Mechanical properties and microstructures of cast dental Ti-Fe alloys

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Binary Ti-Fe alloys of varying concentrations of Fe between 5–25% were made, and their castings evaluated in terms of microstructures and mechanical properties. The aim of this study was to explore the composition of Ti-Fe alloys that offers improved wear resistance of titanium. X-ray diffraction and microstructural observation revealed that 5–7% Fe, 8–15% Fe, and 20–25% Fe consisted of α+β, single β, and β+Ti-Fe phases, respectively. The hardness of alloys with 8–13% Fe was almost equal to that of Co-Cr alloys but lower than of the other Ti-Fe alloys. Elongation of the Ti-Fe alloys was negligible. However, dimples were observed in specimen containing 7–11% Fe. Alloys with 9% Fe demonstrated the highest strength of more than 850 MPa. We believe that Ti-Fe alloys with 8–11% Fe may be applicable in development of an alloy with good wear resistance due to the exhibited properties of high hardness and ductility albeit low.

Keywords: Ti-Fe alloy, Mechanical property, X-ray diffraction, Microstructure, Titanium alloy

INTRODUCTION

Titanium has excellent biocompatibility, good corrosion resistance, and high specific strength. In the medical field, titanium is applied as a substitute material for hard tissues as well as fixation devices implanted in the body for a long duration. In the dental field, some of the applications of pure titanium, Ti-6Al-4V alloy and Ti-6Al-7Nb alloys include: making of dental implants, denture metal frames, and clasps.

One of the disadvantages of titanium is poor wear resistance mainly due to low hardness. Moreover, its high chemical activity and low thermal conductivity worsens wear resistance causing it to adhere to other materials because of frictional heat. Consequently, when applied in joint replacement, ordinary functional forces lead to abrasion wear of the artificial head. Similarly, repeated occlusal forces cause an increase in the gap between dental implant and the abutment. Cases of loose abutment screws and metal allergy due to titanium alloy abrasion powder have also been reported.

Attempts to improve wear resistance of titanium through several surface treatment methods have been reported. Hard coatings formed through surface treatment such as chemical and physical vapor deposition or thermal spraying methods are hard but thin. The treated surfaces showed more than 2,000 Vickers hardness (Hv), and a decreased wear amount of hard coatings was approximately 1/200 that of Ti-6Al-4V alloy when subjected to a sliding test. However, the hard coatings are generally brittle, thus treated surfaces tend to have a distinct interphase between the layer of coating and base material. On the other hand, the hardness of a thermal diffusion treated titanium surface, which forms solid-solution layer with oxygen, nitrogen or carbon was reported to be 1,200 Hv. There is no distinct or separate solid-solution layer/base material interface for thermal diffusion treated surfaces. However, the solid-solution layers formed are brittle and the thickness of the layers formed is uncontrollable.

Although hardness is the main contributing factor of wear resistance, other determinants include: strength, elongation and toughness. If two metals are of equal hardness, the metal with higher elongation or higher toughness has better wear resistance. Although all the above treatment methods improve wear resistance by forming a super hard surface on titanium or its alloys, bulk metal (untreated metal) with appropriate hardness and elongation shows excellent wear resistance. For example, dental Co-Cr casting alloys (350–390 Hv) of lower hardness than surface treated titanium but with good elongation shows excellent wear resistance. Therefore, a metal can have excellent wear resistance without undergoing surface treatment as long as it has both appropriate hardness and elongation. Since titanium shows more than 30% higher elongation and lower hardness (138 Hv) compared to Co-Cr alloys, its hardness and wear resistance can be improved through the process of alloying.

Titanium has two alloy phases and undergoes allotropic transformation at 882°C. α-phase is a hexagonal close packed structure (hcp) that is stable at a low temperature whereas β-phase is a body-centered cubic lattice structure (bcc) stable at high temperature. The allotropic transformation temperature can be lowered by alloying titanium with a β stabilizing element. Au, Ag, Cu, Co, Cr, Fe, Mn and Pd are eutectoid β stabilizing elements. Metastable β-phase can be gained through rapid cooling of some types of titanium alloys within the β-phase region. Metastable β-titanium alloys have high strength, high hardness and good elongation since the
metastable β-phase shows characteristics similar to the stable β-phase (bcc). Okuno et al. reported that iron as a β stabilizing element; increases the hardness quality of titanium remarkably in comparison with the others \(^\text{21}\). Furthermore, iron is an essential element in the body of human beings and has relatively low risk of inducing allergic reactions \(^\text{23}\). Based on the above considerations, possibility of developing dental titanium alloys with excellent wear resistance by alloying Ti with Fe was explored.

Industrial studies focusing on the mechanical properties of some heat treated wrought Ti-Fe alloys exist \(^\text{24-27}\). However, little has been reported on composition, microstructures and mechanical properties of cast dental Ti-Fe alloys. Industrial Ti-Fe-O-N alloy series were developed \(^\text{28}\), even though they are not binary alloys. Their dental castings have been reported to show higher strength and hardness than titanium castings \(^\text{29}\). However, simple binary alloys like Ti-Fe are notably superior with advantages in respect to production, microstructure control and other applications compared to multicomponent alloys like Ti-Fe-O-N. It is possible to form an alloy with titanium surface as well as control the concentration of Fe in a binary system alloy. In this study, binary Ti-Fe alloys with 5–25 mass% Fe were prepared, and their castings evaluated in terms of microstructures formed and mechanical properties. Based on the obtained data, we deduced the composition range of Ti-Fe alloys likely to show improved wear resistance compared to titanium. Thereafter wear resistance tests were conducted on specimen whose composition was within the range thought to show improved wear resistance and compared to that of titanium.

**MATERIALS AND METHODS**

**Preparation of specimen**

The equilibrium phase diagram of Ti-Fe system \(^\text{30}\) is shown in Fig. 1. Ti-Fe alloys with the composition of 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 20, and 25% Fe were prepared. The desired amount of Ti sponge (>99.8%, grade S-90, Osaka Titanium technologies, Amagasaki, Japan) and pure Fe (>99.95%, Hirano Seizaemon Shoten, Tokyo, Japan) was melted in an argon-arc melting furnace (TAM-4S, Tachibana Riko, Sendai, Japan) to form a 30 g ingot of each alloy.

Each ingot of the various Ti-Fe alloys was cast to form test specimen using magnesia investment (Symbion-TC, i-Cast, Kyoto, Japan) in argon gas-pressure dental casting machine (Autocast HC-III, GC, Tokyo, Japan). Specimen for tensile tests were 3.0 mm and 15mm in diameter and gauge length respectively, in compliance with ISO 22674. Slabs of 10×10×1.0 mm were made and utilized as specimen for X-ray diffractometry, microstructural observation, hardness, and wear tests. All the cast slabs specimens were abraded to a depth of 300 μm using 180–1000 grit SiC paper to remove the hardened surface layer.

**X-ray diffractometry**

X-ray diffraction (XRD) was performed at room temperature using Cu Kα radiation generated at 30 kV and 10 mA in an X-ray diffractometer (D2 PHASER, Bruker AXS, Tokyo, Japan). Crystallography Open Database was used in phase identification.

**Microstructural observation**

The specimen surfaces were mirror-polished using diamond suspension of up to 1 μm particle size then etched with etching solution (HF:HNO₃:H₂O=1:4:95) for 30–70 s. The etched microstructures were observed under optical microscope (PMG3-614U, Olympus, Tokyo, Japan).

**Vickers hardness test**

Vickers hardness of the specimen was determined using micro Vickers hardness tester (HM-102, Mitutoyo, Kawasaki, Japan) with a 1.961 N load and 15 s dwell time (n=7).

**Tensile tests**

Tensile tests were carried out using universal testing machine (AG-IS, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm · min⁻¹ and room temperature (n=6). The ultimate tensile strength, yield strength (proof strength of 0.2% non-proportional extension), and elongation after fracture were determined. The fractured surfaces were observed under scanning electron microscopy (SEM; JSM-6060, JEOL, Tokyo, Japan).

**Wear test**

Based on the mechanical properties exhibited, Ti-10% Fe alloy which showed potential of improved wear resistance was selected as the specimen to be...
subjected to wear test. Two body sliding type of wear testing device (Model C320, Yoshimitsu Seiki, Tokyo, Japan) was used to perform the reciprocating wear test for both pure titanium and Ti-10% Fe alloy. A 3.0 mm diameter steel ball (SUJ2: 844 Hv), whose hardness was sufficiently higher than that of test specimen used in this study, was used as an indenter. The spherical indenter was fixed in a manner that ensures the load is vertically transmitted to the test specimen. The test conditions were: 4.9 N weight, 1.5 mm strokes, a rate of 60 cycles per minute and 20,000 cycles under water so as to mimic wet conditions. After the wear test, the depth of the wear marks produced were measured using a surface profilometer (Surfcom 480A, Tokyo Seimitsu, Tokyo, Japan) (n=9).

Statistical analysis
The data obtained was statistically analyzed using one-way ANOVA and Tukey HSD tests at a significance level of α=0.05.

RESULT

X-ray diffractometry
The XRD patterns obtained for pure titanium and Ti-Fe alloys are showed in Fig. 2. Only α-titanium peaks were observed in pure titanium. Both α-titanium and β-titanium peaks were observed in 5–7% Fe alloys. The intensities of the α-related peaks were high, while those of the β-related peaks were low. Only β-titanium peaks were observed in 8–20% Fe alloys. There were no α-titanium peaks observed for composition of 8% Fe and above. Peaks of β-titanium and TiFe were observed in the 25% Fe alloy.

Microstructural observation
Microstructures of pure titanium and Ti-Fe alloys are shown in Fig. 3. Pure titanium showed acicular structures which are characteristic of α-titanium. In the 5–7% Fe alloys, acicular structures were observed in equiaxed grains of β-titanium. In alloys with 8% Fe and more, the acicular structures disappeared, and...
only equiaxed structures were observed. The size of β grains in all the 8–25% Fe alloys was almost the same. Intermetallic compound, TiFe, was found along the grain boundaries and within the β grains of the 20 and 25% Fe alloys.

Vickers hardness
Vickers hardness results of Ti-Fe alloys is shown in Fig. 4. The hardness of Ti-Fe alloys was significantly higher ($p<0.01$) than that of pure titanium. Hardness of the alloys increased with increase in iron content up to 6% Fe then decreased across alloys containing 6–8% Fe. It remained almost the same ($p>0.05$) for alloys with 8–13% Fe. Once more, the hardness increased with increase in iron content for alloys containing more than 13% Fe.

**Tensile properties**
Tensile strength of the Ti-Fe alloys tested is shown in Fig. 5. The tensile strength of 5% Fe alloy (553 MPa) is significantly higher ($p<0.01$) than that of pure titanium. The strength decreased with increase in iron content for 5–6% Fe alloys then increased for alloys with 6–9% Fe. The highest tensile strength in this study (867 MPa) was of 9% Fe alloy. The strength decreased again in alloys with more than 9% Fe but the 20 and 25% Fe alloys were too brittle for tensile strength to be obtained.

Elongation of the Ti-Fe alloys was very small. Alloys with 8–11% Fe showed less than 1% while those of the other compositions were almost 0.

Yield strength of only alloys with 8–10% Fe was obtained. Alloys of other compositions were not examinable for yield strength due their low elongation. Yield strength values for 8, 9, and 10% Fe alloys were 631 (±66), 744 (±162), and 630 (±47) MPa, respectively. The yield strengths of alloys containing 8–10% Fe were significantly higher ($p<0.01$) than that of pure titanium 259 (±10) MPa.

**Fractography**
SEM images of the typical fractured surfaces of the Ti-Fe alloys subjected to the tensile tests are shown in Fig. 6. Dimples, which are typical of a ductile fracture, were visible on the specimen representing alloys with 7–11% Fe but absent in the alloys with more than 12% Fe. The fractured surfaces of specimen with 20 and 25% Fe showed a cleavage structure.
the eutectoid temperature, in addition to the TiFe precipitated by the eutectoid reaction. This is however dependent on the composition of the alloy and the cooling rate. Since alloys containing 20 and 25% Fe are located within the hyper-eutectoid region, the amount of TiFe precipitated in their microstructures is expected to be high. However, only traces of TiFe were observed in the microstructures of 20 and 25% Fe. The amount of TiFe precipitated from 20% Fe was minute and the TiFe peak could not be detected. The temperature at which precipitation of TiFe in β-phase and eutectoid reaction \( \beta \rightarrow \alpha + \text{TiFe} \) occurs is relatively low as shown in the equilibrium phase diagram of Ti-Fe system. The reaction hardly occurred during cooling of the alloy castings under conditions in this study. It has been reported that the intermetallic compound TiFe, does not significantly reduce corrosion resistance of titanium\(^{22}\). On the other hand, intermetallic compounds are known to be hard and brittle\(^{31}\). Therefore, we suggest that the amount of iron added to titanium should be less than 20% for application as a dental alloy.

**Mechanical properties**

Vickers hardness of the Ti-Fe alloys was significantly higher than that of pure titanium. Alloying with iron improved the hardness of titanium. There was only one peak of hardness between 5–7% Fe. Precipitation hardening of the dual-phase alloy with \((\alpha + \beta)\) structure, and the solid-solution hardening of the \(\alpha\) and/or \(\beta\)-phases, may have contributed to the high hardness of alloys containing 5–7% Fe. When the amount of iron reached 8%, the alloy phases changed from \(\alpha + \beta\)-phases to \(\beta\) and its hardness decreased. Beyond 8%; as the concentration of iron increased, the hardness also increased owing to solid-solution hardening of the \(\beta\)-phase. Additionally, precipitation of intermetallic compound TiFe at 20% Fe or more further increased the hardness of the alloys. The measured hardness value of Ti-Fe alloys was higher than that of hardened type 4 dental gold casting (237–264) and Ti-6Al-4V (320–341) alloys\(^{19,32}\). The hardness of alloys with 8–13% Fe was almost equal to that of Co-Cr alloys (350–390) whereas that of the other concentrations of Fe was even higher\(^{19}\).

The elongation of Ti-Fe alloys was very small. However, dimples, which are a feature of ductile fractures, were observed in the alloy specimen containing 7–11% Fe. β-titanium is known to have a large elongation since its bcc structure has more slip planes than hcp structure found in α-titanium. With the exception of alloys with 7% Fe all the other compositions that exhibited dimples had single \(\beta\)-phase. Elongation of Ti-Fe alloys with more than 12% Fe was very small in spite of having single \(\beta\)-phase. Iron in the solid-solution acts as an obstacle on slip planes. The obstruction is strong since the atomic radii of iron and titanium differ greatly. Therefore, the large amount of iron in solid-solution may have contributed remarkably to the reduced elongation of Ti-Fe alloys. Elongation of alloys containing 20 and 25% Fe was almost 0%. Their fractured surfaces showed the cleavage structure, which contained the brittle TiFe.

**DISCUSSION**

**Alloy phases**

Iron is one of the β stabilizing elements. Based on the equilibrium phase diagram of Ti-Fe system (Fig. 1)\(^{30}\), the presence of iron expands the width of the β region towards the low temperature direction. When rapid cooling of Ti-Fe alloys with an expanded β region is carried out from the β region, a transformation to metastable β-phase (mostly α). Observation of its microstructure revealed fine acicular structure of α-titanium and equiaxed grains of β-titanium. As the concentration of mass% Fe increased; alloys that contained 7–8% Fe changed phases from dual α+β to single β. Alloys with 8% Fe and more did not show the acicular structures of α but typical equiaxed structures of β were evident. Our findings concur with a previous study by Takada et al. which reported that dental casting Ti-Fe alloys with 5% Fe showed α+β-phases whereas those with 10–20% Fe had a single β-phase\(^{22}\). It was established that the metastable single β-phase can be acquired by alloying titanium with at least 8% iron.

Ti-Fe system has an eutectoid point of α-titanium and TiFe at 17% Fe and 595°C. An intermetallic compound (TiFe) is precipitated within the cast alloy as a result of the eutectoid reaction. TiFe is also formed during cooling from the β region of the alloy towards the eutectoid reaction. TiFe precipitated within the cast alloy as a result of the eutectoid reaction. TiFe is also formed during cooling from the β region of the alloy towards the eutectoid reaction.
Tensile strength of alloys with 5% Fe was significantly higher than that of pure titanium. Precipitation strengthening by the dual-phase (α+β) structure, as well as solid-solution strengthening of the α- and/or β-phases, probably contributed to the high strength of 5% Fe. In spite of α+β structure being exhibited, the tensile strength of alloys with 6 and 7% Fe was similar to that of titanium. The existence of ω-phase in the 6 and 7% alloys though undetected on XRD patterns may have reduced their strength. ω-phase; commonly known to be hard and brittle, is also difficult to identify through XRD. The α-phase sometimes precipitates in β-phase during casting depending on the cooling rate and composition of alloy18). Additionally, low elongation of 5% Fe may have precipitated small amounts of ω. Therefore, the high hardness of 5–7% Fe may be associated with the precipitated albeit undetected ω-phase. Beyond 7% Fe up to 10% Fe, tensile strength of the alloys increased with increase in Fe concentration. Solid-solution strengthening of the β-phase contributed to the increased strength. It is known that β-titanium alloy with high concentration of alloying element has high strength. The alloy with 9% Fe demonstrated the highest strength of more than 850 MPa in this study. However, for alloys with more than 10% Fe, the tensile strength decreased with increase in Fe concentration probably due to decrease in elongation.

The yield strength of alloys with 8–10% Fe was more than 600 MPa. The value is over 2.5 times greater than that of titanium and satisfies the ISO 22674 criteria for type 5 metallic materials20). The elongation however did not meet the set ISO criteria.

**Possibility of improved wear resistance**

Metals with excellent wear resistance should have both suitable hardness and elongation. For example, a Co-Cr alloy, a dental alloy associated with excellent wear resistance does possess high hardness and large elongation. There was little research work found that investigated wear resistance of dental titanium alloys without surface treatment. A review article19) on characterization of titanium alloys, including Ti-6Al-4V, concluded that a reduction in ductility improved wear resistance of titanium. Koike et al. reported that industrial Ti-Fe-O-N alloys which had a small amount of Fe (1%), and Ti-6Al-4V alloy showed; higher bulk hardness, lower elongation, and better wear resistance than commercially pure titanium20). Therefore, a combination of high hardness and low ductility in Ti-Fe alloys perhaps enhances the wear resistance. Hardness of all the Ti-Fe alloys tested in this study was significantly higher than that of pure titanium, and Ti-6Al-4V alloy. Although the hardness of alloys with: 5–7% Fe (α+β), 20 and 25% Fe (β+TiFe), was higher than that of Co-Cr alloy; their elongation was almost 0. On the other hand, hardness of alloys with 8–13% Fe (β-phase) was equal to that of Co-Cr alloys, although the fractured surfaces of 8–11% Fe showed ductility. Although Ti-Fe alloys with 8–11% Fe exhibited high hardness but low ductility; we postulate they are possible candidates for development of an alloy with good wear resistance. The results of wear tests done in this study confirmed that the wear resistance of Ti-10% Fe alloys was better than that of titanium as expected. However, since factors which influence wear resistance are complex, we cannot deny that the wear resistance of other Ti-Fe alloys which show higher hardness than Co-Cr alloys may be fairly good. Therefore, there is need to conduct detailed wear resistance test on other Ti-Fe alloys and investigate the influence that hardness and elongation bear on wear resistance.

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