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Evaluation of Surface Roughness and Biological Behavior of Ti-Nb Alloys

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ABSTRACT: Titanium - niobium alloys has perfect biocompatibility and high tendency to attach living tissue, making it preferable Ti- base alloys for orthopedic implants. This is Due to the combination of excellent mechanical properties high corrosion resistance. The work aims to evaluate the cell activity of Ti –Nb with different Nb percentage; (10 %wt., 20%wt. and 30%wt) produced by powder technology . also chemical surface treatment carried out by dual acid alkaline etching to create suitable surface roughness before deposition, while deposition process accomplished by pack cementation .The Characterizations of samples includes: microstructure observation by SEM , x-ray diffraction (XRD), MTT Assay (cell viability) and MTT assay (cell adhesion). From the SEM figures it is obvious that as niobium percentage increase there was large change in microstructure texture comparing with pure sample or other less Nb percentage refer that the niobium percentage (30%) was preferable to obtain fully and stable beta(β) phase in Titanium–Niobiom alloys. From XRD patterns the β phase would appear after Nb addition in different percentage and there was slightly increased in peak intensity with increasing niobium content. From MTT graphs it was found that the increasing in Nb content (30%wt.) Lead to high cell activity (viability) after 3 days of exposure in MG-63 cells and there was remarkable increase in cell viability and cell attachment compared with pure Ti- samples.

KEYWORDS: titanium – niobium alloys, dual acid alkaline etching, β- phase, MG-63 cells.

INTRODUCTION

Titanium is an personable metallic biomaterials due to high osseointegration strength, and also have desirable properties, such as relatively low modulus, strong fatigue strength, formability, machinability, corrosion resistance, and biocompatibility, are highly utilized in biomedical devices and implant, particularly like in uses of cardiac and cardiovascular and in replacements of hard tissue [1,2]. Hard tissues are most damaged because of aging, accidents, and other causes. In the response of the biological environment to artificial medical devices, the surface of the material plays an extremely important role [3,4]. The normal production steps typically result in an oxidized, polluted surface layer in medical implants made of Ti that is most stressed and deformed of plastically, non-homogenously and very badly defined [5,6]. For biomedical uses, certain "native" surfaces are obviously not suitable and some surface treatment must be done. The unique surface characteristics that are distinct during the bulk but most needed are more important purpose for making surfaces modifications to Ti device [7,8]. For the short and long-term performance of implants, geometry and surface topography are critical. In the last decade, implant surfaces were produced in a concerted effort to provide bone in a faster and improved method of osseointegration [9]. Pure Titanium has low strength and incomplete mechanical resistance. Thus, these inhibit the employing pure titanium alloys as implants which are classified as long-term applications. Then, Ti-alloys, consider to being more beneficial in mechanical behavior [10,11]. Ti–6Al–4V is the closely utilized as Titanium - base alloys, has been review for neurological cytotoxicity and high elastic modulus because of the release of aluminium and vanadium ions inside the body after implantation [12,13]. For this purpose, both mechanical characteristics and biological portion must be taken jointly in design and application of Titanium alloys. Titanium–Niobium alloys do not involved harm or toxic elements and exhibit a minimum Young’s modulus (E) the same as Young modulus of human bone, because of the supplement of Niobium stabilizer of beta phase structure(β), have large awareness. In vivo and in vitro biological estimate of pure metallic and alloying elements, involving tantalum, niobium and Zirconium, referring that they have less cytotoxic effect than Titanium alloys or even non-toxic [14,15]. Ti-Nb alloys can be used both for hard and soft tissues [16]. Mechanical characteristics of Ti-Nb depend on niobium percentage which is alternatively effect on amount of β phase presented in current alloy Therefore, minimum percentage is about 35–40 wt% of Niobium content must involve to obtain a fully and stable beta(β) phase structure in Titanium–Niobium alloys [17,18].

MATERIAL AND METHODS

The experimental includes the preparation of the four samples (Ti - pure), (Ti-10%Nb), (Ti-20 %Nb) and (Ti-30%Nb) by using powder metallurgy technique. Table (1), illustrated the properties of powders titanium and niobium powders.

Table 1: Properties of powders.

Element	Color	Shape	Mesh	Radius
Ti	silver	Spherical	200	70µ
Nb	Light gray	Spherical	200	30µ

After choosing the weighted percentages for four specimens, the metals powder was weighted by utilizing the sensitive balance where the total weight of all specimens is 5gm. Then powders are mixed in a boll mill for 30 min at 80 rpm speed for each sample, while Compaction process of the powder of each sample is done by using the hydraulic press machine in which powders were pressed under pressure of 10 tan for 15 min and then samples with same diameter of 15 mm and 5mm height for each type of alloys was produced. Sintering process was done by utilizing CVD quartz tube furnace at temperature around 1100 °C for 5 hr under vacuum (argon gas) to prevent samples oxidation. Result samples with diameter 15 mm. The surface treatment involved clean the surface from all dirt and impurities before chemical treatment to get a suitable clean surface for one hour then they were removed and dried. After that the samples were ready for the Acid and alkaline treatment, which they utilizing to remove contamination and oxide in order to have clean and good surface finishes. The samples were immersed in HCl acid with concentration 0.5 mM for four hours at 40ºC, and then the samples were immersed again in NaOH with concentration 10 mM for 24 hours at 60ºC. The second step is the Characterization of Samples a variety of tests were conducted in order to get various properties of samples. The tests were conducted (Scanning electron microscope (SEM), Surface roughness investigation by atomic force microscopy AFM, X-ray Diffraction (XRD), MTT Assay (cell viability) and MTT assay (cell adhesion).

RESULTS AND DISSOCIATION

Scanning electron microscope (SEM)

An electron microscope was conducted for scanning selected implant samples observations were conducted for four implant samples. These were used to make suitable observations of the surface topography and identifying the effect of both niobium addition and chemical surface treatment on the surface textures. Figure (1) showed for surface texture of pure titanium alloy after the completion of the sintering process, where the surface exhibit a high degree of roughness as a result of the manufacturing process and chemical treatment (acid – alkaline etching) which results in increasing the surface efficiency. in addition to that the surface porosity of sample alloys was high due to the size differences gap between the raw powders The same thing can be seen in other samples that includes the additions of niobium , figure (2) ,(3),(4) which shows the microstructure of the(Ti-10%Nb,Ti-20%Nb,Ti-30%Nb) respectively, from figures there was considerable changes in surface morphologies due to both the previous reasons and the appearance of beta phase in such samples which attributes to the effect of niobium Finally from SEM figures it is obvious that as Nb percentage increase there was large change in microstructure texture comparing with pure sample or other less Nb percentage as showed in figure (4) refer that the niobium percentage (30%) was preferable to obtain fully and stable beta (β) phase stracture in Ti–Nb alloys.

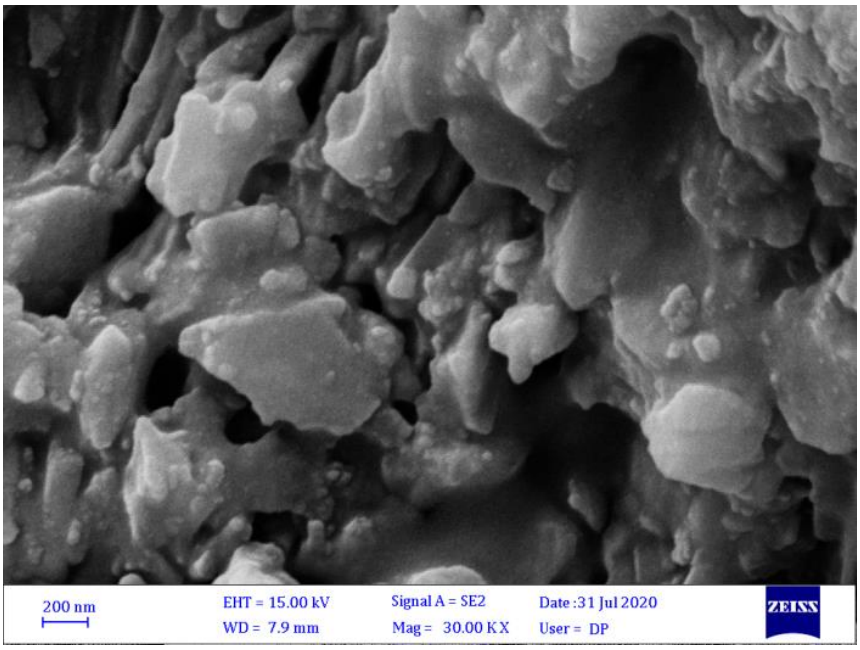


Figure 1: SEM image for pure Ti sample.

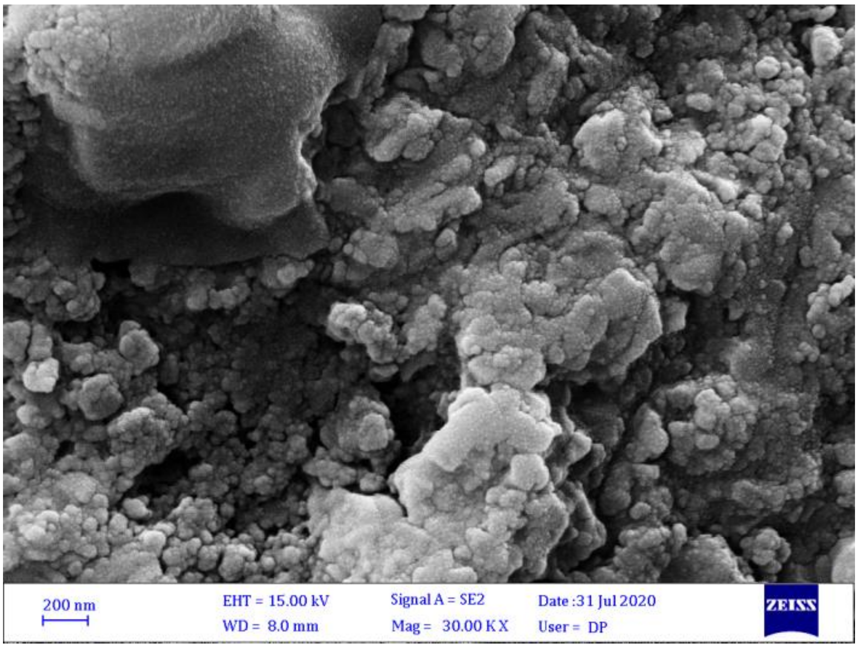


Figure 2: SEM image for Ti-10%wt. Nb sample.

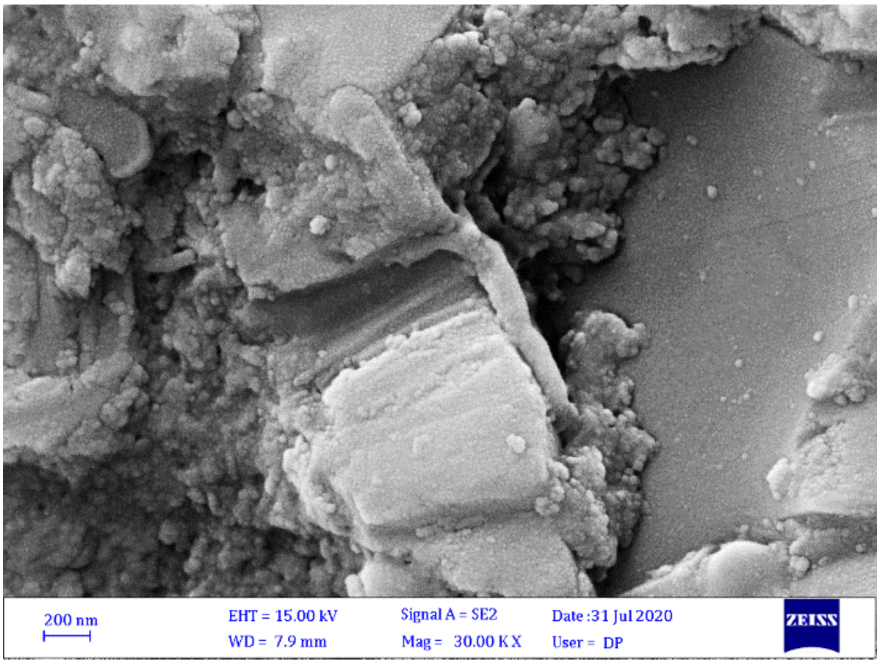


Figure 3: SEM image for Ti-20%wt.Nb sample.

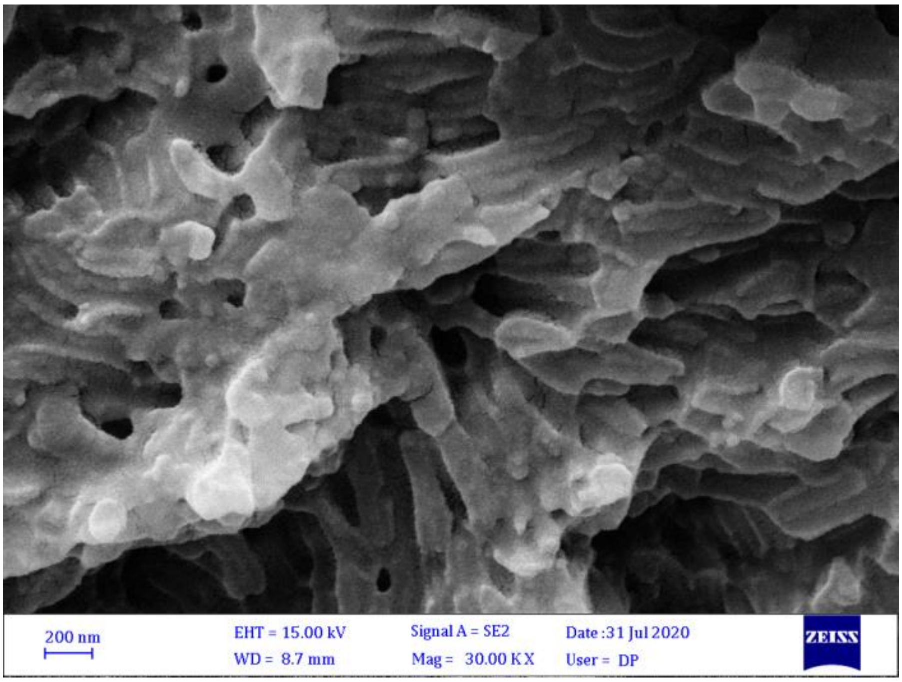


Figure 4: SEM image for Ti-30%wt. Nb sample.

Surface Roughness Investigation

This test was done in order to identify the topographic change for samples as well as the amount of roughness that have been produced with and without niobium additions. The topography of surfaces was showed by Atomic-force microscopy (AFM). Figure (5) displays the value of roughness of the master sample (pure Ti) and samples with different niobium additions. From figure it can be observed that pure titanium sample have high surface roughness compared with other samples. Also, it can be notice that the increasing in niobium addition lead to decrease in result surface roughness due to the effect of the appearance of β phase which considered as harder and stronger than α phase. it can be concluded that as increasing in niobium content up to 30% aid to form high amount of β phase in alloy that make the surface more hard and have less roughness

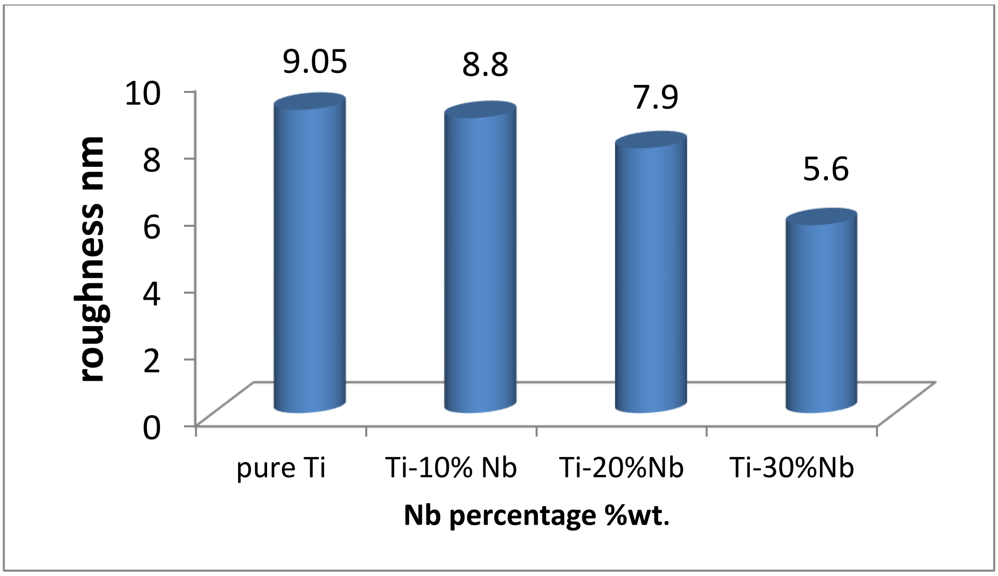


Figure 5: Surface roughness of samples

X-ray diffraction (XRD)

The XRD test was conducted by utilizing, Shimadzu X-ray diffract meter (type xrd- 6000) operated at 40 kV and 30 mA with Cu K α 1 radiation. In order to find out the structure and identify the phases of each sample, X-ray diffraction tests were done for with and without niobium additions. Figure (6) show the XRD pattern of pure titanium sample without niobium addition which refers to an alloy with single α phase. Also there are no presents of pure metals that indicate to the suitable time and temperature of sintering utilized in this work which results in full and complete sintering reactions. Figures (7, 8, and9) illustrated the X-ray diffraction patterns of samples with different Niobium percentage (10%,20%,30%) respectively form figures it can be seen the appearance of β phase in addition to α phase due to the addition of niobium which consider as beta stabilizer elements. the β peak positions in the X-ray diffraction patterns are same for all three samples. However, the β phase was slightly increased with increasing niobium content. Due to the varying in total amount of niobium in such alloy, and the solubility of niobium in the α-Ti based solid solution rose when the niobium content approached to 30%.

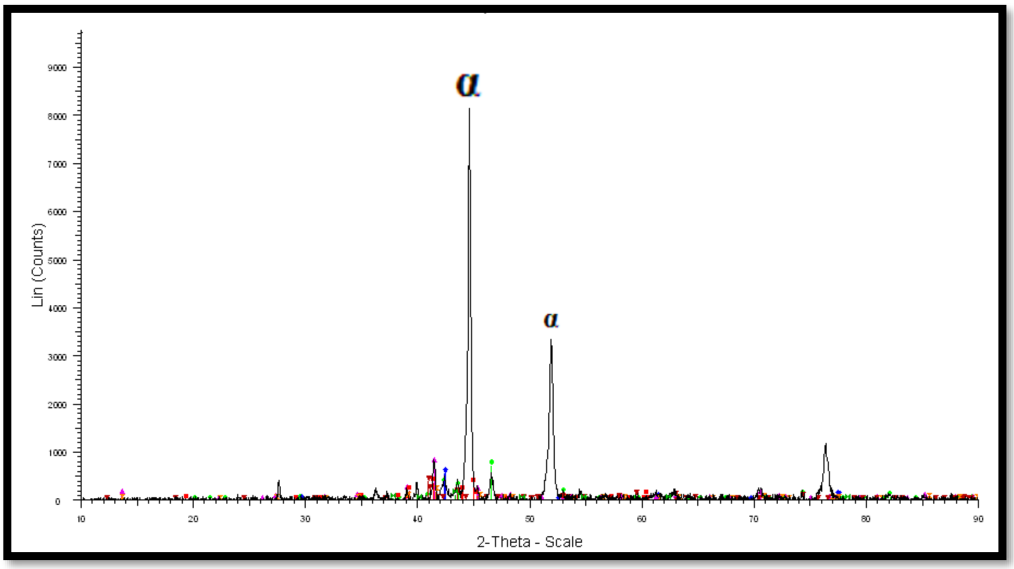


Figure 6: XRD pattern of pure Ti sample

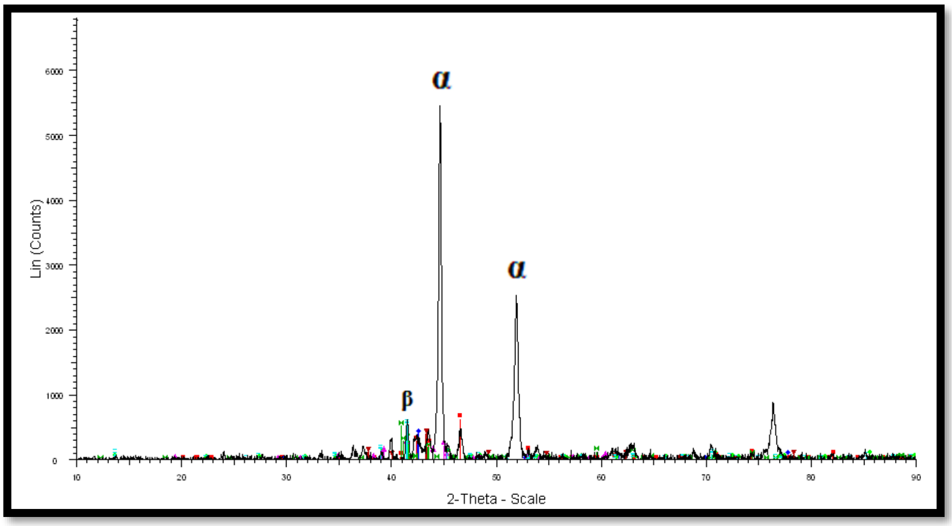


Figure 7: XRD pattern of Ti-10% Nb sample

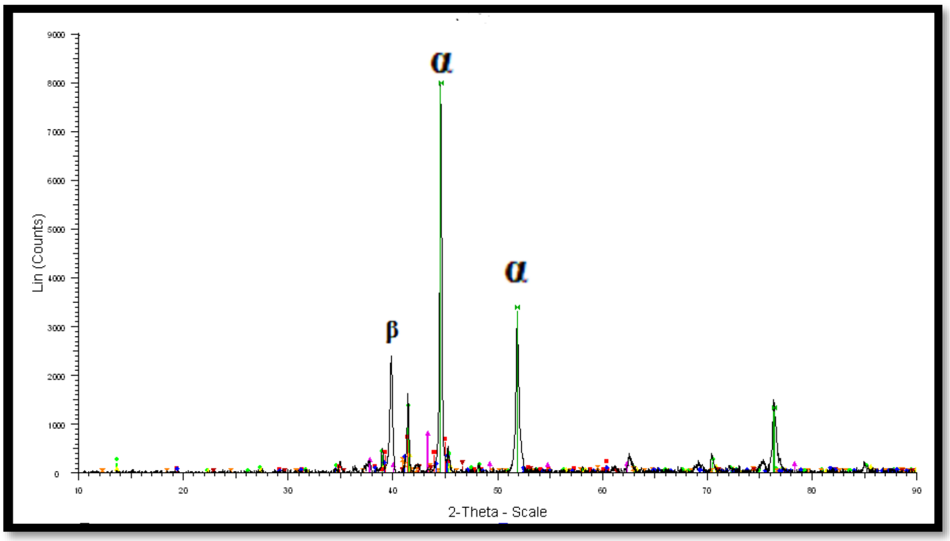


Figure 8: XRD pattern of Ti-20% Nb sample

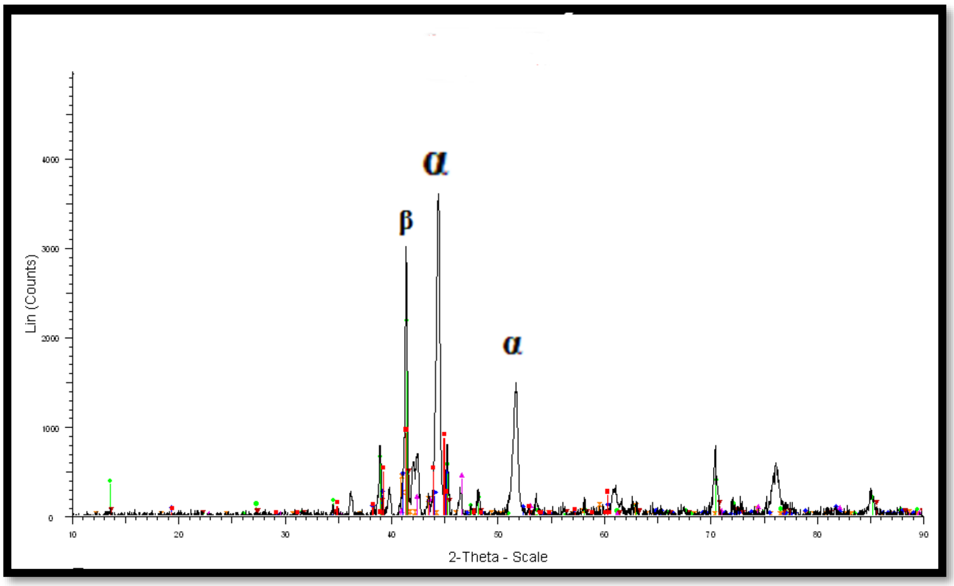


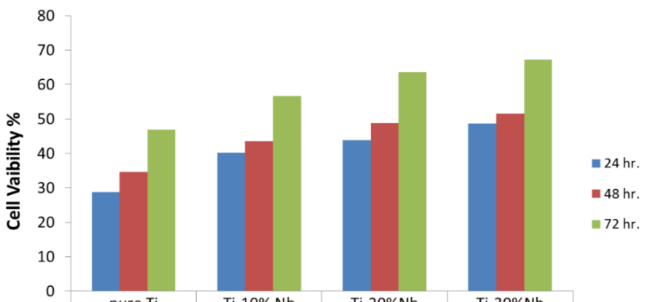
Figure 9: XRD pattern of Ti-30% Nb sample

Vitro Test (MTT Assay with MG-63 Cells)

MG-63 cell lines were cultured in T-75 flask in RPMI-1640 medium containing 10% bovine blood serum (FBS and 1% penicillin streptomycin antibiotic). After cell culture in the flask and 80% of the flask floor was filled by cells, the cells were removed by the trypsin EDTA-0.25% enzyme. Cell suspension was centrifuged at 1200 rpm for 5 minutes. After centrifugation, the supernatants of the cells were discarded and the sedimented cells were suspended in the new culture medium. Cell counting was done by trypan blue staining on the neobar under the microscope. The surfaces were sterilized by immersion in 70% ethanol for 2 h. To perform cytotoxicity assay, 5000 MG-63 cells per square centimeter were cultured on the surfaces. Cells were cultured on the surfaces for 14 days to evaluate the levels of cytotoxicity and cell growth on the surface. The cultured cell sample of the plate was considered as the control sample. After 3, 7, 10 and 14 days, the supernatants of the cells were removed and MTT solution (0.5mg / ml) was added to the samples and incubated in dark medium at 37 ° C for 5.3 hours. After completion of the mentioned time, the MTT solution was removed from the plate and the resulting purple dye dissolved in DMSO. The purple absorbance at 570 nm was read by ELISA reader. MG-63 cells were cultured on the surfaces for 2 days according to the above protocol to evaluate cell adhesion. After 2 days, samples were fixed with 4% glutaraldehyde for 1 hour. After 1 hour, the glutaraldehyde solution was extracted and the samples were dehydrated with ethanol, 90%, 80%, 70%, 60% for 15 min and prepared for electron microscopy.

Cytotoxicity and Cell Viability

The three-dimensional Cell growth of human MG63 fibroblast cultures was observe by mitochondrial dehydrogenase activity (MTT-assay) in (3, 7, 14) days exposure periods. The results show that the niobium addition expose large effect on cell viability, as have be seen in the MTT graphs, figure (10) show the cell viability of samples with and without niobium additions from figure, pure titanium alloys showed non cytotoxic effects due to an increasing in cell viability with time of exposure, which permit the inquiry of positive cell reaction with the titanium surface. Also Cell viability showing no sort of aggression result from titanium or niobium materials, The benefit of niobium additions compared with commercial pure titanium alloys, is the presence of active and non- cytotoxic beta phase as showed in figure, On the other hand, use of powder technology as manufactured process showed an increase in the surface roughness which presents several attractive features, make the surface more attractive to the bone. From MTT graphs it was observed that the titanium and titanium- niobium alloys surface became more active after 7 days of exposure as monitored in cell viability columns. In Figure (10) at 20% and 30% niobium showed remarkable increase in cell viability compared with pure titanium because of the increasing in a bio active beta phase at the alloy surface that allows Cell growth in the three dimensional very fast in the same period of exposure.



	pure Ti	Ti-10% Nb	Ti-20%Nb	Ti-30%Nb
24 hr.	28.7	40.3	43.8	48.7
48 hr.	34.6	43.6	48.9	51.6
72 hr.	46.8	56.7	63.6	67.3

Figure 10: MTT assay (cell viability) of samples.

CONCLUSION

It has been shown that all tested samples result in good positive response to MG63 osteoblast-like cell activity on their surface. The Biocompatibility MTT assay with MG-63 Cells demonstrate that cell viability showing no sort of aggression result from titanium or niobium materials, the benefit of niobium additions, when compared to commercial pure titanium alloys, is the presence of active and non- cytotoxic beta phase which presents several attractive features, make the surface more attractive to the bone.

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