



Precipitation of β' phase and hardening in dental-casting Ag–20Pd–12Au–14.5Cu alloys subjected to aging treatments



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ABSTRACT

The age-hardening behavior of the dental-casting Ag–20Pd–12Au–14.5Cu alloy subjected to aging treatment at around 673 K is well known, and this hardening has been widely employed in various applications. To date, the age-hardening of this alloy has been explained to attribute to the precipitation of a β phase, which is a B2-type ordered CuPd phase or $PdCu_xZn_{1-x}$ phase. In this study, results obtained from microstructural observations using a transmission electron microscopy and a scanning transmission electron microscopy revealed that a fine $L1_0$ -type ordered β' phase precipitated in the matrix and a coarse-structure region (consisting of Ag- and Cu-rich regions) appeared after aging treatment at 673 K and contributed to increase in hardness. The microstructure of the coarse β phase, which existed before aging treatment, did not change by aging treatment. Thus, it is concluded that the fine β' phase precipitated by aging treatment contributed more to increase in hardness than the coarse-structure region and coarse β phase.

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1. Introduction

Ag–Pd–Au–Cu alloys, substitute materials for high-cost Au alloys, are well-known dental materials for prosthetic restoration because these alloys have good formability, good mechanical properties, and good corrosion resistance [1–8]. It is essential that these alloys have good mechanical properties because they are prone to high occlusal forces during their function. The conventional hardening method for Ag–Pd–Au–Cu alloys is aging treatment around 673 K. The Ag–20Pd–12Au–14.5Cu alloy exhibits age-hardening [9] and this hardening has been explained by the precipitation of CsCl(B2)-type ordered CuPd phases (β) [10]. The investigation of the microstructure and mechanical strength of the Ag–20Pd–12Au–14.5Cu alloy has revealed that the β phase, which is known to induce precipitation hardening upon aging treatment, is coarse and the microstructure of this phase hardly changes by heat treatment [11]. Moreover, it has been reported that the β phase may not be a CuPd ordered phase but a $PdCu_xZn_{1-x}$ phase [4,12]. It has been mentioned that Zn grows as a primary crystal in this phase by bonding to Pd and reduces hardening [12]. The Ag–20Pd–12Au–14.5Cu alloy exhibits a unique hardening behavior in addition to the conventional age-hardening behavior. This unique hardening behavior is demonstrated through the enhancement in the mechanical strength of the alloy by high-temperature solution treatment without any aging treatment [13,14]. This unique hardening behavior has been explained to be attributed to the precipitation of $L1_0$ -type ordered phases

(β') [15]. To date, our research group has clarified that in comparison with the coarse β phase, the fine β' phase precipitated by high-temperature solution treatment may contribute more to the unique hardening of the Ag–20Pd–12Au–14.5Cu alloy [11]. It has been considered that precipitation of the β' phase significantly contributes to the hardening of the Ag–20Pd–12Au–14.5Cu alloy. In addition, it has been reported that in the Ag–25Pd–25Au–10Cu alloy—a type of Ag–Pd–Au–Cu alloys— β' phases precipitate by aging treatment [16]. Therefore, it is necessary to consider the contribution of the β' phase to the hardening of the Ag–20Pd–12Au–14.5Cu alloy subjected to aging treatment. The aim of this study was to clarify the relationship between the microstructure and hardening of the Ag–20Pd–12Au–14.5Cu alloy subjected to aging treatment. Ag–20Pd–12Au–14.5Cu alloys are used as dental casting alloys for dental restorations (crown and inlay). This type of applications requires the mechanical strength because high occlusal forces are applied during eating. On the other hands, this type of alloys is expensive because they consist of precious metals. The hardness is related to the tensile strength, the stiffness, and the wear resistance and can be measured using small size of specimens. Therefore, the hardness was measured for evaluation of mechanical properties in this study.

2. Materials and methods

2.1. Materials and heat treatment processes

The materials used in this study were commercial dental Ag–20Pd–12Au–14.5Cu alloys (Ishifuku Kinpara S12, Ishifuku Metal Industry Co., Ltd.) fabricated by a wrought process (hereafter referred to as S12). Their chemical composition was 51%Ag–20%Pd–12%Au–14.5%Cu–2.5%

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others (Zn, Ir, In) (mass%). The dimensions of S12 were 12.0 mm × 6.0 mm × 1.0 mm. In order to investigate the age-hardening behavior of S12, aging treatments at various temperatures were carried out as schematically shown in Fig. 1(a). Seven S12 were subjected to solution treatment (ST) at 1023 K for 3.6 ks in vacuum of about 1 Pa and were then quenched in cold water (hereafter referred to as ST1023). Moreover, each ST1023 was subjected to aging treatments (AT) at each temperature of 573, 623, 673, 723, 773, 823 and 873 K for 1.8 ks in vacuum of about 1 Pa and was then quenched in cold water (hereafter referred to as ST-AT573, ST-AT623, ST-AT673, ST-AT723, ST-AT773, ST-AT823, ST-AT873). In order to observe the changes in the microstructure of ST-AT673, which exhibits an age-hardening behavior, further solution treatment was carried out at 1023 K for 3.6 ks in a vacuum of about 1 Pa, followed by quenching in cold water (hereafter referred to as ST-AT-ST1023), as schematically shown in Fig. 1(b).

2.2. Microstructural observations

ST1023, ST-AT573, ST-AT623, ST-AT673, ST-AT723, ST-AT773, ST-AT823, ST-AT873, and ST-AT-ST1023 were wet-polished using emery papers of up to 4000 grit, and were then mirror-polished using an alumina suspension. Commercial alumina suspensions with a diameter of 1 μm and 0.05 μm (Baikalox, Baikowski) were used to mirror-polish specimens. X-ray diffraction (XRD) analysis of these alloys was carried out using a diffractometer (D8 DISCOVER, Bruker) (operating at 40 kV and 40 mA) to identify the constitutional phases. A transmission electron microscope (JEM-2000EXII, JEOL) was used at an acceleration voltage of 200 kV for observing the microstructures of the alloys. For this, ST1023, ST-AT673, and ST-AT-ST1023 were cut into 2.0 mm × 2.0 mm × 1.0 mm pieces by diamond saw. These specimens were ground to a thickness of 50 μm by using emery papers of up to 4000 grit, and were then mirror-polished using an alumina suspension. The specimens were thinned to 10 μm in thickness by dimple grinder. After that, the specimens were thinned until a hole was made by ion milling. In addition, the microstructure of ST-AT673 was observed using a scanning transmission electron microscope (STEM) (TITAN80-

300, FEI) equipped with a high-angle annular dark-field (HAADF) detector at an acceleration voltage of 300 kV. The elemental distributions in ST-AT673 were analyzed using an energy-dispersive X-ray spectroscopy (EDX) detector installed in the STEM.

2.3. Mechanical evaluation

The hardness measurements were performed on each mirror-polished specimen using a Vickers microhardness tester (Shimadzu microhardness tester HMV-1ADW, Shimadzu Corp.) with a load of 4.9 N and a holding time of 15 s. Each specimen was tested 10 times and the average value of these iterations was used as the hardness of the specimen. Vickers hardness (Hv) was considered as the average hardness of 3 specimens. Furthermore, data of Vickers hardness was statistically analyzed by one-way ANOVA.

3. Results and discussion

The Vickers hardness of ST1023, ST-AT573, ST-AT623, ST-AT673, ST-AT723, ST-AT773, ST-AT823, and ST-AT873 are shown in Fig. 2. The error bars are calculated using standard deviation. The Vickers hardness of ST1023 was found to be about 170.1 Hv. Moreover, it increased with increasing AT temperature and showed the maximum value of about 283.4 Hv for ST-AT673. Vickers hardness then decreased with increasing AT temperature.

Fig. 3 shows the compositional phases of ST1023, ST-AT573, ST-AT623, ST-AT673, ST-AT723, ST-AT773, ST-AT823, and ST-AT873 as determined from their XRD profiles. It appears that the peak intensity and positions of the β phase, which is known to induce age-hardening [10], hardly changed by aging treatment. In ST-AT673, the (110) peak of the β' phase appeared at around 31–32° (Fig. 3(b)). It is assumed that the precipitation of the second phase like the β' phase contributed more to the increase in hardness than that of the β phase, the composition and microstructure of which did not change by aging treatment.

Fig. 4 shows the changes in the hardness of ST1023, ST-AT673, and ST-AT-ST1023. The hardness of age-hardened ST-AT673 decreased by

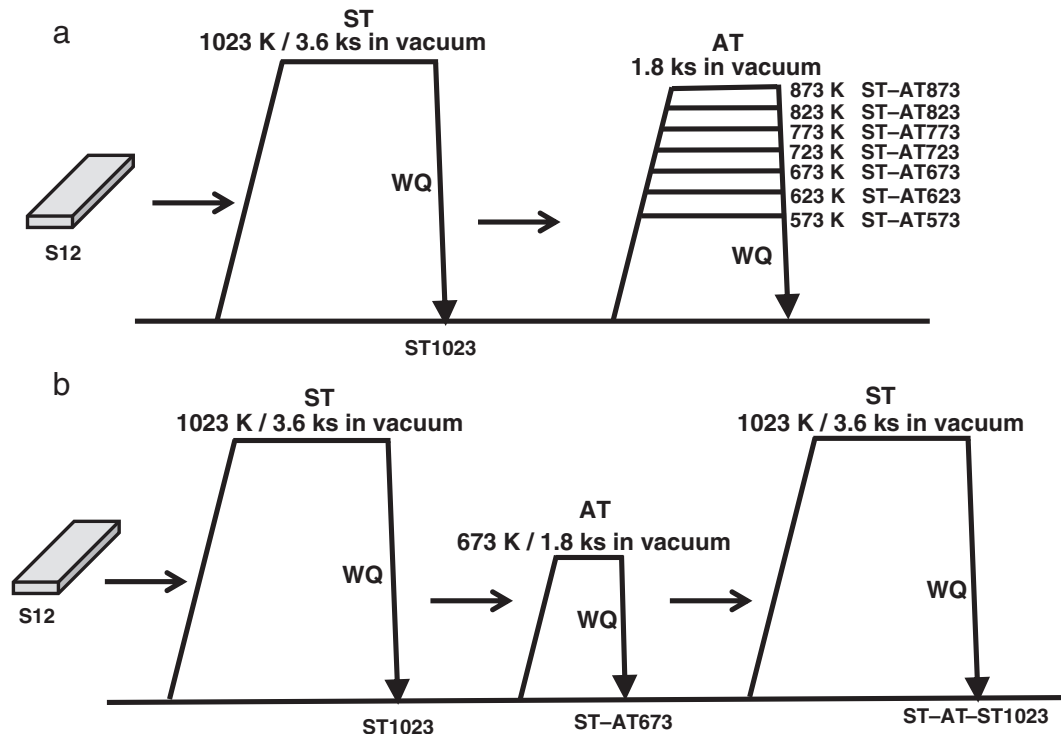


Fig. 1. Schematic drawings of heat treatments: (a) aging treatments at temperatures in the range of 573–873 K, and (b) aging treatment and subsequent solution treatments. ST: solution treatment followed by water quenching (WQ), AT: aging treatment followed by WQ.

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