実験報告書様式（一般利用課題・成果公開利用）

（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。）

MLF Experimental Report

提出日 Date of Report

13rd February, 2018

課題番号 Project No.

2017B0129

実験課題名 Title of experiment

Simultaneous investigation of nanoscale precipitates and recovery in thermally aged Fe-Cu alloy and their comparison with magnetic hysteresis

実験責任者名 Name of principal investigator

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装置名 Name of Instrument/(BL No.)

TAIKAN (BL-15)

実施日 Date of Experiment

18th Dec.-19th Dec., 2017 (36h)

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）

Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

- Samples: Iron-Copper alloy (Fe-1wt.%Cu)
- Samples were cold-rolled with rolling reduction (RR) up to 20% (0, 10, and 20%).
- Samples with dimensions of 15 x 15 x 2 mm were aged at 480 ^{o}C. For each RR, 9 samples with different aging time up to 10000min were prepared as listed in Table1.

		Aging time (min.)								
Sample	Rolling reduction (%)	0	10	50	100	500	1000	2000	5000	10000
Fe-1wt%Cu	0	○	○	○	○	○	○	○	○	○
	10	○	○	○	○	○	○	○	○	○
	20	○	○	○	○	○	○	○	○	○

Table 1: Measuring samples (Totally 27 samples)

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Small angle and wide angle neutron scattering measurements were carried out at room temperature. Horizontal magnetic field of 1 Tesla was applied with an electromagnet. We observed strong magnetic contribution for small-angle neutron scattering for all measured samples, which are reflected in butterfly patterns of scattering intensity in the 2D detector. According to the equation of SANS intensity, $I(\mathbf{Q}) = I(\mathbf{Q})_{nuc}+I(\mathbf{Q})_{mag}\sin^2\theta$, the magnetic scattering intensity, $I(\mathbf{Q})_{mag}$ was extracted from the butterfly intensity patterns. Here, θ is the angle between the applied magnetic field and scattering vector. $I(\mathbf{Q})_{nuc}$ and $I(\mathbf{Q})_{nuc}+I(\mathbf{Q})_{mag}$ were determined from scattering intensities along and perpendicular to the magnetic field, respectively, and $I(\mathbf{Q})_{mag}$ was obtained from their difference. Figure 1 shows the aging time dependence of $I(\mathbf{Q})_{mag}$ for samples with RR=0 and 20%.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Before aging, there is no significant magnetic scattering related to nanoscale features for both samples. After aging at 100min, magnetic contribution appears at Q~0.1 A^{-1} and it continuously develops with aging time up to 10000min, associated with a shift toward the lower Q. The observed $I(\mathbf{Q})_{mag}$ was analyzed using a software "Irena" and volume weighted size distribution of non-magnetic Cu precipitates was determined.

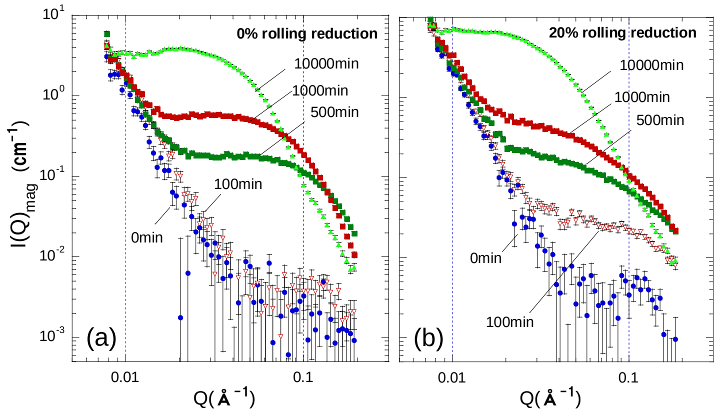

Figure 1: Aging time dependence of magnetic scattering intensity as a function of Q for (a) RR=0 and (b) RR=20% samples.

Figure 2 shows the volume-weighted size distribution f(R) for RR=0 and 20% samples. With aging time, the intensity f(R) develops, associated with a shift of the peak toward the higher radius. After aging time of 1000 min, f(R) becomes broad and the broadening is significant for RR=20% sample.

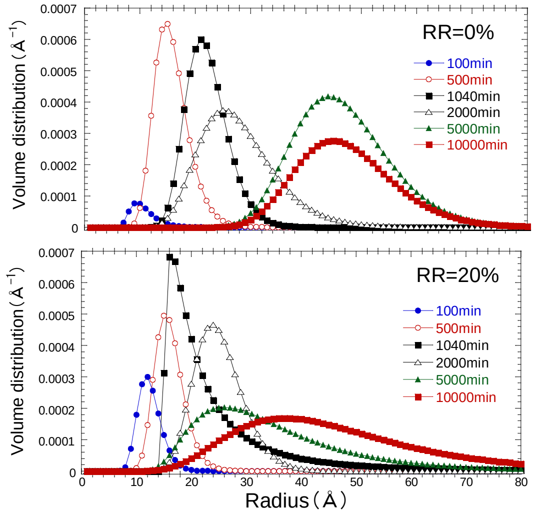

Figure 2: The aging time dependence of volume-weighted size distribution for RR=0 and 20% samples.

Figure 3 shows the aging time dependence of volume fraction and mean size of Cu precipitates for all measuring samples. For all the samples, the volume fraction steeply increases after 100 min. and attains ~ 0.6 % at 10000 min. For RR=10 and 20 % samples, the volume fraction appears just after ~ 10 min. Mean diameter also steeply increases after 100 min. and attains ~ 100 A at 10000 min. This behavior is consistent with that of hardness and the volume fraction for RR=0% after 100 min is found to be linearly proportional to coercivity of major magnetic hysteresis loop.

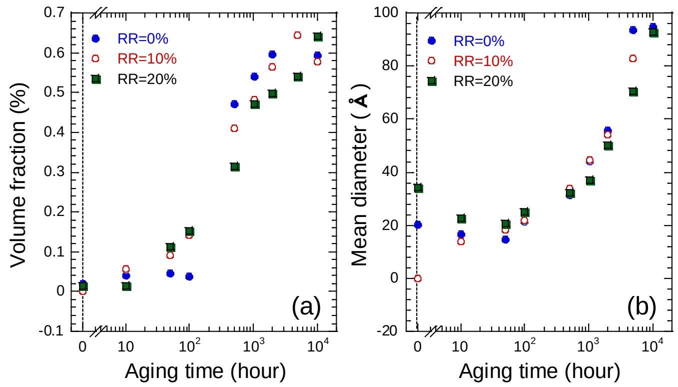

Figure 3: The aging time dependence of volume fraction and mean diameter of Cu precipitates.

For wide angle scattering data, the significant broadening of nuclear Bragg peaks was observed before aging for the RR=20 % sample. Just after aging, the peaks become narrower and the width becomes almost constant after 100 min. This behavior is very similar to the aging time dependence of coercivity for RR=20 %. The results directly demonstrate that the decrease of coercivity just after aging for RR=20 % is due to recovery such as rearrangement of dislocations.