (d) 4 h HBEM powders***

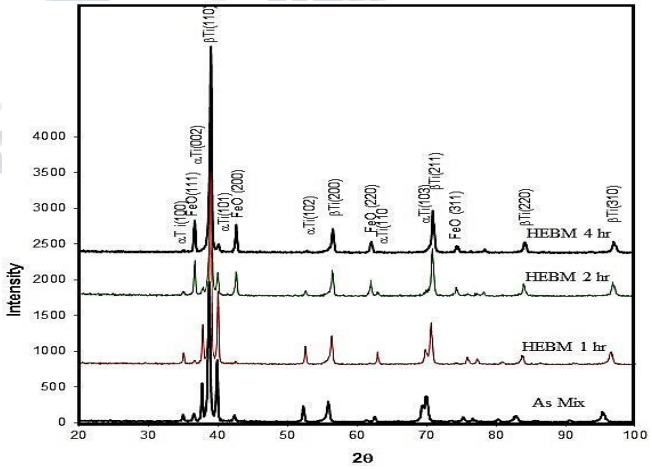


***Fig. 3 XRD diffraction patterns of the bulk samples produced by CIP of different powders at 1000℃ [29].***

From this figure, the sintered samples using as mixed consists of large white area (particles) within black matrix and the white particles increase as milling time increase. Also the particles size of the mixture decreased with increasing milling time, and in the meantime, the homogeneity of the mixture also increased dramatically.

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Fig. 7 EDS point analysis for point "1" and "2" in Fig. 6(a). The chemical compositions of the dark and light phases in the sample at (point 1) almost twice higher than that of Nb (Ti~65% and Nb ~32%), while a concentration of Nb (about 41%) of the light phase (point 2) was much closer to that of Ti (about 54%). From this result, it is clear that the dark phase is a metastable □□Ti phase containing Nb, while the light phase is Nb rich □-Ti phase. This is confirmed by Ti-Nb phase diagram [24]; the □-Ti phase increased with increasing Nb content at temperature lower than the □-Ti to □-Ti transformation temperature (882℃). Fig 8 shows X- ray mapping images indicating the distribution of Ti, Nb and Sn in the bulk samples produced by CIP at 4hrs milling and sintering temperature at 1000℃. From this figure, it can be seen that Ti, Nb and Sn formed a □-Ti based homogeneous solid solution. This was confirmed by the XRD analysis Fig 3.

The variation of relative density versus temperature of sintered specimens by CIP under a pressure of 60 MPa and sintered at 1000 oC using as mixed powder and milled powder for 1 hr, 2 hrs and 4 hrs are shown in Fig 9. The density of specimens increased with increasing temperature up to 1000℃ and then slightly decreased with temperature. The increase of density at the temperature range from 900 to 1000℃ reaches to the maximum value of 0.87 at milling time 4 hrs and the sintering temperature of 1000oC. It can be noticed that, as the temperature increases, porosities decrease might be due to the higher liquid phase transaction during sintering that could be the main reason for increasing relative density But the slight decrease of density with temperature is due to the grain gross leads to increasing the inter-granular vacancies. At the same temperature the relative density increases due to the decrease of particles size as the milling time increases.

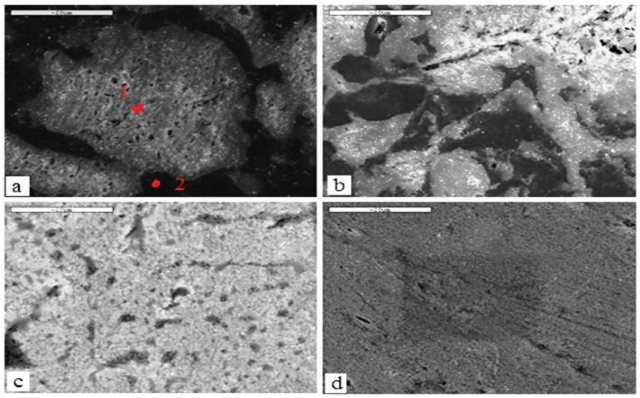


***Fig.4 Shows the SEM micrographs for Ti-36Nb-3.5Sn alloys produced by mechanical alloying sintered at 1000 oC at (a) as-mixed and (b) 1 hr, (c) 2 hrs, and (d) 4 hrs HBEM powders***

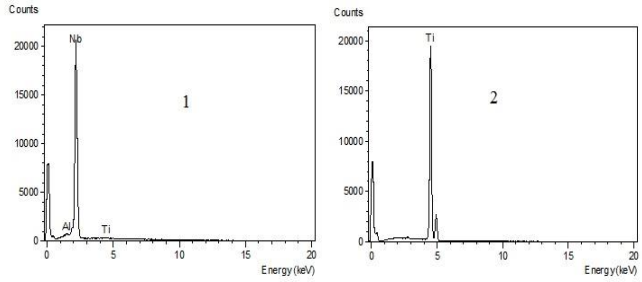


***Fig. 5. EDS point analysis for points 1 and 2 in Figure 4a***

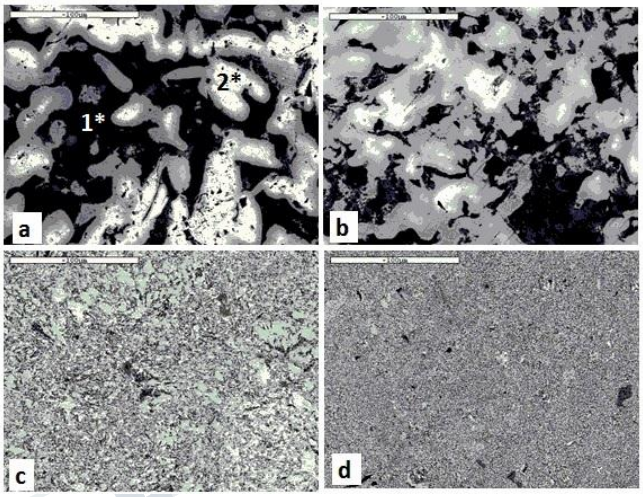


***Fig. 6 Backscattered electron SEM micrographs of the bulk samples produced by CIP of different powders at 1000℃: (a) as-mixed powder (b) 1hr milling; (c) 2hrs milling (d) 4hrs milling.***

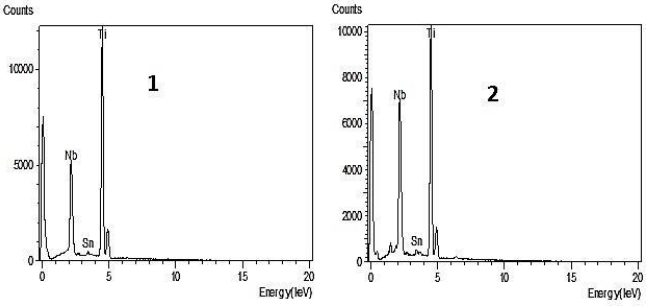


***Fig. 7 EDS point analysis for point "1" and "2" in Figure 6(d).***

### 3.3. Mechanical properties

#### 3.3.1 Hardness

Vickers hardness values (Hv) of the bulk Ti–xNb–3.5Sn alloy with different milling time and sintering temperature using CIP under identical sintering conditions are shown in

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Fig.10. The results show that the hardness of the alloy increases as the sintering temperature increases at the same milling time. In addition, for the same sintering temperature, the hardness (Hv) of the bulk alloys also increased with increasing milling time, converging to a value of 390 Hv with milling time 4 hrs using high energy ball mill and 1050℃ sintering temperature. The lowest hardness of 350 Hv was reported for samples at as mix and sintering temperature 900℃. The increasing of hardness might be due to the increasing the milling time could be improving homogenization and densification in addition to the increase of sintering temperature might reduce the porosities and increase the making of solid solution that could be a reason for increasing the hardness. Also The effect of Niobium on the Vickers hardness values (Hv) of the bulk Ti–xNb–3.5Sn alloy with different milling time at sintering temperature 1000oC using CIP are shown in Fig.11. The results indicated that slightly increasing of hardness as niobium contents increase. The increasing of Vickers hardness values with increasing niobium contents is attributed to the increasing the amount of solid solution leads to increase the homogeneity.

**3.2 Young' Modulus**

Fig. 12 shows the elastic modulus of the bulk Ti–xNb–3.5Sn alloy with different milling time and sintering temperature using CIP under identical sintering conditions.

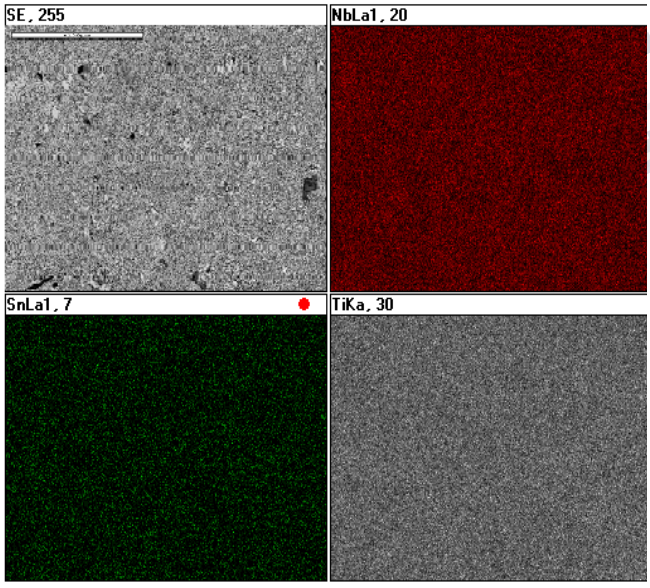


***Fig. 8 X-ray mapping images for the distribution of Ti, Nb and Sn in the bulk sample produced by CIP of 4-hrs milled powder at 1000℃.***

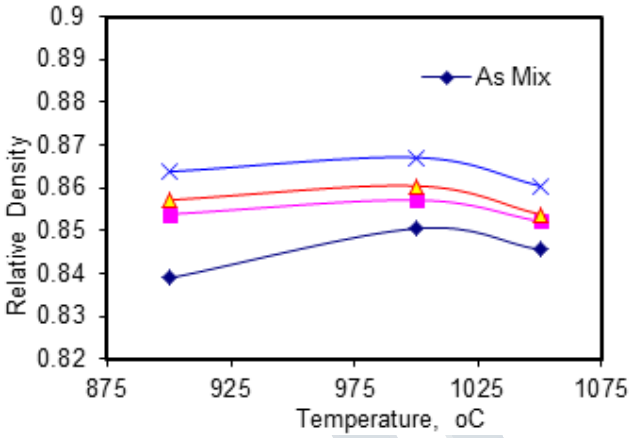


***Fig. 9 Effect of temperature on the relative density in the produced Ti-36Nb-3.5Sn alloys by CIP at different milling time .***

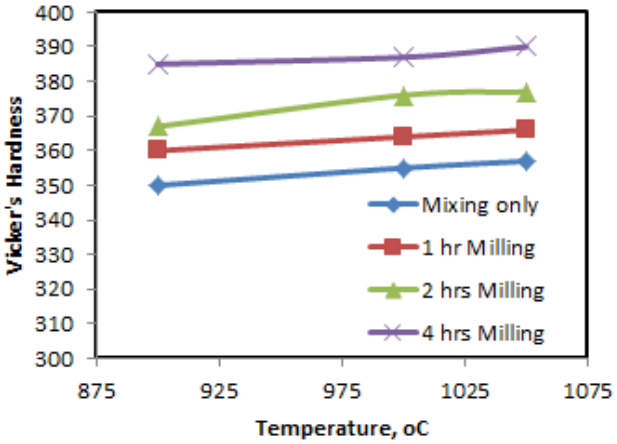


***Fig.10 Effect of the temperature on the hardness of the sintered specimens at: as-mixed, 1hr, (3) 2 hrs-, 4hrs milled***

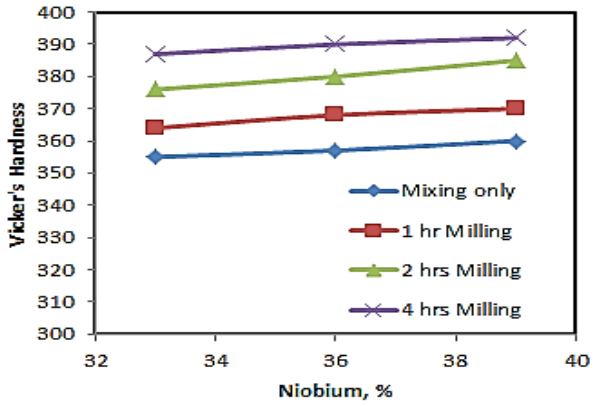


***Fig.11 Effect of Niobium contents on the Vickers hardness of the sintered specimens at***

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(1) as-mixed, (2) 1hr-milled, (3) 2 hrs-milled, (4) 4hrs-milled -The results show that the Young's modulus of the alloy decreases as the sintering temperature increase up to 1000oC then the Young's modulus increases at the same milling time. In addition, at the same sintering temperature, the Young's modulus of the bulk alloys also increased with increasing milling time, converging to a value of 66.4 GPa with milling time 4 hrs using high energy ball mill and 1050℃ sintering temperature. The lowest value of Young's modulus was reported for samples at as mix and sintering temperature 1000℃. In spite of the hardness increases with increasing the milling time, the elastic modulus is almost constant at all milling time. This due to the increasing of □□□I phase in the sintered specimens with increasing milling time leads to the Young' modulus stability [24]. while the elastic modulus of Ti-6Al-4V Ingot is about 110 MPa. It can be conclude that the addition of Nb and Sn to the sintered Ti–xNb–2.5Sn alloys is reduced the elastic modulus to two third of the elastic modulus belongs to Ti-6Al-4V ingot. Also The effect of Niobium on the Young's modulus of the bulk Ti–xNb–3.5Sn alloy with different milling time at sintering temperature 1000oC using CIP are shown in Fig.13. The results indicated that linearly decreasing of Young's modulus as niobium contents increase. The decreasing of Young's modulus values with increasing niobium contents is attributed also to the increasing of solid solution amount leads to increase the homogeneity.

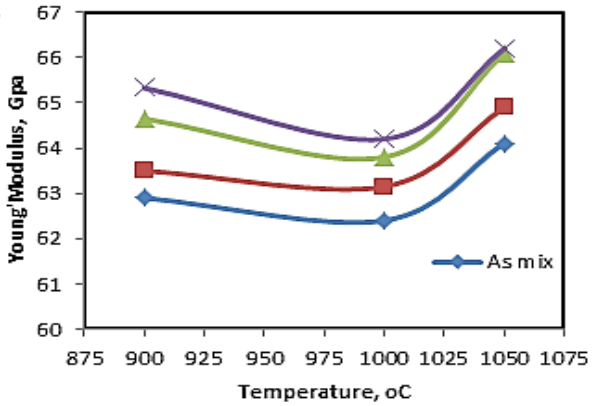


***Fig. 12 The results of young's modulus of the sintered Ti–36Nb–3.5Sn specimens at different temperature :*** as-mixed, 1hr, (3) 2 hrs-, 4hrs milled

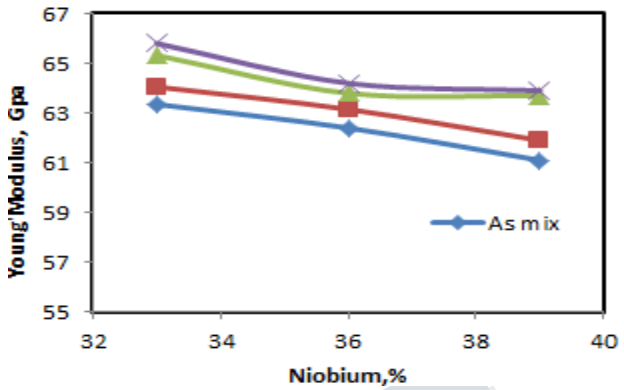


***Fig.13 Effect of Niobium contents on the Vickers hardness of the sintered specimens at*** : as-mixed, 1hr, (3) 2 hrs-, 4hrs milled

## IV. CONCLUSIONS

This study was conducted to observe the phase transformation of Ti-x%Nb-3.5%Sn during milling for various milling times using high-energy ball milling machine (HEBM). Also another objective are to observe microstructure, as well as mechanical for a new biomedical material of Ti-x%Nb-3.5%Sn sintered at different temperature and different particle sizes of milled powder.

The results are summarized as below.

1. The XRD analysis validated the complete transformation of the produced powder from □□Ti to □□Ti after 4 hrs milling time using HEBM in powder state and almost transformed to □□Ti after sintering at 1000 oC using CIP.

2. Decreasing particle size made a stabilizing of □□Ti by Nb and made high surface area between Ti and Nb encourage to create good homogeneity solid solution. and the sintered powder using CIP at 4hrs HEBM powder is almost the □□Ti.

4. The sintered temperature at 1000 oC to ensure that completely transformed from □□Ti to □□Ti.

5. The hardness of the sintered alloys is increased with increasing sintering temperature at the same milling time. Moreover, for the same sintering temperature, the hardness (Hv) of the bulk alloys also increased as the milling time and niobium contents increase, reached to a value of 395 Hv of the sintered alloys at 1050 oC using 4hrs HEBM powder. The lowest hardness of 348 Hv was reported for sintered samples at 900 oC using as mixed powder.

6. The Young' modulus of the sintered Ti–xNb–3.5Sn alloys is almost constant even if the hardness increased. The presence of Nb and Sn in the sintered Ti–Nb–3.5Sn alloys is reduced the elastic modulus comparing with the elastic modulus belongs to Ti-6Al-4V ingot.

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