



# Age-hardening by the formation of metastable phases in an In-added Au–Ag–Cu–Pd alloy

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## ABSTRACT

The age-hardening of an In-added Au–Ag–Cu–Pd alloy was examined by characterizing the hardening behavior, phase transformations and changes in microstructure, and elemental distribution during aging over the temperature range 200 to 500 °C. The major product phases, Ag-rich  $\alpha_1$  and AuCu I, were formed homogeneously from the parent  $\alpha_0$  phase by the transformation of precursor metastable phases in the grain interior. This led to a significant age-hardening response. A minor product phase, InPd, containing Cu and Zn, contributed to the overall hardness increase by initiating a grain boundary precipitation reaction. Continued long-time aging led to a hardness decrease due to overaging, which involved the formation of a lamellar microstructure composed of the stable Ag-rich  $\alpha_1$  and AuCu I phases. At the longest aging time at 400 °C, the lamellar structure consumed the matrix  $\alpha_1$  and AuCu I phases.

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## 1. Introduction

Dental casting gold alloys for crown and bridge fabrication can be sufficiently hardened by a proper heat treatment to withstand occlusion in the oral environment. The age-hardening mechanism of dental casting gold alloys is variable according to the alloy composition. In the case of an alloy mainly composed of Au, Ag and Cu, the age-hardening is generally related to the phase decomposition of the single parent phase into an Au-containing Ag-rich phase and the AuCu I or AuCu II phases [1,2] from the fact that Au is soluble with Ag and Cu at any atomic ratio but Ag and Cu have limited mutual solubility [3]. However, even for the alloys that develop the same final product phases by aging, the mechanism of the phase transformation can be different if, for example, the product phases are formed through a metastable state [4,5]. Accordingly, the mechanism of hardening and subsequent softening by overaging may be different. Formation of a metastable phase usually increases the hardness in the alloy. However, the subsequent transformation of the metastable phase into the

stable phase may cause either a decrease or an increase in hardness depending on the related microstructural changes.

In the present study, the phase transformations occurring in an Au–Ag–Cu–Pd alloy containing In and small amounts of minor elements were studied in order to determine their relationship to changes in hardness, microstructure and elemental distribution. The characterizations were performed by means of hardness testing, X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectrometric (EDS) analysis.

## 2. Experimental Details

The alloy used in the present study was a light yellow semi-precious dental alloy used in crown and bridge fabrication (Goldenian C-61, Shinhung, Korea). The chemical composition of the alloy is listed in Table 1. The initial shape of the specimen was plate-like with dimensions of 10 mm × 10 mm × 0.6 mm, and was procured in a rolled and annealed state.

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**Table 1 – Chemical composition of the alloy used**

Composition	Au	Ag	Cu	Pd	In	Pt	Zn	Ir
wt.%	61	20	13	3.3	1	1	0.5	0.2
at.%	41.12	24.61	27.17	4.11	1.16	0.68	1.01	0.14

Before hardness testing, as recommended by the manufacturer the specimens were solution-treated at 760 °C for 10 min under an argon atmosphere, and then rapidly quenched into ice brine to preclude thermal changes on cooling. They were subsequently aged isochronally for 10 and 20 min in the temperature range of 200 °C to 500 °C, and were isothermally aged at 350 °C and 400 °C for various periods of time. All aging was done in a molten salt bath (25% KNO<sub>3</sub> + 30% KNO<sub>2</sub> + 25% NaNO<sub>3</sub> + 20% NaNO<sub>2</sub>), followed by quenching into ice brine. Hardness measurements were made using a Vickers micro-hardness tester (MVK-H1, Akashi Co., Japan) with a load of 300 g-f and a dwell time of 10 s. Vickers hardness results were recorded as the average values of five measurements.

For the X-ray diffraction study powder specimens which passed through a 330-mesh screen were obtained by filing the plate-like samples. After being vacuum-sealed in a silica tube and solution-treated at 760 °C for 10 min, they were isothermally aged at 400 °C for various periods of time in a molten salt bath, and then quenched into ice brine. The X-ray diffraction profiles were recorded by an X-ray diffractometer (D/Max-2400, Rigaku Denki Co. Ltd., Japan). The X-ray diffractometer was operated at 30 kV and 40 mA, using Ni-filtered Cu K $\alpha$  radiation.

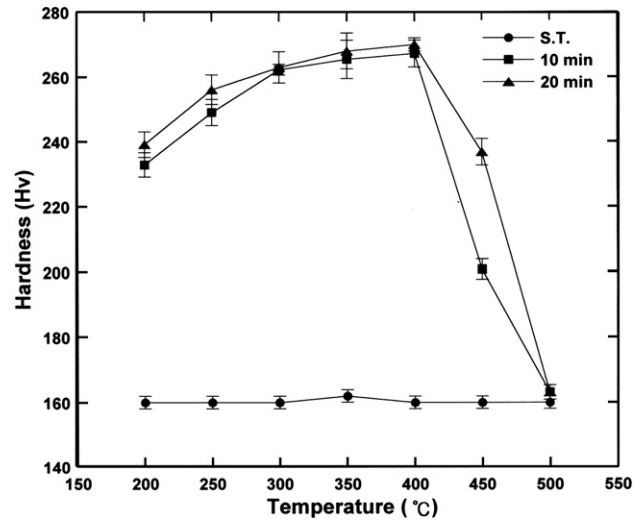
For the scanning electron microscopic observations, the plate-like samples were subjected to the required heat treatment, and then prepared by utilizing standard metallographic techniques. A freshly prepared aqueous solution of 10% potassium cyanide and 10% ammonium persulfate was used for the final etching of the samples. The specimens were examined at 20 kV with a scanning electron microscope (S-2400, Hitachi, Japan).

Energy dispersive spectrometric (EDS) analysis was done in order to observe the distributional changes of each element in the alloy during the aging process. For the EDS, the specimens were prepared in the same manner as was used for the scanning electron microscopy. An energy dispersive X-ray spectrometer (INCA x-sight, Oxford Instruments Ltd., UK) of a field emission scanning electron microscope (JSM-6700F, JEOL, Japan) was used at 15 kV to examine the specimens.

### 3. Results and Discussion

#### 3.1. Hardness Changes

To determine an appropriate aging temperature for the alloy, the specimens were isochronally aged in the temperature range of 200 °C to 500 °C. Fig. 1 shows the isochronal age-hardening curves of the specimen alloy solution-treated at 760 °C for 10 min, and then aged in the temperature range 200 °C to 500 °C for 10 and 20 min. The alloy exhibited age-hardening after aging at all temperatures from 200 °C to 400 °C. At 500 °C the hardness was essentially the same as that of the solution-treated condition. Relatively little



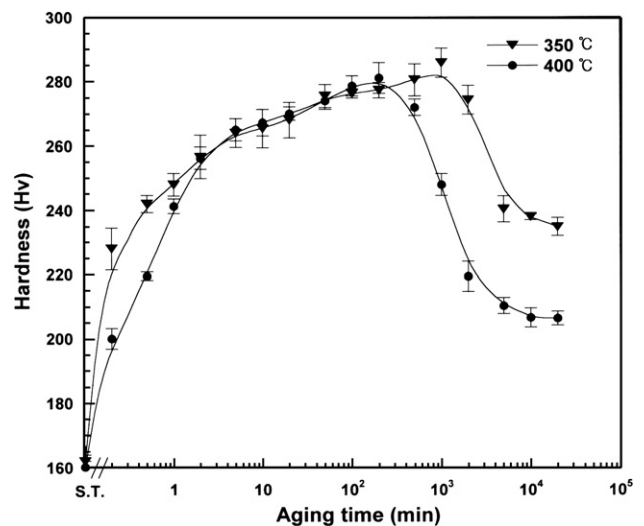
**Fig. 1 – Isochronal age-hardening curves of the subject alloy aged in the temperature range of 200 °C to 500 °C for 10 and 20 min.**

difference was seen between the specimens aged 10 and 20 min.

Fig. 2 shows the isothermal age-hardening curves of the alloy solution-treated at 760 °C for 10 min and then aged for various times at 350 °C and 400 °C. The age-hardening curves and age-hardening ability were similar at 350 °C and 400 °C, which suggested the same age-hardening mechanism at both aging temperatures, although overaging occurred more quickly at 400 °C. After attaining maximum hardness, a rapid decrease initially occurred, followed by apparently constant values at times greater than about 10,000 min.

#### 3.2. Phase Transformation

Fig. 3 shows the variations of the X-ray diffraction (XRD) pattern during 400 °C isothermal aging. In the solution-treated



**Fig. 2 – Isothermal age-hardening curves of the subject alloy aged at 350 °C and 400 °C.**

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