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Characterization of Microstructure and Mechanical Properties of High-Purity Iron Added with Copper

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Abstruct An influence of the addition of copper (0.5, 1.0 and 1.5 mass% Cu) on the microstructure and mechanical properties of high purity iron (99.998 mass%) was characterized. The microstructure and microhardness of high-purity iron based samples, which were rolled at room temperature and subsequently annealed, were investigated in this work. The microstructure of the samples has been observed by electron back scattering diffraction (EBSD) and the mechanical properties have been studied by using micro-Vickers hardness test. The results of microstructural observation showed that deformation band was formed in high purity iron by rolling at room temperature, and it was recovered by annealing up to about 900 K. The microhardness results showed that the softening of high-purity iron occurred by annealing up to about 900 K, while the hardness of iron added with about 0.5-1.5 mass% copper was kept over 100 Hv and at the early time of annealing reached a maximum. The hardness of iron added with a small amount of copper may be attributed to precipitation hardening as well as solution hardening. The orientation of crystal in recrystallized grain was almost same as that of deformed grain.

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Key words: *high purity iron, iron-copper alloy, recrystallization, EBSD, Vickers hardness, annealing, precipitation*

1. Introduction

Copper is one of important alloying elements or impurity elements in steel, which should play an important role in steel products. However, an influence of the addition of copper on the microstructure and mechanical properties of steel or iron-bases alloys is still unclear, since copper is strongly interacted with other elements such as sulfur. This prompts us to characterize an influence of the addition of copper to the microstructure and mechanical properties of high purity iron. The microstructure and microhardness of high-purity iron based samples, which were rolled at room temperature and subsequently annealed, were investigated in this work. The microstructure of the samples has been observed by electron back scattering diffraction (EBSD) and the mechanical properties have been studied by using micro-Vickers hardness test.

2. Experimental

2.1 preparation of ultra high purity iron and Fe-Cu alloys

99.997 mass% ultra high purity iron(denoted as UHP-Fe) and 99.998 mass%Copper which were made by the anion-exchange separation method1) were used in this experiment.. Using both metals, button type 0.5, 1.0 and 1.5 mass% alloys were also produced by Ar-H_2 plasma arc melting. The size of the button is about 5 mm high and 50 mm diameter. Chemical composition of UHP-Fe and alloys were shown in Table 1 and Table 2, which were obtained by GDMS analysis (Glow Discharge Mass Spectrometory) after cut off about 0.5 mm from bottom of button. Concentration of Cu in each alloy was 0.46, 0.91 and 1.58 mass%, respectively.

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Table 1. GDMS analysis of purified Fe

Elements	mass ppm	Elements	mass ppm
C	< 2.6	Co	2.00
N	< 2.4	Ti	0.59
O	< 2.4	Se	0.53
Si	0.41	Mo	0.37
P	0.28	Nb	0.19
S	0.31	Ni	0.15
Mn	0.04	Sr	0.15
Cu	22.0	Cr	0.01

2.2 Preparation of specimen and roll working

After rectangular button was made by cut off fringe of a button, the test material was cut down about 4 mm thickness by using cutting machine. Both sides of sections were polished by emery papers and followed chemical polishing with H_2O_2 + HF solution.

Each test specimen was roll-worked up to 50% reduction in thickness by roll machine with cleaned surface rolls at room temperature. Regarding UHP-Fe, roll working was done up to 90% reduction. The roll-working process was done with oil free. Each specimen was cut about 5mm in length and polished with the H_2O_2 + HF solution.

2.3 Annealing, Hardness test and Observation of Microstructure

In an annealing of specimen were annealed on a boat made of the UHP-Fe with Ar + H_2 atmosphere in quartz tube. Horizon type furnace which was able to open and close a side insulating material of up-side was in order to rapid heating and cooling. Attainment time at 773 K and 1073 K were about 5 and 15 min, and 15 and 20 min to reach at room temperature, respectively. After annealing, the recrystallization behavior was investigated by the hardness and observation of microstructure.

Micro Vickers hardness test for rolled plane was performed about 10 points in the condition of 1N or 2N load. The micro Vickers hardness of as-melted and as-rolled specimens are shown in Table 2.

Table 2. Micro Vickers hardness of as-cast and as-rolled specimen

	as melted	50%reduction	90%reduction
UHP-Fe	62	98	105
0.5%Cu alloy	82	121	
1.0%Cu alloy	85	134	
1.5%Cu alloy	100	161	

Micro structure was observed by optical microscope and SEM. The crystal orientation of crystal grain was measured by EBSD.

3. Results and Discussion

3.1 Microstructure of deformed and annealed specimen

The results on microstructural observation showed that deformation band was formed in high purity iron by rolling at room temperature, and it was recovered by annealing up to about 900 K. The microhardness results showed that the softening of high-purity iron occurred by annealing up to about 900 K, while the hardness of iron added with about 0.5 mass% copper was kept over 100 Hv. The hardness of iron added with a small amount of copper may be attributed to precipitation hardening as well as solution hardening. The results on microhardness tests seem to be consistent with the data by microstructural observation.

The macro structure of as-cast button has been shown in Fig. 1. The dendrite structure is observed at surface and bottom of button, and columnar grain is also observed at the inner of button. In Fig. 2, an image by orientation image microscopy (OIM) for the [001] pole figure in as-rolled (reduction 50%) UHP iron has been shown. Many parts of all crystal planes have a [101] orientation and also deformed structures n were observed.

An OIM image of specimen reduced 50% and annealed at 873 K for 0 min (time 0 indicates an attainment to annealing temperature of sample) has

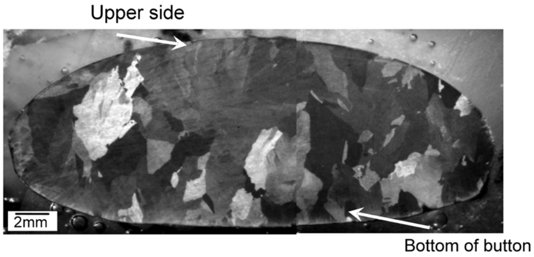

Fig. 1. Macro structure of as-cast 0.5 mass% Cu.

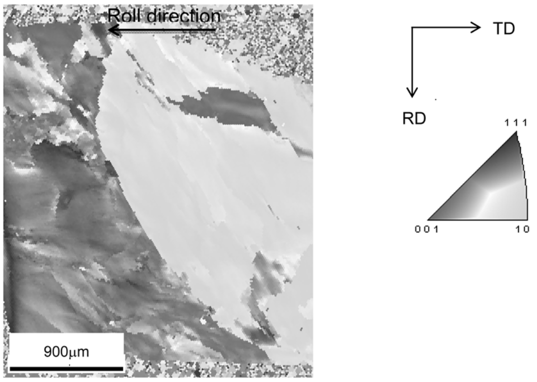

Fig. 2. OIM image of as-rolled UHF pure iron (Reduction 50%).

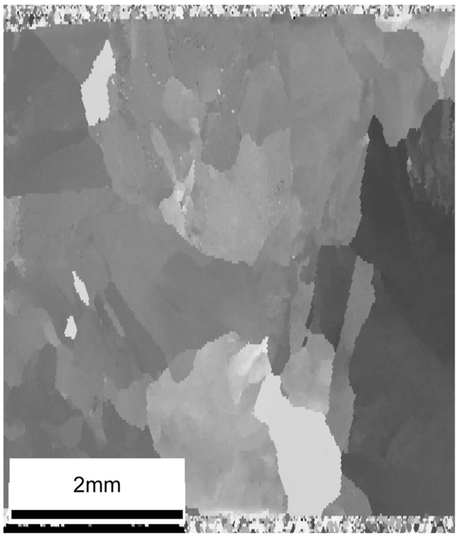

Fig. 3. OIM image of 50%reduction UHP iron annealed at 600C for 0 min.

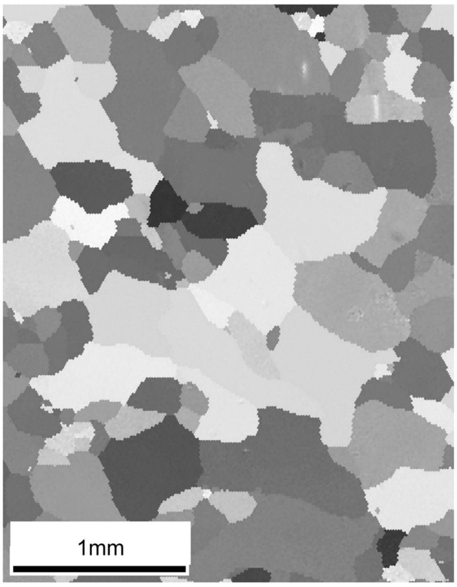

Fig. 4. OIM image of 50%reduction UHP iron annealed at 600C for 300 min.

shown in Fig. 3. It is recognized that crystal orientation shows [110] and [100] orientation. Some small size of crystal grains which is re-crystallized are observed, therefore in a period of the attainment time a recrystallization has already occurred. In Fig. 4, an OIM image for specimen which has been annealed at 873 K for 300 min has been shown. The orientations of crystal planes have various orientations. Same size big crystal grain and small crystal grain are observed, therefore it is considerable that the recrystallization process is under way. According to Abe et.al.2), the different orientations of re-crystallized grains have grown at the hot-forging process. The present result consist with that of Abe et.al.2).

3.2 Measurement of Hardness

Typical time dependence of micro Vickers hardness at 823 K is shown in Fig. 5. At beginning of annealing, the hardness of Fe-Cu alloys is higher than that of as-cast and as-rolled, and decreased with increasing time. The values of hardness are increased with concentration of Cu. On the other hand the hardness of UHP-Fe is decreased with increasing time from the beginning of annealing. After long time of annealing, the hardness number

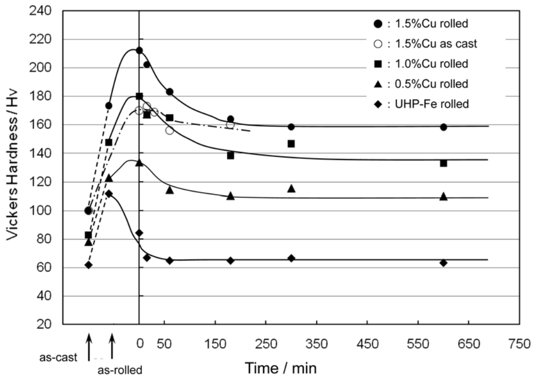

Fig. 5. Hardness versus annealing time at 873 K for Fe-Cu alloys and UHP-Fe deformed 50% and 90%, respectively.

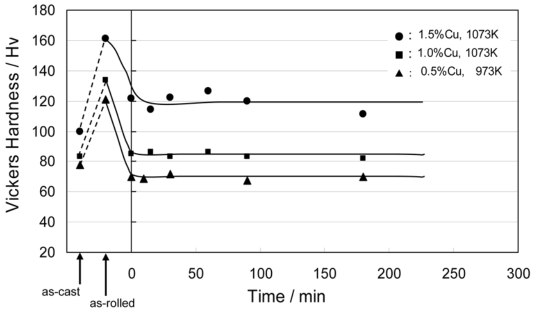

Fig. 6. Hardness versus annealing time at 973 K and 1073 K for Fe-Cu alloys deformed 50%.

of UHP-Fe becomes comparable to that of as-melted specimen. In generally, the hardness of rolled or forged metal is decreased by annealing, as shown in the UHP-Fe, and so-called the recovery and re-crystallization. According to Fe-Cu phase diagram2), in the experimental temperatures of the present work, all of the concentrations of Cu in Fe-Cu alloys samples are in the outside of solubility limit, therefore a Cu-rich phase can be precipitated. The increasing of hardness in Fe-Cu alloys at early stage of annealing is contributed with the precipitation phenomena.

In Fig. 6 it is shown the time dependence of hardness at 1073 K for UHP-Fe, Fe-1.5%Cu and Fe-1.0%Cu alloys. The hardness number is decreased with increasing annealing time. The solubility limit of these alloys is sited in inside of the solubility limit at this temperature, therefore the precipitation hardening is not occurred.

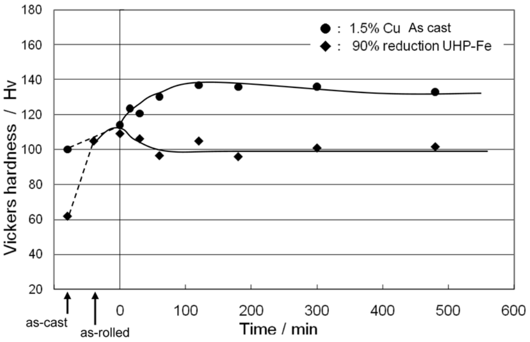

Fig. 7. Hardness versus annealing time at 653 K for 1.5% Cu as cast and UHP- Fe deformed 90%.

Typical time dependences of hardness at low temperature 653 K for 90% reduced UHP-Fe and as melted 1.5%Cu alloy are shown in Fig. 7. In UHP-Fe, the hardness decrease slightly at early stage of annealing and become a constant. It can be considered that the recovery phenomenon has occurred. On the other hand, in as melted 1.5%Cu alloy there is the increase of hardness at early stage, and after that follows constant values. The increasing of hardness is due to the precipitation of Cu rich phase.

Ogawara et.al.3) have reported that the temperature of re-crystallization for high purity iron (purity is 99.998%) is about 673 K. It is considerable that the recrystallization temperature of the present high purity iron is nearly the same temperature.

4. Conclusion

Using self-made ultra high purity iron and copper, the characterization of microstructure and mechanical properties accompanying roll-working and annealing of high-purity iron and its alloys added with copper has been investigated. The conclusions are as follows,

1. The results on microstructural observation show that deformation band is formed by rolling at room temperature, and it is recovered and recrystallized by annealing. The different orientations

of the recrystallized crystal grain is grown after annealing.

2. In both as-cast and as-rolled of alloys, in the high temperature regions where is outside of solvus of alloy, the Vickers hardness reaches a maximum at the early stage of annealing and subsequently decrease to become a constant. The increasing of hardness due to the precipitation of Cu rich phase. In a attainment process to annealing temperature, the recrystallization and precipitation phenomena have already occur.

3. In high purity iron roll-worked, the hardness is decreased with the annealing time by the process of the recovery and recrystallization, however in the lower temperature no the recrystallization behavior is observed clearly in the hardness.

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