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EXAFS study on Fe precipitates in CuFe alloys induced by thermal aging and plastic deformation

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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. In our previous study [1], we have observed the local structure around Fe atoms in Cu-3at.%Fe alloys and investigated the relationship between micro structure around Fe atoms in Cu matrix and hardness by using EXAFS measurements and Vickers hardness measurements. In addition to these results, in this report, we have investigated the effects of Fe precipitations on magnetic properties, and deformation, and then studied the relationship between change in these properties and micro structure around Fe atoms, by means of EXAFS measurements.

2 Experiment

We prepared specimens of supersaturated Cu-1at.%Fe alloy by the following methods. Starting raw materials were pure Cu and pure Fe the atomic ratio of which was 99 : 1. They were melted by the high frequency induction melting method. The resulting ingots were cold-rolled into sheets about 1 mm thick. As a solution-treatment, the sheets were annealed at 1273 K for 1 hour, and they were quenched into iced-water. This solution treatment ensured a homogeneous distribution of Fe atoms in Cu matrix.

Solution-treated specimens were thermally aged at 873 K for various time intervals (1 - 120 hours) in argon atmosphere. After the thermal-aging, we performed the micro Vickers hardness measurements at room temperature. The hardness for the solution-treated specimen was also measured. The applied load for the Vickers hardness measurement was 100 gf.

The magnetic properties for the solution-treated specimens and the thermally aged specimens were investigated by using a SQUID magnetometer. In this measurement, the applied magnetic field was -5 T to 5T, and the measurements were performed at 300K.

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 a pure Fe foil 5 μm thick was obtained by the transmission method. We used the computer software, WinXas[2] for analyzing the obtained EXAFS spectra. In the analysis, all EXAFS spectra were Fourier transformed using k^3 weighting with the k range from 2 - 3 to 11 - 15 $Å^{-1}$. Besides, EXAFS simulations were performed using the computer code, FEFF [3]. The simulation was calculated for the state of Fe such as the case of an Fe atom surrounded by Cu fcc lattice for the 1 - 5th nearest neighbor Cu atoms of an Fe atom.

Some thermally aged specimens and solution treated specimen were cold-rolled at the reduction of 70 %, and investigated the effect of the deformation by using a SQUID magnetometer and EXAFS measurements.

3 Results and Discussion

Fig. 1 shows the Vickers hardness change for the Cu-1at.%Fe themally aged at 873 K as a function of aging time. The Vickers hardness increases, and reaches a maximum value after 30 hour-aging, and then decreases with increasing the aging time. The age-hardnening curve was considered as due to the appearance of Fe presipitations and their growth in the specimen.

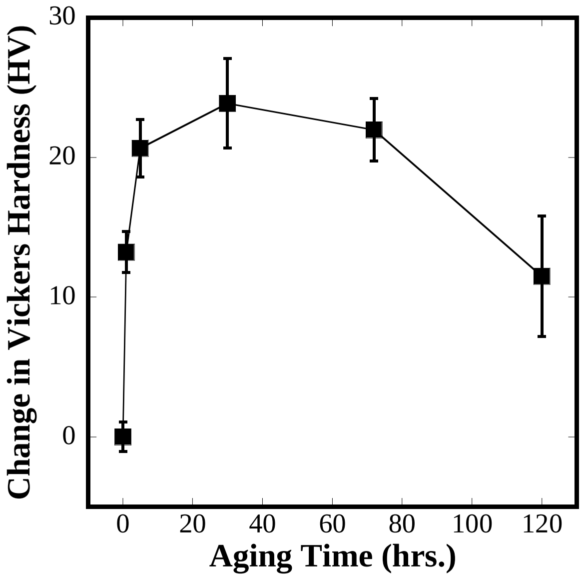

Fig. 1 Vickers hardness change for the Cu-1at.%Fe themally aged at 873 K as a function of aging time.

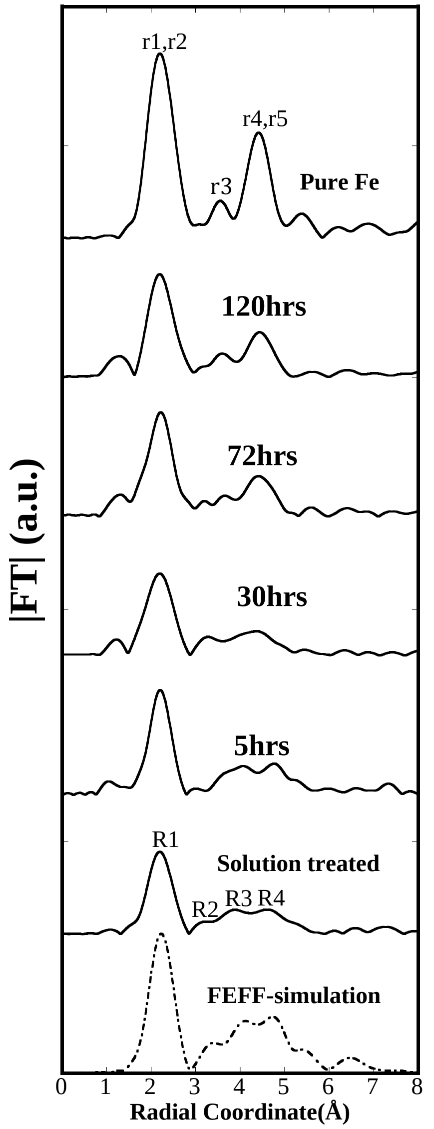

Fig. 2 FT EXAFS spectra near Fe K absorption edge for the specimens thermally aged at 873 K. For comparison, the spectra for the solution-treated specimen, the pure Fe foil, and the simulated one by using FEFF code are also shown.

Fig. 2 shows the Fourier transformed (FT) EXAFS spectra near Fe K absorption edge for the specimens which were thermally aged at 873K. 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 spectra for the specimens thermally aged at 873 K for 5 or 30 hours are also similar to the simulation spectrum by using FEFF code. However, as can be seen in Fig. 1, in the beginning of Fe precipitations for CuFe specimens, the Fe precipitates mainly have the fcc structure as a result of coherent growth in fcc Cu matrix. Therefore, the results of the hardness and the EXAFS measurements indicate that Fe precipitates with the fcc structure were produced. On the other hand, the shape of the experimental FT spectra for the specimen thermally aged at 873 K for 72 or 120 hours are similar to that for the pure Fe foil. This result suggests that the structure of Fe precipitates gradually changes from the fcc structure to the intrinsic bcc structure with increasing the thermal aging time and therefore with the growth of Fe precipitates.

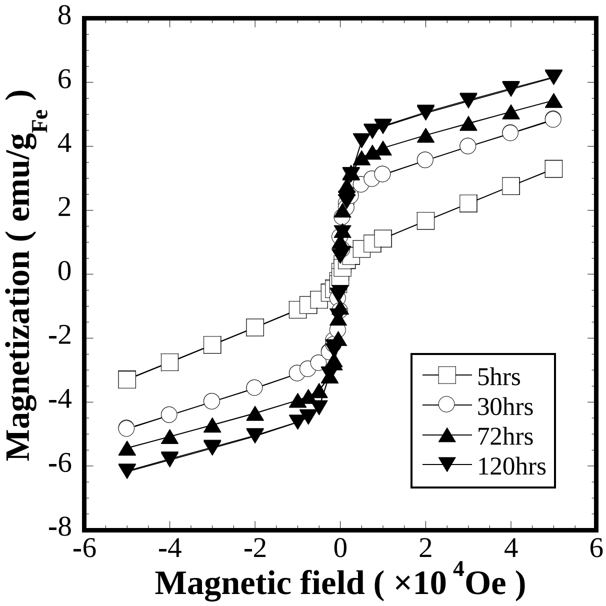

Fig. 3 Magnetization for the thermally aged specimens at 873 K as a function of magnetic field.

Then, we discuss the change in magnetization induced by thermal-aging. Since magnetic Fe atoms were homogeneously distributed in non-magnetic Cu matrix, the solution treated specimen shows the paramagnetism. Fig. 3 shows the magnetization for the thermally aged specimens as a function of magnetic field at 300K. All the thermally aged specimens show the ferromagnetism. The magnetization for the thermally aged specimens is increased with increasing the aging time. Since the amount of ferromagnetic Fe precipitates which have bcc structure were increased with increasing the aging time, the shapes of FT-EXAFS spectra for the sufficiently longtime-aged specimens which were aged for 72, 120 hours were similar to that for pure Fe foil (Fig. 2).

In order to investigate the effects of deformation on magnetic properties and the atomic structures of Fe precipitates, the 30 hour-aged specimen and solution-treated specimen were cold-rolled by the reduction of 70 %. Fig. 4 shows the FT-EXAFS spectra for the specimens which were cold-rolled after 30 hour-aging or after solution-treatment. For comparison, the spectra for the solution-treated specimen, and the pure Fe foil, are also shown. As mentioned above, in the 30 hour-aged specimen, Fe precipitates were produced by thermal-aging, and have mainly fcc structure. On the other hand, the shape of the FT-EXAFS spectrum for the specimen which was cold-rolled after 30 hour-aging was similar to that for the pure Fe foil. Furthermore, the FT-EXFAS spectrum for the solution-treated specimen was hardly changed by cold rolling. These results imply that Fe precipitates in the 30 hour-aged specimen which had mainly fcc structure, were transformed from fcc structure to intrinsic bcc structure by the deformation (cold-rolling). The shape of FT-EXAFS spectrum for the cold-rolled specimen after solution-treatment, whose Fe atoms were homogeniously distributed in the Cu fcc matrix, was hardly changed from that for solution-treated one.

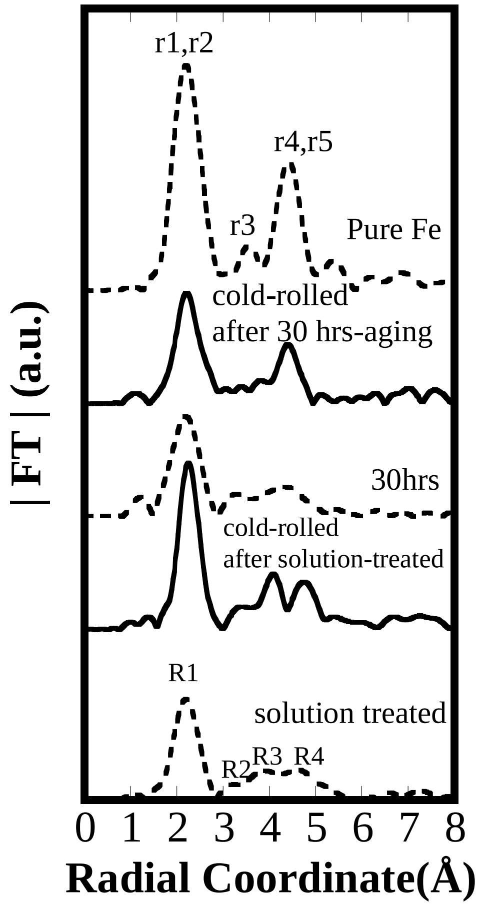

Fig. 4 FT EXAFS spectra near Fe K absorption edge for the specimens thermally aged at 873 K, and cold-rolled ones. For comparison, the spectra for the solution-treated specimen, and the pure Fe foil, are also shown.

After cold-rolling, the magnetization for solution-treated specimen was not changed from that one before cold-rolling. Fig. 5 shows the magnetization for the specimen which was thermally aged for 30 hours and the cold-rolled one after 30 hour-aging as a function of magnetic field at 300K. Since there were a large amount of non-magnetic Fe precipitates with fcc structure, the magnetization for the specimen which was thermally aged for 30 hours were very small. On the other hand, the cold-rolled specimen after 30 hour-aging has a huge magnetization. This experimental result can be explained as follows. Since a large amount of non-magnetic Fe precipitates with fcc structure were transformed to bcc structure by the deformation (the cold-rolling), the magnetization was greatly increased. This result confirms the EXAFS result which is shown in Fig.4.

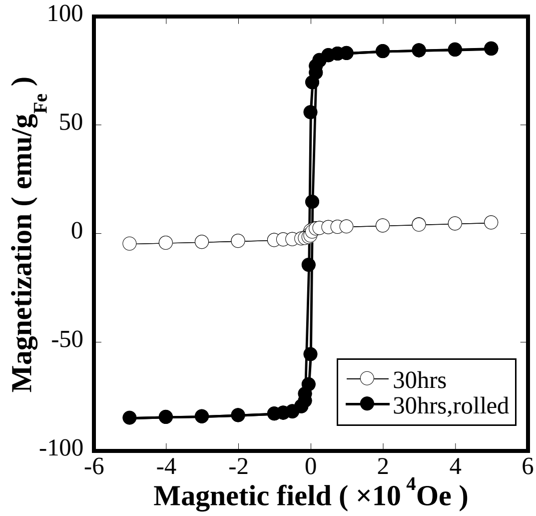

Fig. 5 Magnetization-magnetic field curves as for the specimen which was only thermally aged for 30 hours at 873 K and for the specimen which was cold-rolled after 30 hour-aging at 873 K.

References
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