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Development of a biocompatible Ti-Nb alloy for orthopaedic applications

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Abstract. Metallic biomedical implants such as titanium-based alloys are very useful for orthopaedic applications due to their excellent properties which responds to changes in temperature and other conditions. However, biological toxicity due to alloying elements and relatively high Young's modulus or mechanical incompatibilities of previously used Ti alloys have necessitated the development of biocompatible alloys with compatible mechanical properties such as beta-titanium alloys. This study aims at production of beta-titanium alloy with enhanced properties by varying milling speeds. Ti and Nb powders were mechanically alloyed using the high energy ball-mill Zoz-Simoloyer® to produce Ti-7Nb alloys by varying the milling speed. The milling process produced irregular shaped powders with increasing particles sizes as the milling speed increased due to fragmentation and cold welding during agglomeration. The mechanical alloying process had good yield. The predominant phases of the inhomogeneously milled alloy were alpha and beta phases.

1. Introduction

Titanium-based alloys are used as biomedical implants for orthopaedic applications such as knee or joint replacements due to their high strength, biocompatibility and good corrosion resistance. Ti6Al4V is a commonly used titanium-based alloy and has been reported to release aluminum and vanadium ions which causes health problems such as Alzheimer disease and ostemomalacia [1-2]. The release of these ions and their oxides also causes inflammation leading to aseptic loosening of the implant and possible to a surgery revision [1,3]. The Young's modulus of the bone is about 4-30 GPa, implants with a higher young's modulus may cause stress shielding effect which is when the implant bears most of the mechanical load shielding the bone from bearing the load required for it to maintain strength and healthy structure leading to implant failure[1,4]. The Young's modulus of titanium-based alloys is dependent

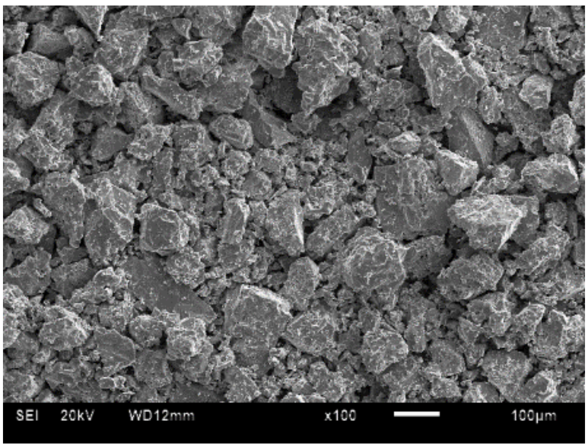
on the chemistry and phase of the alloy, with the beta-phase having the lowest and the martensitic phases having relatively low young's modulus and the ω-phase has the highest young's modulus [4-5]. Beta-stabilizing elements such as niobium, zirconium and tantalum have displayed non-toxic behaviour and they show good compatibility with direct contact with cells and relatively good cell growth and mitochondrial activity[3]. Ti-Nb alloys have been prepared by different powder metallurgy routes and casting techniques, producing alloys with different microstructures and mechanical properties [6-8].
Mechanical alloying is a process capable of preparing complex alloys with specific microstructures and good mechanical properties [9-10]. This alloying process produces homogeneous materials that are prepared by mixing pure or pre-alloyed powders and then milled together with a grinding medium such as grinding steel balls [6], [7]. It is a high energy ball milling process in which the alloying of the powder particles occurs due to fracture, plastic deformation and cold welding of particles [11-12].The mechanical alloying process makes possible the production of alloys from elemental powder that are difficult to alloy. It produces novel crystalline phases and a very small range of grain sizes up to nanometres [6]. Important parameters that mostly affect the milled products are the milling speed and time, the grinding ball to powder weight ratio and process environment.
In this study, the mechanical alloying process of Ti-7Nb was studied by varying the milling speed in order to evaluate the alloying of CP-Ti to Nb using the high energy ball milling.

2. Method and materials

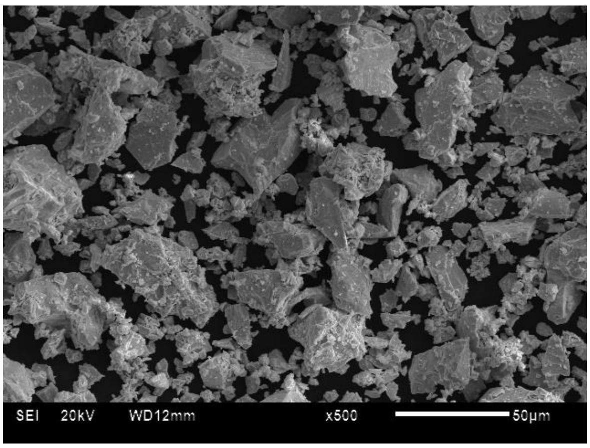
The elemental powder of titanium (99.8 % purity, -150 µm), and niobium (99.8 % purity, -25 μm) were used to prepare Ti-7Nb alloys using high-energy ball milling process. The Ti-Nb alloys were milled using the Zoz-Simoloyer® under vacuum in a steel vial and grinding balls. The Zoz-Simoloyer® was purged with argon three times prior to mechanical milling. The Ti-Nb alloys were milled for 6 hours and at milling speed of 800, 900, 100, 1100 and 1200 rpm with a ball-to-powder ratio (BPR) of 10:1.
The as received and milled powders were characterised using the Scanning Electron Microscope (Jeoul™ JSM6510 SEM) equipped with energy dispersive X-ray spectrometry (EDX). The conventional sieving particle analysis were used to determine the morphology and the particle size distribution. Some milled powders were cold mounted, ground to a 4000 grit finish under running water with SiC papers and polished with the colloidal silica to a mirror like surface finish, then characterize using SEM/EDX. The phase analysis of the as received and milled powder was done using X-ray diffraction (XRD) coupled with the PANalytical X'Pert™ Pro powder diffractometer in θ–θ configuration with an X'Celerator™ detector with Fe filtered Co-Kα radiation (λ=1.789Å).

3. Results and discussion

The CP-Ti and Nb elemental powder are shown in Figure 1 a and b respectively. Both powders exhibit irregular morphology. The particle size distribution of the CP-Ti and Nb powder is shown in Figure 1 c and d respectively. CP-Ti powder has larger particles with an average particle size of 92.71 µm and Nb powder has smaller particles with average particle size of 18.61 µm.



(a)



(b)

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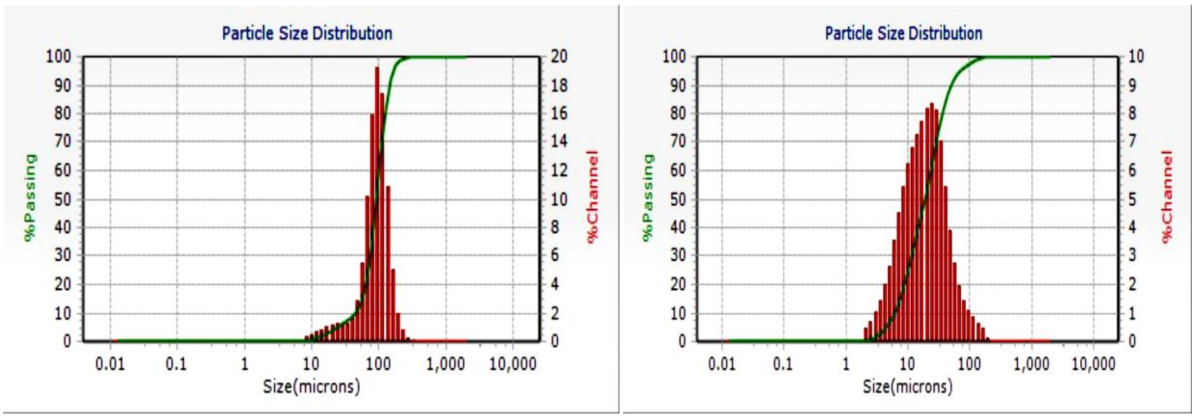

(c) (d)

Figure 1: (a) CP-Ti, (b) Nb elemental powder and, the particle size distribution of (c) CP-Ti and (d) Nb powder.

The yield of the milled powder at different milling speeds varies from 80 to 100 % as shown in Figure 2. The good recovery of the milled powders shows good yield characteristic for the mechanical milling process and non-sticking of the powder to the grinding vial and balls.

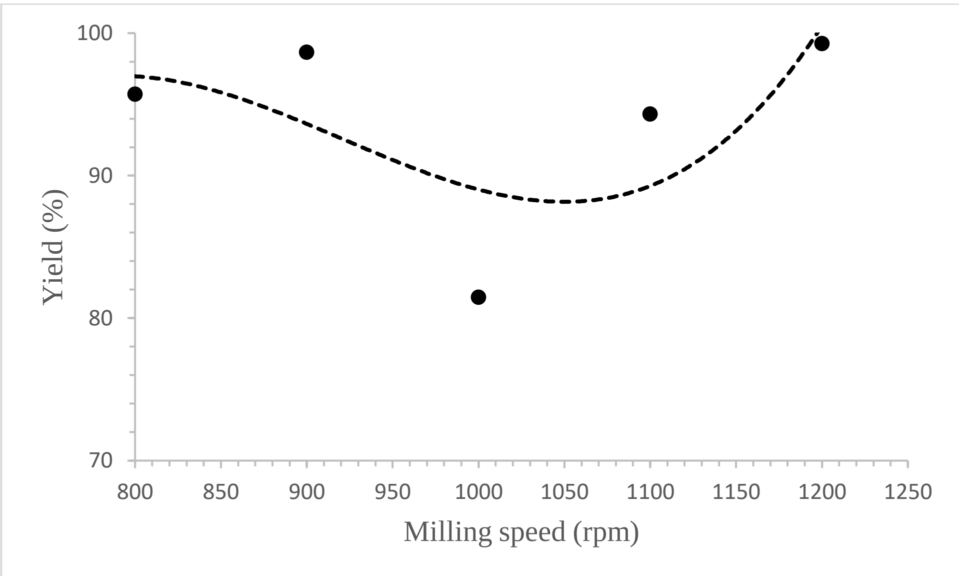

Figure 2: The yield of the milled powders at different milling speeds.

The particle size distribution of the milled powders at different milling speeds is shown in Figure 3. The particle size increases with increasing milling speed due to continuous collisions between the powder and milling medium causing agglomeration and cold welding of the powder particles.

The size of the particles increased with increasing milling speed. There is a wide distribution of size with larger particles which indicates that cold welding and agglomeration of the particles is the prevailing process than fracturing of the powder particles as the milling speed increased. The collision between the grinding balls, the walls of the vial and the powder facilitates the mixing of the powders and gives the kinetic energy for mechanical alloying the powder [9]. The elemental powders were mixed as shown by the elemental distribution in Figure 3 for all the milling speeds

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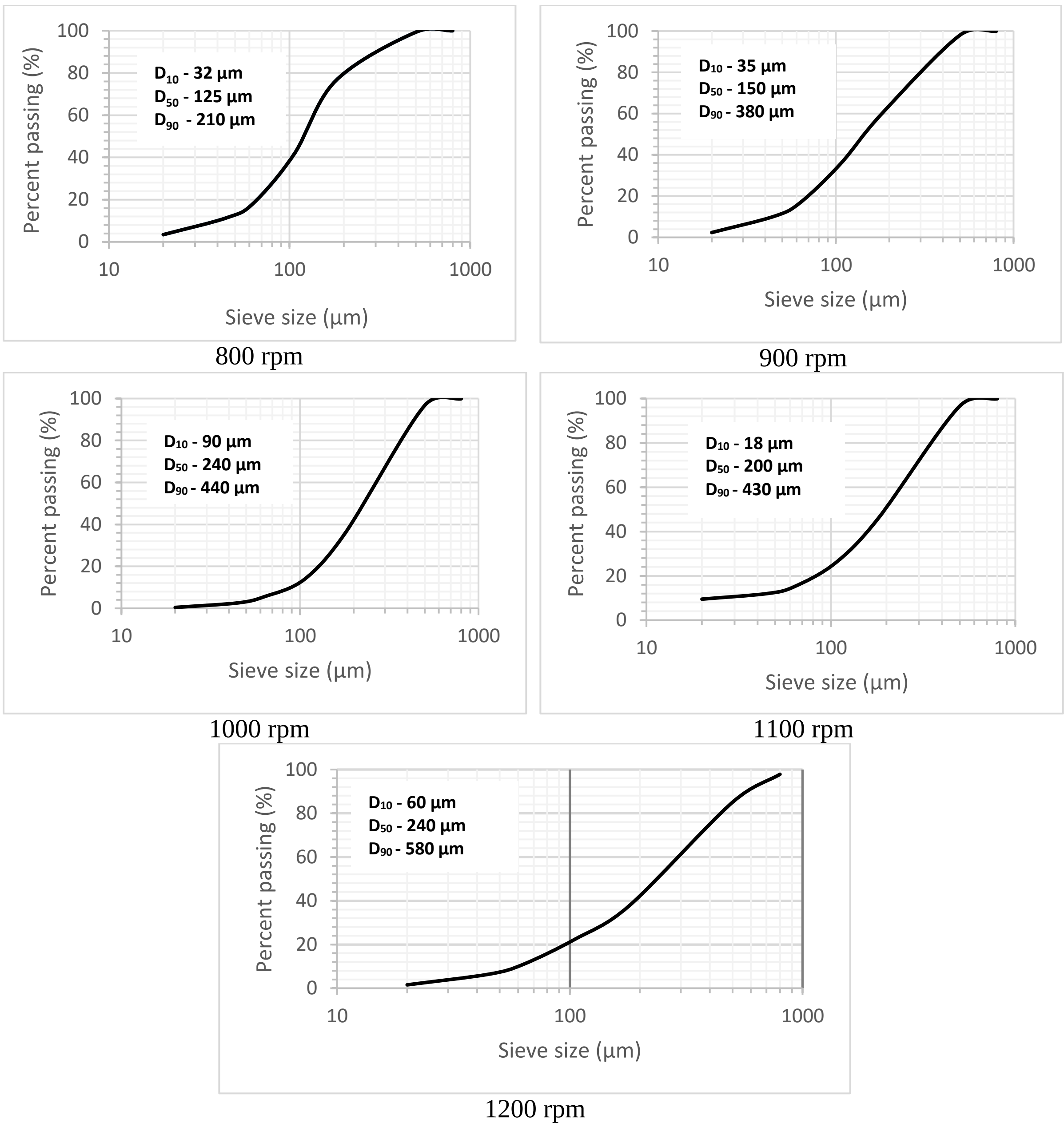

Figure 3: The particle size distribution of the milled powders at different milling speeds.

The morphology and EDS chemical element mapping of the powder milled at 800, 900, 1000, 1100 and 1200 rpm are given in Figure 4. The mechanical milling process produced flakes and irregular shaped granules due to continuous fracture, plastic deformation and cold welding of the powder particles.
The EDS chemical analysis of the powder milled at different speeds is given Table 1. The presence of Ti and Nb is revealed, while the iron (Fe) contamination in the milled powder is peculiarly prevailing at low milling speed.

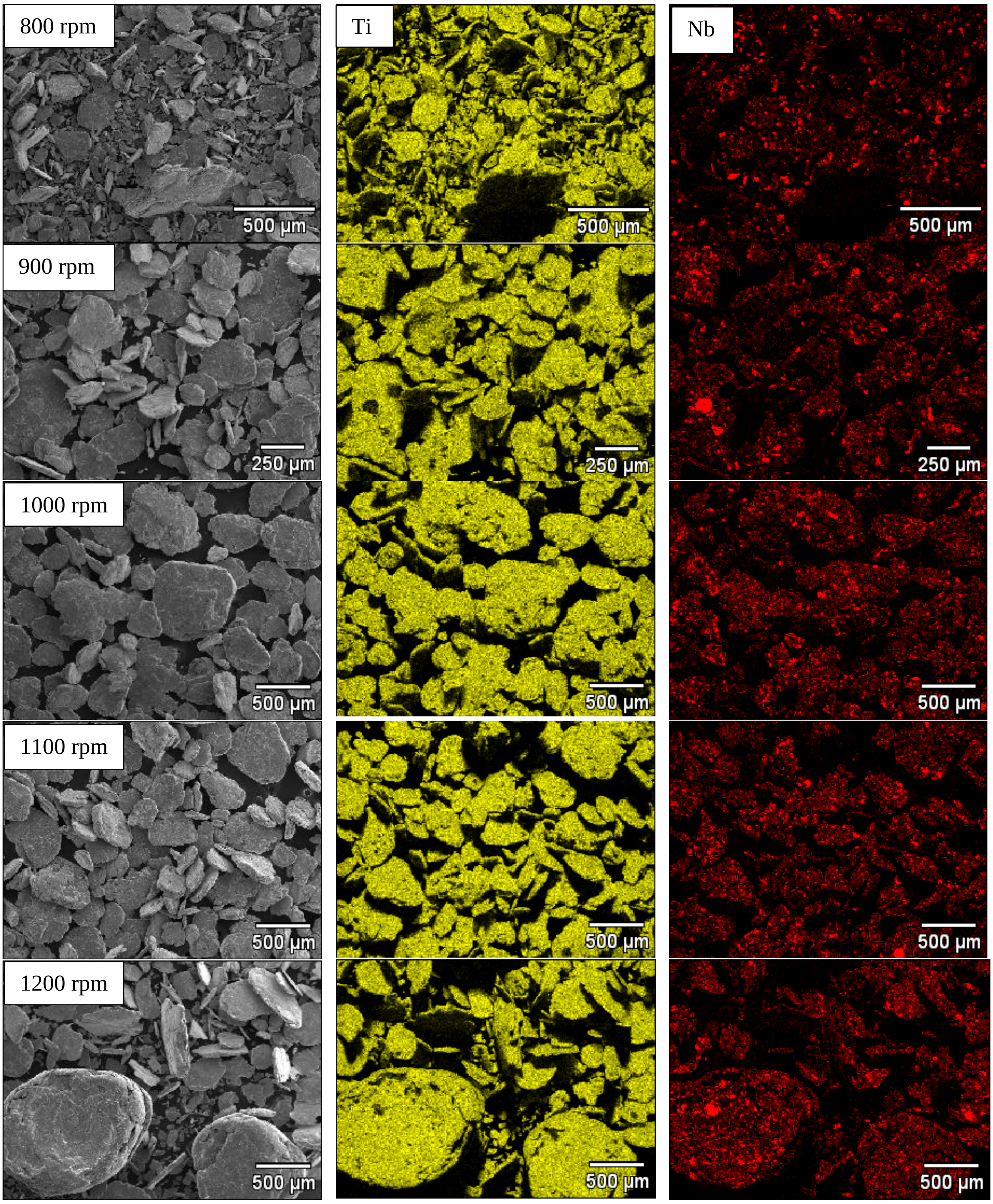

Figure 4. The morphology and EDX-mapping of the Ti-7Nb powder milled at powder milled at 800, 900, 1000, 1100 and 1200 rpm.

Table 1: The chemical compositions of the Ti-7Nb milled powder.

Milling speed (rpm)	Ti (wt. %)	Nb (wt. %)	Fe (wt. %)
800	91.706	7.656.	0.638
900	93.315	6.385	0.300
1000	92.773	6.960	0.267
1100	94.075	5.709	0.217
1200	94.175	5.570	0.251

The morphology of the mounted Ti-7Nb powder is given in Figure 5. The EDS analysis revealed the Ti-rich phase (light-grey region) and the Nb-rich phase (white region). The Ti and Nb were blended uniformly from 900 rpm milling speed, at 800 rpm the Ti and Nb rich phases are not uniformly distributed as shown in Figure 5 (800 rpm). The Nb-rich phase became predominant in the Ti-rich phase matrix as milling speed increased.

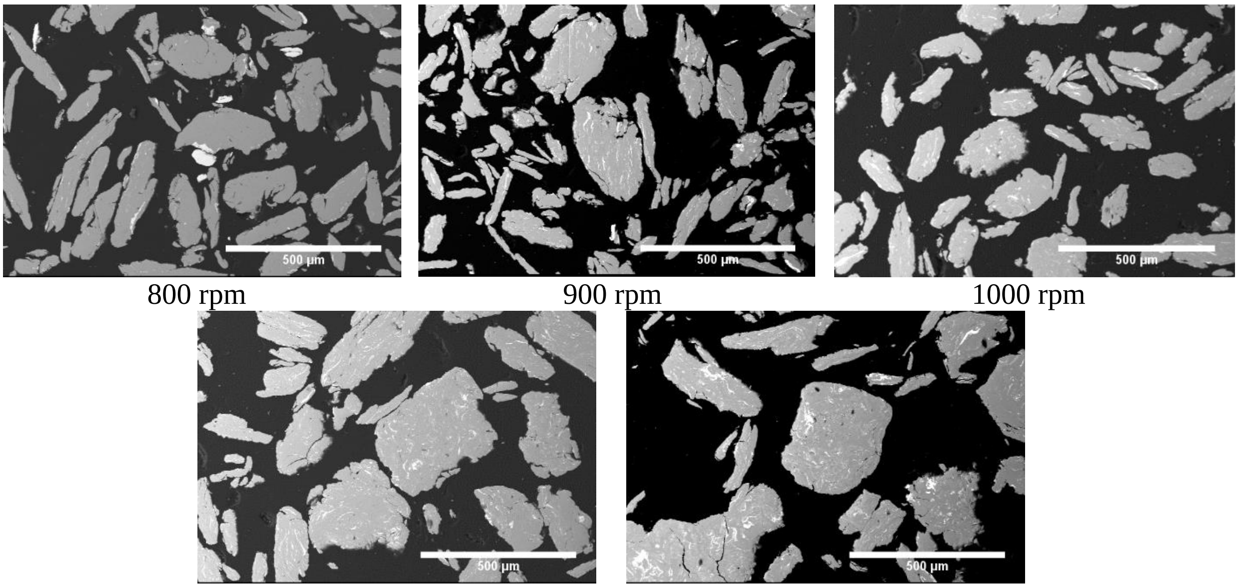

Figure 5: The morphology of the Ti-7Nb milled powder at 800, 900, 1000, 1100 and 1200 rpm.

The XRD patterns of the CP-Ti and Nb mixed elemental powder and Ti-7Nb milled powder at 800, 900, 1000, 1100 and 1200 rpm are shown in Figure 6. The hexagonal close packed crystal structure (hcp) or α-phase (Ti-rich phase, Figure 5), and face centred cubic structure (bcc) or β-phase (Nb-rich phase, Figure 5) which is stabilised by Nb element are revealed. The elemental Nb peaks (as mixed elemental powder) disappeared with increasing milling speed which suggests the occurrence of phase formation and consequently the alloying process during the milling of elemental powder of Nb and CP Ti. The peaks broaden as the milling speed increases which is due to the refinement of the crystallite size and the strains due to plastic deformation in powder particles [10].

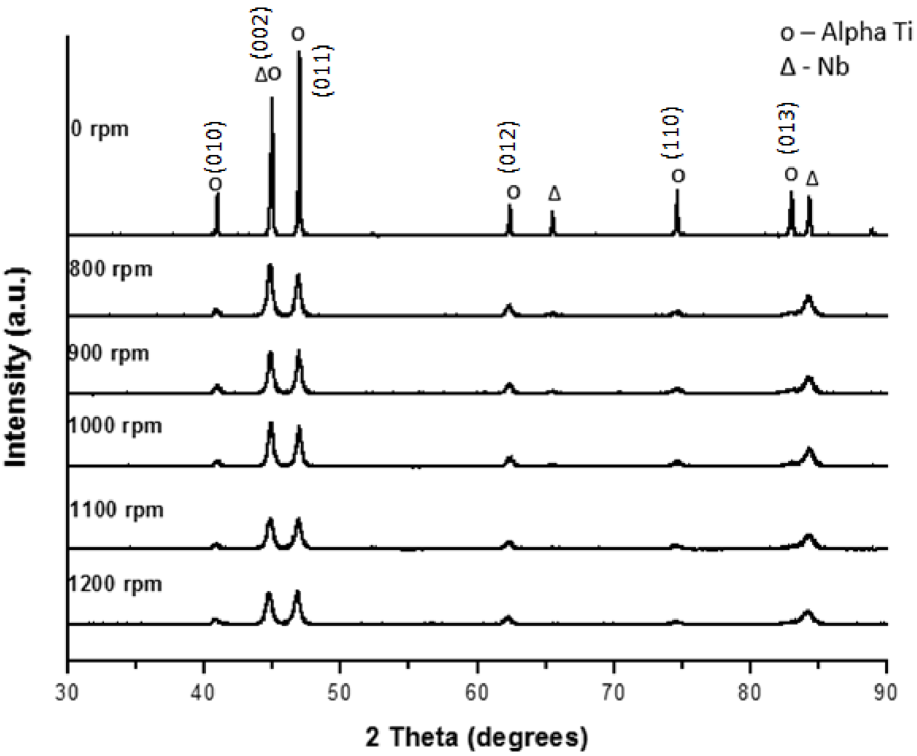

Figure 6: The XRD patterns of the CP Ti and Nb mixed elemental powder and Ti-7Nb milled powder at 800, 900, 1000, 1100 and 1200 rpm.

4. Conclusions

A mixture of CP-Ti and 7 % wt. Nb was mechanically milled using the high energy ball-mill Zoz-Simoloyer®, the following conclusions were drawn:

- The mechanical alloying process had good yield for the milled powder with little to no sticking of the powder to the grinding medium which means there is no need for a process control agent.
- The particle size increased with increasing milling speed due to fragmentation and cold welding during agglomeration of particles.
- The elemental powder of CP-Ti and Nb inhomogeneously alloyed in order to form single phase of alpha and beta phase in the milled powder.

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