EXAFS study on Fe precipitates in CuFe alloys induced by the thermal aging

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1 Introduction
The extended X-ray absorption fine structure (EXAFS) measurement is a useful tool for the observation of local atomic arrangements around selected atoms. Furthermore, this measurement is nondestructive testing, which is different from TEM observations or atom probe tomography methods. And we can obtain the averaged information for the whole of specimens.

By using EXAFS measurements, we observed the local structure around Fe atoms in CuFe alloys. Fe precipitates in Cu matrix are interesting as granular magnetic materials, if they exist as magnetic precipitates (α-Fe) in non-magnetic matrix (Cu). Fe precipitates are also expected to act as strong obstacles against dislocation motions. Therefore, if they are produced in Cu matrix, we will obtain a material which has a large hardness, but remains a good electrical conductivity. Then, we performed thermal aging on CuFe alloys to produce Fe precipitates in Cu matrix. In this report, we show the results of EXAFS measurements and the Vickers hardness measurements for CuFe alloys.

2 Experiment
We prepared specimens of supersaturated Cu-3at.%Fe alloy by the following methods. Starting raw materials were pure Cu and pure Fe the atomic ratio of which was 97 : 3. They were smelted by the high frequency induction melting method. The resulting ingots were rolled into sheets about 1 mm thick at room temperature. As a solution-treatment, the sheets were annealed at 1273 K for 1 hour, and then were quenched to iced-water. This solution treatment ensured a homogeneous distribution of Fe atoms in Cu matrix.

Solution-treated specimens were thermally aged at 973 K or 873 K for various time intervals (10 minutes - 92 hours) in argon atmosphere. After this thermal aging, we performed the micro Vickers hardness measurements. The hardness for the solution-treated specimen was also measured. The applied load for the Vickers hardness measurement was 200 gf.

To observe the local structures around Fe atoms in Cu matrix of the CuFe specimens, we performed the EXAFS measurements around the Fe K absorption edge (7.11 keV) at the 27B beamline of the synchrotron radiation facility of High Energy Accelerator Research Organization (KEK-PF). The EXAFS spectra were obtained using a 7 element germanium detector by the fluorescence method at room temperature. As a reference data, the EXAFS spectrum for the pure Fe foil 5 μm thick was obtained by the transmission method. We used the computer software, WinXas [1], for analyzing the obtained EXAFS spectra. In the analyses, all EXAFS spectra were Fourier transformed using k³ weighting with the k range from 2 - 3 to 11 - 15 Å⁻¹. Besides, EXAFS simulations were performed using the computer code, FEFF [2]. The simulation was calculated for the state of Fe such as the case of a Fe atom surrounded by Cu fcc lattice for the 1 - 5th nearest neighbor Cu atoms of a Fe atom.

3 Results and Discussion
Fig. 1 shows the Vickers hardness change for the Cu-3at.%Fe thermally aged at 973 K as a function of aging time. The Vickers hardness increases, and reaches a maximum value after 10 minutes aging, and then decreases with increasing the aging time. The age-hardening curve was considered as due to the appearance of Fe precipitates and their growth in the specimen.

Fig. 1 Vickers hardness change for the Cu-3at.%Fe thermally aged at 973 K as a function of aging time.

Fig. 2 shows the Vickers hardness change for the Cu-3at.%Fe thermally aged at 873 K as a function of aging time. The age-hardening curve was also considered as due to the Fe precipitations and growth of precipitates in the specimens. Besides, for specimens thermally aged at 873 K, the maximum value of the Vickers hardness is observed at the aging time for 3 hours, which is much longer than for specimens thermally aged at 973 K. Therefore, Fe precipitates were also produced by 873K
aging in Cu matrix, but the growth rate for precipitates is slower by the thermal aging at 873 K than that at 973 K.

![Fig. 2 Vickers hardness change for the Cu-3at.%Fe thermally aged at 873 K as a function of aging time.](image)

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Fig. 3 shows the Fourier Transformed (FT) EXAFS spectra near Fe K absorption edge for the specimens which were thermally aged at 973K. For comparison, the spectra for the solution-treated specimen, the pure Fe foil, and the simulated one by using FEFF code are also shown. The shape of the experimental FT EXAFS spectrum for the solution-treated specimen is similar to the simulation spectrum by using FEFF code. This result implies that Fe atoms are well dispersed in Cu matrix, and each Fe atom is surrounded by Cu atoms with the fcc structure. The shape of the experimental FT spectrum for the specimen thermally aged at 973 K for 5 hours is also similar to the simulation spectrum by using FEFF code.

![Fig. 3 FT EXAFS spectra near Fe K absorption edge for the specimens thermally aged at 973 K.](image)

Fig. 3 FT EXAFS spectra near Fe K absorption edge for the specimens thermally aged at 973 K. For comparison, the spectra for the solution-treated specimen, the pure Fe foil, and the simulated one by using FEFF code are also shown.

On the other hand, the shape of the experimental FT spectrum for the specimen thermally aged at 973 K for 72 hours is similar to that for the pure Fe foil. This result suggests that Fe precipitates in the specimen thermally aged at 973 K for 72 hours did not transform from the fcc structure to the intrinsic bcc structure. This is because Fe atoms diffuse in Cu matrix much more slowly by the thermal aging at 873 K than at 973 K. Therefore, Fe precipitates hardly grow enough to transform to the bcc structure.
Fig. 4 FT EXAFS spectra near Fe K absorption edge for the specimens thermally aged at 873 K. For comparison, the spectra for the solution-treated specimen, the pure Fe foil, and simulated one by using FEFF code are also shown.

References

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