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Investigation of metastable γ′ precipitate using HRTEM in aged Cu–Be alloy

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ABSTRACT

Plate-like precipitates in Cu–1.9Be alloy have been studied using high-resolution transmission electron microscopy (HRTEM). The metastable γ′ phase and its matrix orientation were determined in samples which were solutionized, after aging at 320 °C for 2, 6 and 72 h. The relationships were determined as follows: [0 1 1] Cu-matrix (α)//[0 0 1] precipitate (γ′) and (1 0 0)α//(1 0 0)γ′. The habit planes were determined to be the $(3\,1\,\bar{1})$ and $(\bar{2}\,1\,\bar{1})$ planes of α for the γ′ phase. The angle between [1 0 0]γ′ and [2 0 0]α was found to be 6° and 6.5° for aging times of 6 h and 72 h, respectively. As the aging time increased, the length and width of the Cu–Be precipitates increased from 10–20 nm to 50–100 nm and 2–5 nm to 10 nm, respectively, along the trace of the habit planes.

1. Introduction

Cu–Be (copper–beryllium) alloy is a sensor material used in precipitation-hardened form for highly sensitive instruments and metrological applications, including central and end pivots, a flexure element in primary balances, or as a spring material in sensors [1,2]. Aged Cu–Be alloy has been widely used due to its high strength and hardness, good electrical and heat conductivities, non-magnetic character, low hysteresis properties and low elastic modulus relative to other light metals [1]. The precipitation sequence in Cu–Be alloys is described by Guinier–Preston (G.P.) zones, → γ″ → γ′ → γ [3–6]. The effect of prior deformation and applied stress on precipitation has been studied by some researchers [7–10]. Discontinuous precipitation was investigated in bicrystal [11] and polycrystal [12] Cu–Be alloys. The metastable and stable phases were characterized by X-ray diffraction [3,8,13], transmission electron microscopy (TEM) [2,5–7,9,11–14] and high-resolution TEM (HRTEM) [4,10,14,15]. However, a variety of results in these studies about the characteristics of the γ′ phase still exist. According to the published results, the γ′ lattice is body-centred tetragonal (b.c.t.) with $a = b = 0.279$ nm [3,5,6] and $c = 0.254$ nm [3]. Some researchers have determined that γ′ has a B2 type (f.c.c. and b.c.c. crystal lattice rearrangement) cubic structure with $a = b = 0.27$ nm [2,5].

In this paper, the transformed metastable phase that exists prior to the stable phase is studied by HRTEM using Cu–Be alloy containing 1.9 wt.% Be. We have found similar results compared with previous studies that have been published in the last 5 years [10,14,15] using lattice parameters, the cubic system and precipitate orientation with its matrix for the γ′ phase. The continuous γ′ phase has a b.c.t. lattice with $a = 0.254$ nm and $b = c = 0.268$ nm.

2. Material and experimental procedure

The alloy investigated was a commercial Cu–Be (UNS C17200) alloy supplied by Brush Wellman (A 25-H) that had a composition of copper (balance) with 1.9 wt.% beryllium and 0.2 wt.% cobalt. The material was supplied as a supersaturated solid solution (annealed at 790 °C for 1 h), water-quenched and 37% cold-worked into a hard temper. Precipitation hardening was applied at 320 °C and aged for 2, 6 and 72 h. The effect of aging time at 320 °C on the hardness is shown in Fig. 1. The hardness of the alloy reached a peak after aging for 3 h and then continuously decreased due to overaging.

The monolayer Guinier–Preston (G.P.) zones are Be-enriched plates that form coherently on {1 0 0} f.c.c. α-Cu matrix planes are obtained by beginning of precipitation (Fig. 1). The other metastable phases of γ″ and γ′, which form prior to the equilibrium γ phase, are obtained by holding the sample at the longer aging temperature. G.P. zones and other intermetallic particles have high dislocation density with regularly distributed precipitates which result low

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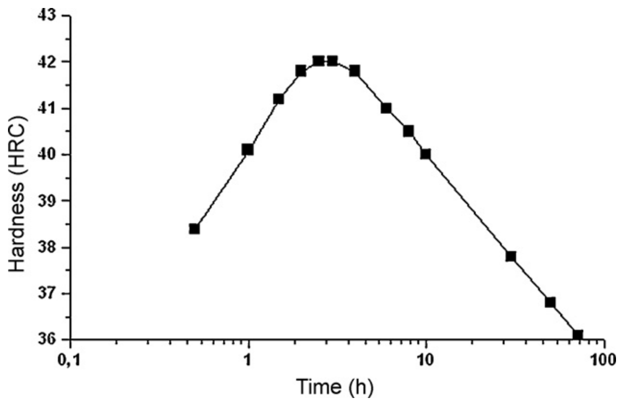

Fig. 1. Hardness of the specimen at 320 °C as function of aging time.

internal friction. Aging parameters, such as holding time and temperature, affect both Young's modulus and internal friction. They may change the phases present and their characteristics. Application of proper heat and/or thermo-mechanical treatments could minimize difference between the Young's moduli of Cu–Be alloys aged at different conditions. For elastic stability which is important for very accurate sensors, the dislocation density and the presence of precipitates and grain boundaries are beneficial. For obtaining lower internal friction, precipitates are desirable by acting as obstacles to the movement of dislocations [1,2].

For transmission electron microscopy (TEM) studies, specimens were cut, and 3 mm diameter discs were punched and ground to a thickness of less than 150 μm. Next, the specimens were perforated electropolishing with an electrolyte solution of 25% nitric acid + 75% methanol at about −30 °C with 12 V in a Struers-Tenupol-5 Double Jet Electropolisher. Moreover, for HRTEM studies, punched and ground 80 μm thick specimens were dimple ground with a Gatan 656 Dimple Grinder. Specimens were ion polished first at 4 kV and again after transparent area was achieved at 2 kV with a Gatan 691 Precision Ion Polishing System (PIPS). The foils were examined with a JEOL JEM 2100 HRTEM with a LaB_6 filament, which was operated at 200 kV. A JEOL side entry double tilt goniometer was used. Images were taken by a Gatan Model 694 Slow Scan CCD Camera. Dark Field (DF), Bright Field (BF) and Selected Area Electron Diffraction (SAED) techniques were used to investigate the microstructure. For digital images, a Gatan Digital Micrograph and Diffpack softwares were used. Moreover, noise filtering and fast Fourier transformation techniques were applied for HRTEM purposes.

3. Results and discussion

3.1. Lattice parameters of the precipitates

Precipitation hardening proceeded from G.P. zones, and the γ″ phase grew in size, and its coherency with the matrix increased; it was transformed to the semi-coherent intermediate precipitate γ′ [5]. The three quenched states are shown in a series of

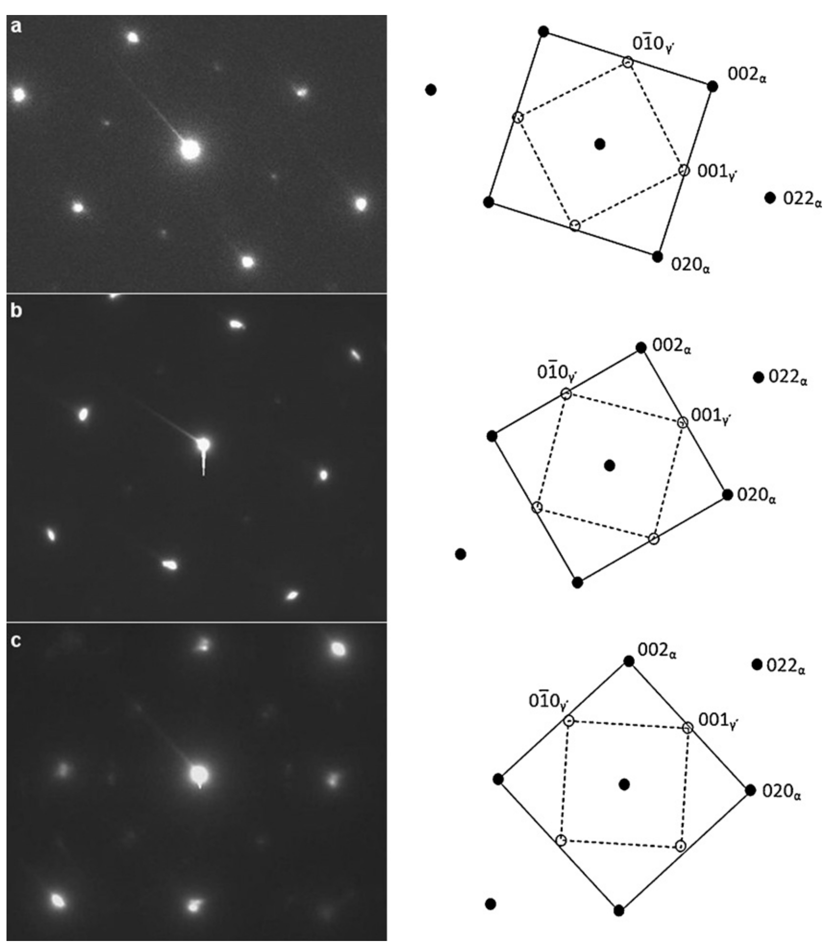

Fig. 2. [1 0 0] α SADP of the samples after aging at 320 °C (a) for 2 h (b) for 6 h (c) for 72 h.