Effect of Microstructure on Tensile Properties and Static Fracture Toughness of Dental Gold Alloy

Mitsuo Niinomi¹, Junji Takeda¹, Toshikazu Akahori¹, Hisao Fukui², Masashi Touyama²,* and Hiroyuki Toda¹

¹Department of Production Systems Engineering, Toyohashi University of Technology, Toyohashi 441-8580, Japan
²Department of Dental Materials Science, School of Dentistry, Aichi-Gakuin University, Nagoya 464-8650, Japan

Tensile tests and static fracture toughness tests were conducted on dental type 4 gold alloys subjected to various heat treatments. The effects of microstructures on tensile characteristics and static fracture toughness are discussed.

The tensile strength of dental type 4 gold alloy increases with the solutionizing temperature. Moreover, the tensile strength of dental type 4 gold alloy increases with aging time at a solutionizing temperature of 1023 K. On the other hand, dental type 4 gold alloy exhibits reduced elongation with an increase in the solutionizing temperature.

Static fracture toughness of dental type 4 gold alloy increases with an increase in the solutionizing temperature. Static fracture toughness of dental type 4 gold alloy aged for 0.3 ks is the highest, and is the lowest when it is aged for 1.8 ks, with the solutionizing temperature at 1023 K.

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1. Introduction

In Japan, gold and silver alloys have primarily been used as dental materials. Gold alloy is expensive. However, since it possesses excellent mechanical and handling properties, and corrosion resistance, it is a widely used material for dental restoration. The tensile properties of a gold alloy, such as tensile strength, elongation, and hardness are only slightly affected by heat treatment, as reported.¹⁻³ However, thus far, there are no cited reports available on fracture characteristics of a gold alloy with regard to its microstructure and heat treatment. Therefore, in the present study, the microstructure of dental type 4 gold alloy was altered by various heat treatments. Subsequently, the effect of microstructures on tensile properties and fracture characteristics was investigated. Furthermore, tensile properties and fracture characteristics of the gold alloy were compared to those of dental Ag–Pd–Cu–Au (12 mass% Au) type alloy, which have already been reported.⁴⁻⁶

2. Experimental Procedures

2.1 Material

The materials used in the present study were rolled plates of a commercial ISO standard type 4 gold alloy, i.e., PGA-2 (ISHIFUKU Metal Industry Co., Ltd.; Au: 68.0, Cu: 15.0, Ag: 9.2, Pt: 5.0, Pd: 2.0, others: 0.8 mass%), and a commercial dental Ag–Pd–Cu–Au–Zn type alloy, i.e., S-12 (ISHIFUKU Metal Industry Co., Ltd.; Ag: 51.0, Pd: 20.0, Cu: 14.5, Au: 12.0, Zn: 2.0, others: 0.5 mass%) with a diameter of 10 mm, where very low rolling reduction ratio was applied.

2.2 Heat treatment

Some of the bars were solutionized in vacuum at 1023 and 1073 K for 3.6 ks and then quenched into ice water. These heat treatments are denoted as 1023WQ and 1073WQ, respectively. Aging treatment in vacuum, at 623 K for 0.3 and 1.8 ks, followed by quenching into ice water was then performed on some of the bars solutionized at 1023 K in vacuum. For S-12, aging treatment at 623 K for 0.3 ks was not conducted. These heat treatments are denoted as 1023WQ₃₆₃ and 1023WQ₁₈₈₆₃, respectively. The remaining bars were also solutionized in vacuum at 1073 K for 3.6 ks. and then air-cooled to room temperature (295 K). These heat treatments are denoted as 1073AC.

2.3 Tensile test

Tensile test specimens with a gage size of 2 × 4 × 20 mm³ were machined from the heat-treated bars with their longitudinal direction parallel to the rolling direction of the bars [Fig. 1(a)]. Tensile tests were conducted using an Instron type testing machine with a capacity of 9.8 kN, at a crosshead speed of...
8.33 \times 10^{-6} \text{ m/s}, at room temperature in air. The load was measured by a load cell located in the testing machine. The strain was measured using a strain gage directly attached to the parallel portion of the specimen. The elongation was measured using a reading microscope.

2.4 Static fracture toughness test
Static fracture toughness test specimens with a size of $4 \times 8 \times 40 \text{ mm}^3$ were machined from the heat-treated bars with their longitudinal direction parallel to the rolling direction of the bars [Fig. 1(b)]. A slit with a width of 0.3 mm and a length of 2.5 mm was introduced into the test specimen. Subsequently, a fatigue pre-crack was introduced into the test specimen according to the ASTM E813 so as to obtain $a_0/W$ ($a_0$: initial crack length and $W$: specimen width) equal to 0.52.

Three-point bend fracture toughness tests were then conducted using the same machine that was used for the tensile tests described in Section 2.3, at a crosshead speed of $5.0 \times 10^{-6} \text{ m/s}$, at room temperature in air. The crack initiation point was detected using the compliance ratio method\textsuperscript{7,8} and the DC electrical potential method.\textsuperscript{9}

2.5 Observations of microstructure and fracture surface
For microstructural observations, small samples were sectioned from the static fracture toughness tested specimens. These samples were then ground, polished, etched, and then characterized using an optical microscope. Phase constitution was characterized by X-ray diffraction. The fracture surface was observed using a scanning electron microscopy (SEM).

3. Results and Discussion
3.1 Microstructure
SEM micrographs of PGA-2 subjected to each heat treatment are shown in Fig. 2. The grain diameter of the alloy subjected to $1023\text{WQ}_{1.8 \text{ ks}}$ is relatively small as compared to that of the alloy subjected to $1023\text{WQ}_{0.3 \text{ ks}}$, \textit{i.e.}, the grain diameter of the alloy that has undergone aging treatment decreases with an increase in aging time. The grain diameter of the alloy subjected to $1073\text{AC}$ is relatively small as compared to that of the alloy subjected to $1073\text{WQ}$, \textit{i.e.}, the grain diameter of the alloy that was air cooled after heat treatment is relatively small as compared to that of the alloy that was water-quenched after heat treatment. These trends are almost similar to the other report.\textsuperscript{10}

XRD profiles obtained from the specimen surface of PGA-2 subjected to each heat treatment are shown in Fig. 3. The peaks of $\alpha$ phases are recognized on the specimen surface of the alloy subjected to each heat treatment. The peaks of the ordered lattice of AuCu I type, which exhibits an fct
3.2 Tensile properties

The tensile strengths of PGA-2 and S-12 subjected to each heat treatment are obtained from a tensile test as shown in Fig. 4. The tensile strength of PGA-2 tends to increase as compared to that of S-12. However, the tensile strength of PGA-2 subjected to 1073WQ is lower than that of S-12 subjected to 1073WQ. The tensile strength of each alloy subjected to solution treatment increases with an increase in the solutionizing temperature. However, the rate of increase of tensile strength of PGA-2 subjected to solution treatment is very small. The tensile strength of each alloy subjected to aging treatment is relatively high as compared to that of each alloy subjected to solution treatment. Furthermore, the tensile strength of PGA-2 subjected to aging treatment tends to increase with an increase in aging time. The tensile strength of each alloy subjected to 1073AC is relatively high as compared to that of each alloy subjected to 1073WQ, i.e., the tensile strength each alloy that was air-cooled after heat treatment is relatively high as compared to that of each alloy that was water-quenched after heat treatment. The tensile strength of PGA-2 exhibits a remarkable increase particularly when compared to that of S-12.

The elongations obtained from a tensile test of PGA-2 and S-12 after each heat treatment are shown in Fig. 5. The elongation of PGA-2 increases as compared to that of S-12. Contrary to the abovementioned trend in tensile strength, the elongation of each alloy subjected to solution treatment decreases with an increase in the solutionizing temperature. The elongation of each alloy subjected to solution treatment is relatively high as compared to that of each alloy subjected to aging treatment. The elongation of PGA-2 subjected to 1073AC is relatively low as compared to that of PGA-2 subjected to 1073WQ. Contrary to this result, the elongation of S-12 subjected to 1073AC is relatively high as compared to that of S-12 subjected to 1073WQ. Furthermore, the elongation of PGA-2 subjected to aging treatment tends to decrease remarkably with an increase in aging time.

It has been reported that, in general, the tensile strength of metals increases with an increase in the hardness. The Vickers hardness values of PGA-2 and S-12 for each heat treatment are shown in Fig. 6. The relationship between Vickers hardness and tensile strength of PGA-2 and S-12 for each heat treatment is shown in Fig. 7. The Vickers hardness of each alloy subjected to solution treatment increases with an increase in solutionizing temperature. Therefore, it is acknowledged that a correlation exists between tensile strength and Vickers hardness in each alloy. Furthermore, the Vickers hardness of PGA-2 subjected to aging treatment increases with an increase in aging time. Therefore, it is considered that the tensile strength increases with aging time.

The rate of increase of tensile strength with an increase in solutionizing temperature of S-12 is relatively high as compared to that of S-12. However, the precipitated phase dissolves easily in the matrix with an increase in the solutionizing temperature. Consequently, the hardness of S-12 increases due to the solid-solution hardening. Therefore, the tensile strength of S-12 exhibits a remarkable increase with an increase in the solutionizing temperature. On the other hand, the hardness of PGA-2...
increases due to the formation of the disordered-ordered lattice. Therefore, it is considered that the tensile strength of PGA-2 did not increase because the lattice did not change from the disordered lattice state.

The mechanisms of age-hardening for PGA-2 and S-12 are different. The hardness of PGA-2 increases due to the lattice strain caused by the formation of the ordered lattice of AuCu I type. The hardness of PGA-2 is known to increase due to the formation of this ordered lattice.\textsuperscript{1-3} The intensity of the ordered lattice of AuCu I type tended to increase with the aging time, as shown in Fig. 3. There is a correlation between the diffracted intensity and volume fraction of phase. Therefore, it is considered that the volume fraction of the ordered lattice of AuCu I type increases with an increase in this diffracted intensity. It has been qualitatively proven that the volume fraction of the ordered lattice of AuCu I type increases with an increase in the aging time. Consequently, it is considered that the grain diameter of the alloy subjected to aging treatment decreases with an increase in the aging time due to an increase in the volume fraction of this ordered lattice. Although in this study, it could not be recognized, it is an established fact that the Ag-rich solution phase precipitates in the grain boundary in the gold alloy.\textsuperscript{10}

On the other hand, the hardness of S-12 increases due to the precipitation of an intermetallic compound referred to as \(\beta\) phase, which results from the aging treatment.\textsuperscript{13}

Large differences in tensile strength and elongation occurred due to differences in cooling rates (air-cooled and water-quenched), for PGA-2 and S-12. This appears to be the differentiating factor that affects the formation processes of the microstructure. In S-12, the \(\beta\) phase precipitates significantly further in the air-cooled alloy than in the water-quenched alloy. Simultaneously, the relatively low-intensity and ductile phase (the Cu-rich phase) also exhibits significant precipitation. Therefore, the improvement in tensile strength and elongation appears to be minor. On the other hand, in PGA-2, the ordered lattice of AuCu I type principally extends to a greater length in the air-cooled alloy than in the water-quenched alloy. Consequently, the hardness increases due to the lattice strain caused by the formation of the ordered lattice of AuCu I type. Therefore, it is considered that the tensile strength increases remarkably with a decrease in elongation in the air-cooled alloy.

### 3.3 Static fracture toughness

The static fracture toughness \(J_{\text{in}}\) obtained from a static fracture toughness test of PGA-2 and S-12, subjected to each heat treatment, are shown in Fig. 8. The static fracture toughness of PGA-2 for 1023WQ\(_{1.8ks}\) and 1073WQ is remarkably high as compared to that of S-12. The static fracture toughness of PGA-2 subjected to solution treatment exhibits a trend similar to that for the tensile strength of PGA-2 with an increase in the solutionizing temperature. The static fracture toughness of PGA-2 subjected to aging treatment decreases with an increase in the aging time. The static fracture toughness of S-12 subjected to 1073AC is almost similar to that of the alloy subjected to 1073WQ. However, the static fracture toughness of PGA-2 subjected to 1073AC is remarkably low as compared to that of the alloy subjected to 1073WQ.

The static fracture toughness of PGA-2 is relatively high as compared to that of S-12. However, the static fracture toughness of PGA-2 subjected to 1023WQ\(_{1.8ks}\) and 1073AC is low as compared to that of S-12. Typical fractograph of PGA-2 subjected to each heat treatment were obtained from static fracture toughness specimen and are shown in Fig. 9. In PGA-2 subjected to 1023WQ\(_{1.8ks}\) and 1073AC, it can be recognized that grain boundary fracture has occurred. Generally, in an Au–Ag–Cu system alloy, the Ag-rich solution phase precipitates in the grain boundary when the ordered lattice of AuCu I type is formed by the aging treatment.\textsuperscript{10} In PGA-2 subjected to 1023WQ\(_{1.8ks}\) and 1073AC, the result of X-ray diffraction shows that the formation of the ordered lattice of AuCu I type increases. The lattice strain accumulates with the formation of the ordered lattice of AuCu I type. Consequently, grain boundary fracture occurs. Therefore, the static fracture toughness value is greatly lowered. On the other hand, PGA-2 subjected to 1023WQ\(_{3.8ks}\) exhibited the highest static fracture toughness value. The result of X-ray diffraction shows that the volume fraction of the ordered phase precipitates increases with an increase in the volume fraction of this ordered phase.
lattice of AuCu I type in PGA-2 subjected to 1023WQ0.3 ks is small as compared to that in the alloy subjected to 1023WQ1.8 ks and 1073AC, as shown in Fig. 3. Consequently, there is a relative increase in elongation. Subsequently, the stress concentration in the grain boundary decreases. As evidence, grain boundary fracture has not occurred, as shown in Fig. 9. This mechanism appears to increase the static fracture toughness value.

It is recognized that tensile strength and static fracture toughness value can be greatly improved by the aging treatment, which is optimum for this gold alloy. In the present study, the optimum heat treatment condition for tensile properties and static fracture toughness of this alloy is 1023WQ0.3 ks.

4. Conclusions

Tensile properties and static fracture toughness of commercial dental type 4 gold alloy subjected to various heat treatments were investigated in terms of heat treatment condition and fracture mechanism in the present study. The following results were obtained.

1. The tensile strength and static fracture toughness value of PGA-2 is relatively high as compared to that of S-12.
2. The tensile strength of PGA-2 subjected to 1023WQ1.8 ks is the highest, with the value of the tensile strength of PGA-2 subjected to 1073AC being approximately equal to it.
3. The elongation of PGA-2 decreases with an increase in the solutionizing temperature and aging time. In particular, the elongation of PGA-2 subjected to 1023WQ is the highest.
4. The static fracture toughness value of PGA-2 subjected to 1023WQ0.3 ks is the highest. The static fracture toughness value of PGA-2 subjected to 1023WQ1.8 ks and 1073AC decreases remarkably with an increase in the formation of the ordered lattice of AuCu I type.

REFERENCES