Mechanical Properties of Nano-Structure Controlled Ti-Hf Alloy for Dental

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ABSTRACT

Ti-Hf alloy system forms an α-β isomorphous system and does not form any intermetallic compounds, which is also beneficial for good mechanical properties. The aim of this study was the improvement of mechanical property and biocompatibility by Hf addition to Ti-alloy. Ti-Hf(10, 20, 30 and 40wt%) alloys manufactured by non consumable vacuum arc melting furnace. All the specimens were nano-structure controlled by heat treatment at 1000 °C for 24hr in Ar atmosphere followed by furnace cooling and quenching into 0°C water. Phase constitutions, microstructure, and mechanical properties of the samples were characterized by XRD, OM, SEM, EDX. micro-hardness test and tensile test. Lamellar structure was transformed to needle-like structure with increased Hf contents. The α-phase peaks were detected for all samples. Hardness and tensile strength of Ti-Hf alloy increased as Hf content increased in the case of both water quenched and furnace cooled sample. Especially, the hardness and tensile strength of water quenched Ti-40Hf alloy was higher than that of others due to formation of needle-like structure.

Keywords: Nano-structure, mechanical property, Ti-Hf alloy, dental implant

1 INTRODUCTION

Cp-Ti have been regarded as suitable structural biomaterials and are currently widely used for replacing failed hard tissues[1-3], such as artificial hip, shoulder and knee joints, dental implants, etc., due to their excellent specific strength and biocompatibility[4-6]. Although the Ti-6Al-4V alloy in an acceptable prosthetic biomaterial, recent studies indicated that the release and accumulation of Al and V ions could have harmful effects on the human body[7-9].

Since Hf belongs to the same group as Ti in the periodic table of elements, it likely possesses excellent biocompatibility. The Ti-Hf alloy system forms an α -β isomorphous system and does not form any intermetallic compounds[10], which is also beneficial for good mechanical properties and corrosion resistance. The aim of this study was the improvement of mechanical property and biocompatibility by Hf addition to Ti-alloy. In the present study, Ti-Hf (10, 20, 30 and 40 wt%) prepared by non consumable vacuum arc melting furnace. All the specimens were nano-structure controlled by heat treatment. And then, the mechanical properties of nano-structure controlled Ti-Hf alloy for dental have been investigated by using XRD, OM, SEM, EDX, micro-hardness and tensile tester.

2 EXPERIMENTAL

2.1 Alloy preparation

Ti- xHf binary alloys, with Hf contents ranging from 10 to 40 wt.% (in 10 wt.% increments) were prepared using Cp-Ti(G&S TITANIUM, Grade. 4, USA) and hafnium(Kurt J. Lesker Company, 99.95% wt.% in purity). Ti-xHf alloys prepared using the vacuum arc melting furnace. The weighed charge materials were prepared by the vacuum arc furnace(vacuum arc melting system. SVT, KOREA), flowing the purified Ar gas into water cooling copper hearth chamber in vacuum atmosphere of 10^{-3}torr, and controlled atmosphere in chamber by method to keep vacuum again. Also, before melting, the constituents were cleaned with methanol to minimize oxygen quantity and surface contaminants in chamber and pure Ti was melted six times with purified argon gas. After that, samples were remelted at least six times by reversing the alloy sponges in order to avoid inhomogeneity, and then, were homogenized for 24hr at 1000°C(Model MSTF-1650. MS Eng, KOREA) in Ar atmosphere followed by furnace cooling and quenching in 0°C ice water.

2.2 Microstructure analysis

The specimens were cut from ingots to 2mm thickness with the speed of 3000 rpm by high speed diamond cutting machine (Accustom-5, STRUERS, Denmark). The specimen for observing microstructure were sanded on SiC paper (grade from 240 to 2000), finished by polishing with 0.3 ㎛ aluminum oxide powder and etched with 2 ㎖ Keller's solution (2 ㎖ HF+ 3 ㎖ HCl+ 5 ㎖ HNO_3+ 190 ㎖ H_2O). The solidification microstructures were observed by scanning electrode microscope(SEM: HITACHI S-3000)

and optical microscope(OM: OLYMPUS BM60M). The X-ray diffraction(XRD) patterns of all specimens were carried out on a diffractometer(D/max-rB) using CuKα radiation from 30° - 90°. Phases were identificated by matching their characteristics peaks with those in the files of the Joint Committee on Powder Diffraction Standard (JCPDS).

2.3 Vickers hardness test

The samples for vickers micro-hardness test were polishied by 0.3 ㎛ aluminum oxide powder from the sample surface. The measurements were measured using a micro-hardness tester(AFFRI, Italy) with a load of 0.3kg and for 10sec. The vickers micro-hardness were obtained by average value after testing of ten times. Tensile test was carried out using universal tensile tester.

3 RESULTS AND DISCUSSION

3.1 Microstructural observations

Figure 1 shows the optical microstructures of Ti-xHf alloys with different Hf contents(10, 20, 30 and 40 wt.%). It controlled by heat treatment at 1000°C for 24hr in Ar atmosphere followed by furnace cooling. It exhibits martensite structure, and lamellar structure was transformed to needle-like structure with increased Ti-Hf contents, and exhibits fine grain boundary by increase of Hf contents[10].

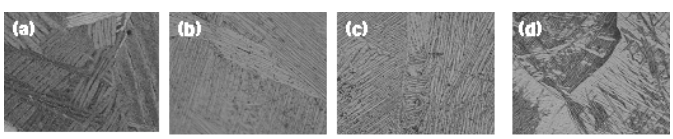

Figure 1: OM micrographs showing the microstructure of heat-treated (F.C) Ti-xHf alloys. (x100)
(a) Ti-10Hf (b) Ti-20Hf (c) Ti-30Hf (d) Ti-40Hf

Figure 2 shows the optical microstructures of Ti- (10, 20, 30 and 40 wt.%)Hf alloys by 0°C water quenched from 1000°C. Nanostructured needle-like traces of martensite were observed at all specimens, and nanostructured amount of traces in quenched alloy was more than that of furnace cooled alloys. The fine needle-like structure was observed in the image of Ti-40Hf alloy. It is confirmed that Hf elements acts to form a nanostructured martensite phase and grain growth was effectively suppressed[2].

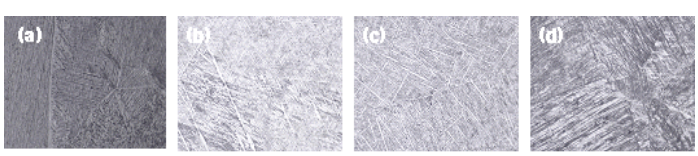

Figure 2: OM micrographs showing the microstructure of heat-treated (W.Q) Ti-xHf alloys. (x100)
(a) Ti-10Hf (b) Ti-20Hf (c) Ti-30Hf (d) Ti-40Hf

The microstructures of the Ti-(10, 20, 30 and 40 wt.%)Hf alloys by heat treatment at 1000 °C for 24hr in Ar atmosphere followed by furnace cooling are shown in the SEM image in figure 3. It exhibits martensite structure, and Ti-10Hf alloy was lamella structure, and Ti-30Hf was complexed that consist of lamella and needle-like structure because Hf can be formed long-range atomic arrangement for furnace cooling[2]. The case of Ti-40Hf was fine needle-like structure.

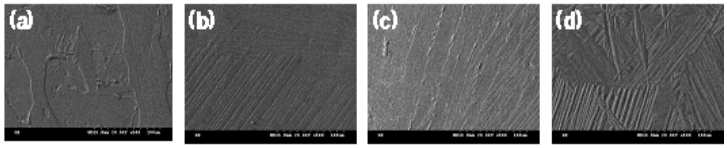

Figure 3: SEM micrographs showing the microstructure of heat-treated (F.C) Ti-xHf alloys.
(a) Ti-10Hf (b) Ti-20Hf (c) Ti-30Hf (d) Ti-40Hf

The addition of various amounts of Ti-xHf alloys cause change in the microstructure, that is, lamellar structure was transformed to needle-like structure with increased Hf contents. Especially, the microstructure of water quenched Ti-xHf specimens more fine than furnace cooled specimen in the image of Figure 4.

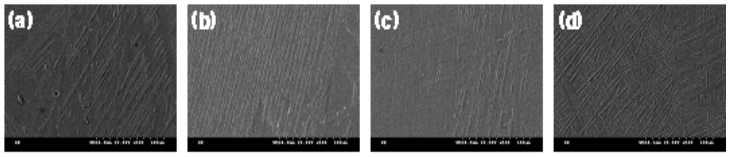

Figure 4: SEM micrographs showing the microstructure of heat-treated (W.Q) Ti-xHf alloys.
(a) Ti-10Hf (b) Ti-20Hf (c) Ti-30Hf (d) Ti-40Hf

Figure 5 shows the X-ray diffraction profiles of furnace cooled(a) and water quenched(b) Ti-(10, 20, 30 and 40 wt.%)Hf alloys. The peaks were indexed using the JCPDS diffraction data of Ti-xHf alloys. It can be seen that only reflections from α-phase were identified by XRD, α-phase in Ti-(10, 20, 30 and 40wt.%)Hf alloys proceed through a phase reaction leading to the formation of h.c.p phase[11].

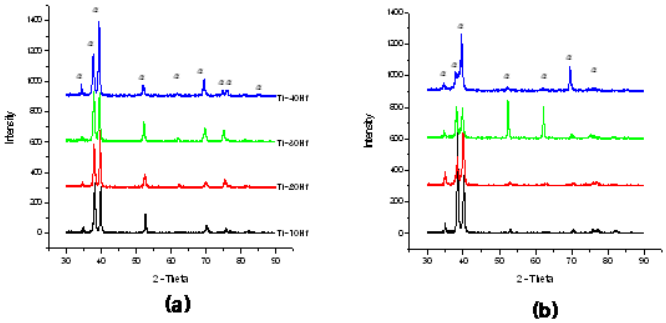

Figure 5: XRD diffraction patterns of Ti-xHf alloys
(a) heat - treated (F.C) , (b) heat - treated (W.Q)

It is confirmed that β phase does not appear in Ti-Hf phase diagram as shown in Figure 6, since α+β phase region is very narrow. The heat treated Ti-(10, 20, 30 and 40 wt.%)Hf alloys from 1000 °C have α–phase structure at room temperature[11, 12].

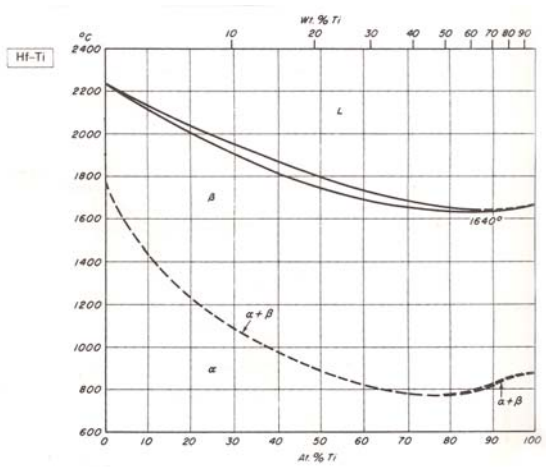

Figure 6: The Ti-Hf phase diagram

The EDX line profile results of Ti-(10, 20, 30 and 40 wt.%)Hf alloys are shown in Figure 7 (a) and (b), respectively. Line of Ti content is red colar and line of Hf content is blue colar. Hf element was entirely distributed in matrix due to homogenization treatment, and Hf peaks showed mainly at needle-like structure. This is that Hf elements acts to form a nanostructured martensite phase and grain growth was effectively suppressed[2].

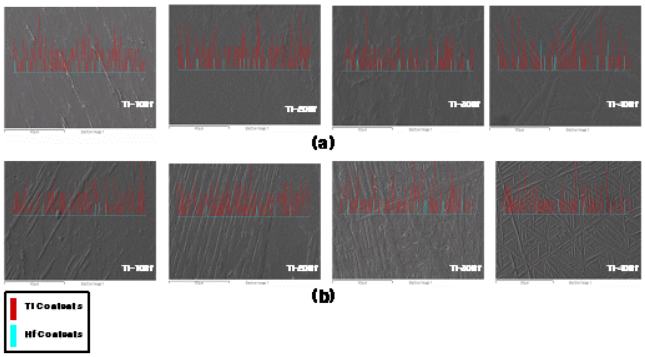

Figure 7: EDX results of heated(F, C) Ti-xHf alloys(a), and heated(W, Q) Ti-xHf alloys(b)

The hardness test results of Ti-(10, 20, 30 and 40 wt.%)Hf alloys are shown in Figure 8. For hardness and tensile strength value of water quenched sample had higher than those of furnace cooled sample. The hardness and tensile strength increased as Hf content increased. Especially, the hardness and tensile strength of water quenched Ti-40Hf alloy were higher than that of other alloys due to formation of nanostructured needle-like structure[13].

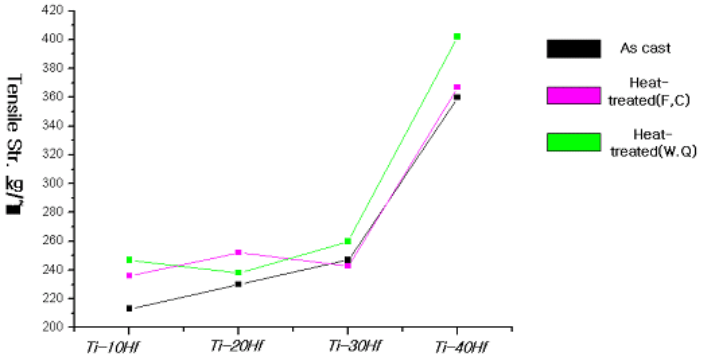

Figure 8: Tensile strength of Ti-xHf alloys with Hf content .

4 CONCLUSIONS

1. Microstructure clearly observed that lamellar structure translated to needle-like structure with Hf contents. Especially, microstructure of water quenched Ti-Hf specimens showed finer than that of furnace cooled specimen.
2. As a result of XRD, α-phase peak was detected for all samples.
3. From the results of mechanical test, the hardness and tensile strength of Ti-40Hf alloy were higher than those of others in the case of both water quenched and furnace cooled sample. Hardness and tensile strength of water quenched sample had higher than those of furnace cooling sample.
4. From the results of EDX, Hf peak appeared mainly at needle-like structure.

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