Extraction of precipitates from age-hardenable Cu–Ti alloys

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An extraction procedure has been developed to separate the precipitates, namely, metastable fine $\alpha$-Cu$_4$Ti with a tetragonal structure and stable coarse $\beta$-Cu$_4$Ti with an orthorhombic structure, from an age-hardenable Cu–Ti alloys. When the alloy was submerged in a 7.0 M nitric acid solution at 273 K, only the Cu solid solution matrix was dissolved chemically, and both precipitates were left as insoluble residue. Subsequent structural and chemical-compositional analyses of the insoluble residue and filtrate determined the mass fraction and composition of the precipitates and matrix with high accuracy.
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1. Introduction

Age-hardenable copper–titanium (Cu–Ti) alloys are widely used as electrical components such as connectors and lead frames because of their excellent strengths and good electrical conductivity. Fig. 1 shows the Cu-rich portion of the Cu–Ti phase diagram [1]. Many studies have firmly established that precipitation in commercial Cu–Ti alloys containing approximately 3 to 6 mol% Ti occurs during aging in the following sequence: the Cu–Ti alloys are generally solution-treated in the region of the Cu solid-solution phase and then aged at temperatures between 600 and 800 K in vacuum. In the initial stage of aging, the supersaturated solid solution of copper with an fcc structure begins to decompose spinodally into two disordered fcc phases to form Ti-rich and Ti-depleted regions. The Ti-rich region then becomes ordered, and a metastable coherent precipitate, denoted as $\alpha$-Cu$_4$Ti, with a tetragonal D$_1$$_a$ structure (Prototype, Ni$_4$Mo; space group, I$_4$/m) forms continuously in the Cu matrix phase [2–7]. During prolonged aging, coarse cellular components composed of the stable intermetallic phase and the terminal Cu solid solution nucleate and grow discontinuously in the grain boundaries, consuming the finely dispersed $\alpha$-Cu$_4$Ti particles [2,3,8–10]. The stable phase generally has been reported to be $\beta$-Cu$_4$Ti with an ordered orthorhombic structure (Prototype, Au$_4$Zr; space group, P nma), although the structure is somewhat controversial [2,11,12].

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Since the mechanical and electrical properties of age-hardenable Cu–Ti alloys are critically influenced by their precipitation behavior, it is important to understand the volume fraction and compositional changes, accompanying the morphological and structural evolution of the α-Cu$_4$Ti and β-Cu$_3$Ti precipitates during aging. However, it is generally difficult to quantitatively evaluate the precipitates in the alloys using conventional X-ray diffraction (XRD) and transmission electron microscopy (TEM) techniques, because the volume fraction of the matrix phase is very large. The purpose of the present study, therefore, is to develop a simple and highly reliable method for evaluating the precipitates in age-hardenable Cu–Ti alloys. We employed an extraction procedure in which chemical dissolution is used to separate the precipitates from the Cu–Ti alloys. Although extraction procedures for the qualitative and quantitative evaluation of the precipitates have often been reported for age-hardenable aluminum alloys [13], steels [14–16], and complex materials [17], there have been few studies published on age-hardenable Cu systems [18,19]. Such an extraction procedure must be available so that the structure of the precipitates in the alloys can be directly confirmed. In addition, the mass fraction and composition of both the precipitates and the matrix could be examined by performing chemical analysis of the insoluble residues and filtrate, respectively.

### 2. Experimental Procedures

An alloy ingot with a nominal composition of Cu-4 mol% Ti was prepared by melting pure copper (99.99%) and titanium (99.99%) in argon gas and then hot-rolling the mixture down to a thickness of 0.2 mm at around 1223 K. From the sheet, strips measuring 55 mm in length and 6 mm in width were cut out. These specimens were homogenized and solution-treated at 1223 K for 30 min in evacuated quartz capsules, and then immediately quenched in ice water. After the surface oxidation layer was removed by mechanical grinding, the specimens were re-encapsulated in a vacuum of less than 2.0 × 10$^{-3}$ Pa and aged at 723 K for 12 h, 96 h, and 240 h.

To examine the chemical solubility of the specimen, the following procedure was carried out. The surface of the aged specimen was ground again and rinsed thoroughly in pure ethanol. Then the specimen was cut into pieces with dimensions of $2 \times 2 \times 0.2$ mm$^3$ so that they would efficiently dissolve in various chemical solutions, as listed in Table 1. Each solution was stirred in an ultrasonic bath during the dissolution process. Since the pieces left some insoluble residue after they were dissolved in nitric acid solutions, each solution was passed through a membrane filter with a pore diameter of 0.05 µm. The filtered residue was washed thoroughly with pure water and then dried in a desiccator. The filtrate and washing solution were collected in a beaker that contained a solution of 9.0 mol/L (M) sulfuric acid to avoid hydrolysis of the titanium during filtration.

The microstructure of the aged specimens and the morphology of the insoluble residues were observed using field-emission scanning electron microscopy (FE-SEM) with a HITACHI SU8000 operated at 15 kV and using TEM with a JEOl JIM-3010 operated at 300 kV. For the FE-SEM microstructure observation, the specimens were mechanically polished using the 2000-grade emery paper and then chemically etched by a 7.0 M nitric acid solution at 273 K for 10 s. For the TEM microstructure observation, the thin-foil specimens were first ground to less than 50 µm in thickness and then subjected to low-angle ion milling with an argon ion beam accelerated below 3 kV. High-purity (99.9999% pure) argon gas was used for the argon ion beam source. For the FE-SEM and TEM morphology observations, the residues were mounted on molybdenum mesh-grids. To obtain structural information on the specimen and insoluble residues, XRD analysis was carried out using a PANalytical X’Pert Pro diffractometer with Cu Kα radiation at 40 kV. The masses of Cu and Ti elements in the specimens were checked by performing inductively coupled plasma-optical emission spectrometry (ICP-OES) using a Thermo Fisher Scientific IRIS Advantage DUO. The mass fractions of the insoluble residue and filtrate separated from the specimens were also determined by ICP-OES measurements, based on the weight of the specimen before it was dissolved in the solution.

### 3. Results and Discussion

#### 3.1. Microstructure of Aged Cu–Ti Alloys

The chemical composition of the aged specimens was found to be Cu–(3.96 ± 0.06) mol% Ti from the ICP-OES measurements, which is close to the nominal composition of Cu–4 mol% Ti.

Fig. 2 shows the FE-SEM images of the specimens aged at 723 K for 0 h, 12 h, 96 h, and 240 h. The specimen aged for 0 h, which was solution-treated, had a single supersaturated Cu solid solution phase with recrystallization grains of approximately 30 µm (Fig. 2(a)). In the specimen aged for 12 h, which is the peak aging condition in terms of hardness [2,20], fine needle-shaped precipitates with lengths of 50 nm to 100 nm were observed in the grains (inset of Fig. 2(b)), together with a small amount of cellular precipitates from the grain boundaries. The volume fraction of the cellular precipitates (instead of needle-shaped Cellular precipitation of Cu–Ti Alloys

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**Fig 1 – Partial phase diagram of Cu–Ti binary system** [1].

![Phase Diagram](image-url)
precipitates) appeared to increase with increasing aging time (Fig. 2(c) and (d)). After 240 h, the cellular precipitates were dominant in the specimen.

Fig. 3 shows the bright-field TEM images and selected area diffraction (SAD) patterns for the specimen aged for 96 h. In the low-magnification images, cellular precipitates of approximately 30 μm in size can be observed, as marked by a solid circle in Fig. 3(a). The SAD pattern from the cellular precipitates contained reflections from an orthorhombic structure assigned to β-Cu₄Ti, together with the fcc structure of the matrix phase [8]. The orientation relationship between the matrix and the β-Cu₄Ti phases is (111)ₘ//(010)ₜ, [110]ₘ//[001]ₜ. At higher magnifications (Fig. 3(b)), modulated contrasts with lengths of 50 nm to 100 nm extended preferentially in the <001> direction of the matrix, as Table 1 - Dissolution of Cu–4 mol% Ti alloys in various solutions and existence of insoluble residues.

<table>
<thead>
<tr>
<th>Solution</th>
<th>Submerging condition</th>
<th>Dissolution of Cu–Ti alloys</th>
<th>Insoluble residue</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₂SO₄</td>
<td>9.0 M</td>
<td>298 (R.T.) 60</td>
<td>Insoluble</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% H₂O₂</td>
<td>298 (R.T.) 180</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% HNO₃</td>
<td>298 (R.T.) 60</td>
<td>Insoluble</td>
</tr>
<tr>
<td>HCl</td>
<td>6.0 M</td>
<td>298 (R.T.) 60</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% H₂O₂</td>
<td>298 (R.T.) 180</td>
<td>Insoluble</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% HNO₃</td>
<td>298 (R.T.) 60</td>
<td>Insoluble</td>
</tr>
<tr>
<td>CH₃COOH</td>
<td>17.5 M</td>
<td>298 (R.T.) 60</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% H₂O₂</td>
<td>298 (R.T.) 180</td>
<td>Insoluble</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% HNO₃</td>
<td>298 (R.T.) 60</td>
<td>Insoluble</td>
</tr>
<tr>
<td>HNO₃</td>
<td>7.0 M</td>
<td>298 (R.T.) 180</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% H₂O₂</td>
<td>298 (R.T.) 180</td>
<td>Insoluble</td>
</tr>
<tr>
<td></td>
<td>+1 vol.% HNO₃</td>
<td>298 (R.T.) 180</td>
<td>Insoluble</td>
</tr>
<tr>
<td></td>
<td>4.7 M</td>
<td>273 45</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>3.5 M</td>
<td>273 90</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>2.8 M</td>
<td>273 300</td>
<td>Dissolved</td>
</tr>
<tr>
<td></td>
<td>1.4 M</td>
<td>273 1440</td>
<td>Reacted but still present</td>
</tr>
</tbody>
</table>

Fig. 2 – FE-SEM images of Cu–4 mol% Ti alloys aged at 723 K for (a) 0 h, (b) 12 h, (c) 96 h, and (d) 240 h.
can be seen. These contrasts are caused by the needle-shaped precipitates of $\alpha$-Cu$_4$Ti, which was also confirmed by the SAD pattern: weak reflections from $\alpha$-Cu$_4$Ti were detected, as marked by the dotted circles, together with fundamental reflections from the Cu solid solution with an fcc structure[2,6]. The orientation relationship between the matrix and the $\alpha$-Cu$_4$Ti phases is $\{001\}_m//\{001\}_\alpha$, $(310)_m//(100)_\alpha$[2-4].

Fig. 4(a) shows the XRD profile of the specimen aged for 96 h. The XRD profile exhibited peaks from the Cu solid solution and $\beta$-Cu$_4$Ti phases, but no peaks from the $\alpha$-Cu$_4$Ti phase because of its small volume fraction. The lattice parameter of the Cu solid solution matrix phase was 0.3619 nm, as calculated by extrapolating the measured values obtained from the peaks to $\theta = 90^\circ$ using the Nelson-Riley function [21]. This value is between the lattice parameters of pure copper, 0.3615 nm, and solid-treated Cu–4 mol% Ti, 0.3635 nm [22], indicating that the concentration of the solute Ti in the matrix phase was lower than 4 mol% because of precipitate formation during aging for 96 h. According to Vegard’s law, the concentration of solute Ti in the matrix phase was estimated to be (0.8 ± 0.3) mol% [22].

3.2. Extraction of Precipitates From Aged Cu–Ti Alloys

One main purpose of this study was to find appropriate conditions for extraction of both the metastable $\alpha$-Cu$_4$Ti and stable $\beta$-Cu$_4$Ti precipitates from age-hardenable Cu–Ti alloys. Therefore, we focus on the specimen aged for 96 h in the following sections because it appeared to contain significant amounts of both precipitates, as shown in Fig. 2(c). In order to survey the chemical dissolution for extraction of precipitates from aged Cu–Ti alloys, we submerged the aged specimens in the various solutions listed in Table 1. The specimens were not soluble in solutions of 9.0 M sulfuric acid, 6.0 M hydrochloric acid, and 17.5 M acetic acid at 298 K and 373 K. When approximately 1 vol.% hydrogen peroxide or nitric acid was added as an oxidation agent to these three acids, the specimens dissolved gradually, although no insoluble residues of the precipitate particles remained after 180 min. In the solutions of 7.0 M nitric acid at 298 K and 273 K, the specimen gently reacted, producing bubbling, and spontaneously strewed black insoluble residue from its surface. Eventually, the specimen dissolved after 20 min, leaving some insoluble residue. When the concentration of nitric acid was decreased to 2.8 M, the time needed to dissolve the specimen increased to 300 min. In the 1.4 M nitric acid solution, the specimen reacted slowly, but did not completely dissolve even after 1440 min.

Fig. 5 shows the FE-SEM and TEM images of the specimen submerged in a solution of 7.0 M nitric acid at 273 K for 30 s and 20 min. The specimen submerged in the solution for 30 s had not yet dissolved but was deeply etched: the Cu layers in
the cellular particles had apparently disappeared and the outlines of needle-shaped particles were visible (Fig. 5(a)). In the insoluble powder residue extracted from the specimen that had been immersed in the solution for 20 min, we found two types of particles: particles constructed of laminated plates of approximately 30 μm in size (Fig. 5(b)) and fine needle-shaped particles of 50 to 100 nm in length (Fig. 5(c)). According to the FE-SEM and TEM images shown in Figs. 2(c) and 3, the former correspond to cellular precipitates of β-Cu₄Ti, and the latter, to α-Cu₄Ti. The sizes and shapes of the precipitates seemed to be similar before and after dissolution.

Fig. 4(b) shows the XRD profile of the insoluble residue extracted from the specimen that was submerged in a solution of 7.0 M nitric acid at 273 K for 20 min. We detected no peaks indexed to copper, indicating that the entire Cu solid solution matrix phase was dissolved in the nitric acid solution. The XRD profile exhibited peaks identified as corresponding to α-Cu₄Ti and β-Cu₄Ti. Fig. 4(b) also shows a fitting pattern obtained from Rietveld analysis of α-Cu₄Ti and β-Cu₄Ti compounds [4,8] and the difference between the measured and Rietveld analysis intensities, which suggest that the calculated pattern of α-Cu₄Ti and β-Cu₄Ti fits the measured pattern well. This is supported by the fact that the indicator of accuracy for Rietveld fitting, S, was 1.27, which indicates that the Rietveld analysis had a high reliability [23]. Table 2 summarizes the lattice parameters and mass fractions of the α-Cu₄Ti and β-Cu₄Ti precipitates from the insoluble residue estimated by Rietveld fitting. The lattice parameters of α-Cu₄Ti and β-Cu₄Ti in the insoluble residue were consistent with those reported in the literature within the experimental accuracy (a = 0.587 nm and c = 0.365 nm for α-Cu₄Ti; a = 0.4528 nm, b = 0.4345 nm, and c = 1.2932 nm for β-Cu₄Ti) [4,8]. The mass fraction of α-Cu₄Ti was estimated to be much smaller than that of β-Cu₄Ti, which seems to be consistent with the microstructure shown in Fig. 2(c). It is therefore confirmed that the constituents of the insoluble residue extracted from the specimen were identical to the precipitates after aging at 723 K for 96 h. Thus, we conclude that both α-Cu₄Ti and β-Cu₄Ti precipitates could be chemically extracted from the specimen by a solution of nitric acid.

Fig. 4 – X-ray diffraction patterns of (a) the Cu-4 mol% Ti alloys aged at 723 K for 96 h and (b) the insoluble residue separated from the alloy by dissolution in 7.0 M nitric acid at 273 K for 20 min. The measured patterns and the patterns calculated using Rietveld analysis are shown with the solid line and open circles, respectively. The difference between the measured and calculated intensities is indicated at the bottom.

Fig. 5 – (a) FE-SEM image of the Cu-4 mol% Ti alloy specimen aged at 723 K for 96 h, and then submerged in 7.0 M nitric acid solution for 30 s. (b) FE-SEM and (c) TEM images showing the insoluble residue separated from the specimen by submersion in the solution for 20 min.
3.3. Quantitative Analysis of Precipitates and Matrix

In order to adequately evaluate the chemical compositions and amounts of both the precipitates and the matrix phase in the specimen, it is necessary to understand the impact of the various extraction conditions, such as the temperature, time, and concentration of nitric acid solution, on the solubility of the Cu–Ti alloys. Fig. 6(a) shows the mass fractions of the Cu and Ti elements in the insoluble residue and in the filtrate for the specimen aged at 723 K for 96 h after it was submerged in 7.0 M nitric acid solution at 298 K and 273 K for 20 min. Here, we denote these mass fractions as \( M_{\text{Cu}r} \), \( M_{\text{Ti}r} \), \( M_{\text{Cu}f} \), and \( M_{\text{Ti}f} \), respectively. In addition, Fig. 6(b) shows the concentrations of Ti in the insoluble residue and in the filtrate, \( C_{\text{Ti}r} \) and \( C_{\text{Ti}f} \), which were calculated as \( M_{\text{Ti}r} / (M_{\text{Cu}r} + M_{\text{Ti}r}) \) and \( M_{\text{Ti}f} / (M_{\text{Cu}f} + M_{\text{Ti}f}) \), respectively. The total mass fraction of Cu and Ti elements in the insoluble residue and filtrate, \( M_{\text{Cu}r} + M_{\text{Ti}r} + M_{\text{Cu}f} + M_{\text{Ti}f} \), was 99.66 ± 0.39 mass% at 298 K and 100.06 ± 0.38 mass% at 273 K. These values are very close to 100 mass%, suggesting that the chemical extraction process correctly accounted for all Cu and Ti elements in the specimen. The combined mass fraction \( M_{\text{Cu}r} + M_{\text{Ti}r} \) in the insoluble residue at 273 K was larger than that at 298 K (lower plot in Fig. 6(a)). In addition, the concentration of Ti in the filtrate, \( C_{\text{Ti}f} \), at 273 K was smaller than the value at 298 K (upper plot in Fig. 6(b)), because the dissolution of the Ti-rich \( \alpha \)-Cu4Ti and \( \beta \)-Cu4Ti precipitates was suppressed at the lower temperature of 273 K.

Fig. 7 shows the values of (a) \( M_{\text{Cu}r} \), \( M_{\text{Ti}r} \), \( M_{\text{Cu}f} \), and \( M_{\text{Ti}f} \) after dissolution by solutions of 2.8 M to 7.0 M nitric acid at 273 K. As mentioned above, the separation was conducted for as long as it looks for the dissolution of the matrix to be completed: 20 min in 7.0 M nitric acid, 45 min in 4.7 M nitric acid, 90 min in 3.5 M nitric acid, and 300 min in 2.8 M nitric acid. When the specimen was dissolved in a solution with a lower concentration of nitric acid, the mass fraction of the insoluble residue \( M_{\text{Cu}r} + M_{\text{Ti}r} \) was lower (lower plot in Fig. 7(a)), and the concentration of Ti in the filtrate \( C_{\text{Ti}f} \) was higher (upper plot in Fig. 7(b)). This suggests that the Ti-rich \( \alpha \)-Cu4Ti and \( \beta \)-Cu4Ti precipitates also dissolved more slowly, like the Cu solid solution, during the prolonged dissolving time, even though the concentration of the nitric acid solution was lower.

Fig. 8 shows the values of (a) \( M_{\text{Cu}r} \) and \( M_{\text{Cu}f} \) for the insoluble residue obtained from the specimen submerged in a solution of 7.0 M nitric acid at 273 K for various dissolving times. After the specimen had been submerged in the solution for 20 min, the mass fraction of Cu and Ti elements in the insoluble residue, \( M_{\text{Cu}r} + M_{\text{Ti}r} \), gradually decreased with dissolving time, because a portion of the precipitates was already dissolved. In summary, in order to separate the precipitates and the matrix from the specimen into the insoluble residue and the filtrate, the chemical dissolution should be carried out using a solution of nitric acid with a high concentration of 7.0 M, at a low temperature of 273 K, for a short dissolving period equivalent to the minimum amount of time needed for the matrix to disappear (20 min).

Since we could separate the precipitates and the matrix phase from the specimen using this extraction procedure, the chemical compositions and mass fractions of both phases could be analyzed directly by performing ICP-OES measurements. Table 3 lists the results for the Cu–4 mol% Ti alloys aged for 96 h. The concentrations of Ti in the precipitates (\( C_{\text{Ti}} \)) and matrix (\( C_{\text{Ti}r} \)) were found to be 21.7 ± 0.3 mol% and 0.8 ± 0.2 mol%, respectively.

<table>
<thead>
<tr>
<th>Table 2 – Constituent phases of the insoluble residue obtained from the Cu–4 mol% Ti alloy specimen aged at 723 K for 96 h; their lattice parameters and mass fractions analyzed by Rietveld fitting.</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha )-Cu4Ti (MoNi4 type, I 4/m)</td>
</tr>
<tr>
<td>Lattice constant (nm)</td>
</tr>
<tr>
<td>( a (=b) )</td>
</tr>
<tr>
<td>0.5864 ± 0.002</td>
</tr>
<tr>
<td>( \beta )-Cu4Ti (Au4Zr type, P nma)</td>
</tr>
<tr>
<td>Lattice constant (nm)</td>
</tr>
<tr>
<td>( a )</td>
</tr>
<tr>
<td>0.4523 ± 0.002</td>
</tr>
</tbody>
</table>
respectively. It should be noted that the value of $C_{Ti}$ is almost constant and independent of the dissolving temperature, time, and concentration of the nitric acid solution (Figs. 6(b) to 8(b)). This is because the precipitates are dissolved homogeneously by the solutions. The Ti concentration of the matrix phase $C_{Ti}$ is very close to that of the Cu solid solution estimated from the XRD

Fig. 7 – (a) Mass fractions measured using the ICP-OES method of the Cu and Ti elements in the insoluble residue and filtrate separated from the aged Cu–4 mol% Ti alloys by dissolution in nitric acid with a concentration from 2.8 M to 7.0 M at 273 K. (b) Concentration of Ti in the insoluble residue and filtrate.

Fig. 8 – (a) Mass fractions measured using the ICP-OES method of the Cu and Ti elements in the insoluble residue separated from the aged Cu–4 mol% Ti alloys by dissolution in 7.0 M nitric acid at 273 K. (b) Concentration of Ti in the insoluble residue.
analysis, Cu–(0.8 ± 0.3) mol% Ti, as mentioned at the end of Section 3.1. This also supports the conclusion that the matrix phase is preferentially dissolved by the solution and that the dissolution of the Ti-rich precipitates barely progresses. The total mass fraction of the α-Cu4Ti and β-Cu4Ti precipitates in the specimen aged for 96 h was determined to be 14.0 mass%. Furthermore, the mass fractions of the α-Cu4Ti and β-Cu4Ti precipitates in the specimen were estimated to be 1.2 mass% and 12.8 mass%, respectively, using the ratio obtained from Rietveld fitting listed in Table 2.

Thus, extraction of precipitates from age-hardenable Cu–Ti alloys makes it possible not only to confirm the structure of the precipitates directly, but also to quantitatively evaluate the chemical compositions and mass fractions of both the constituent precipitates. Therefore, further research on qualitative and quantitative analyses of age-hardenable Cu–Ti alloys based on the present method, which will be published in the future, will clarify the precipitation behavior.

4. Conclusion

In this study, an extraction procedure for age-hardenable Cu–Ti alloys was established to directly evaluate the structure of the precipitates and to analyze the mass fractions and chemical compositions of the precipitates and matrix. Dissolution using 7.0 M nitric acid solution at a low temperature of 273 K for 20 min, which was the minimum amount of time needed to dissolve the Cu solid solution matrix, was critical for the extraction of precipitates from the alloys. For example, by dissolving the Cu–4 mol% Ti specimen aged at 723 K for 96 h in the solution, we could obtain both the α-Cu4Ti and β-Cu4Ti precipitates as an insoluble residue. The shapes and sizes of the precipitates were not significantly different before and after dissolution, implying that the precipitates had high-isotropic corrosion resistance against the 7.0 M nitric acid in comparison to the Cu solid solution matrix. The chemical compositions and mass fractions of the precipitates and matrix phase could be analyzed with high accuracy by performing ICP-OES measurements of the insoluble residue and filtrate, respectively. Therefore, the extraction method used in this study is a promising technique that can shed light on the precipitation behavior of age-hardenable Cu–Ti alloys.

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