TEM Observation of Cr Fibres in Cu–15Cr–0.5Fe In Situ Composites

Shoujun Sun, Shigeki Sakai and Hirowo G. Suzuki

Materials Processing Division, National Research Institute for Metals, Tsukuba 305-0047 Japan

Different from the continued hardening of Cr phase in Cu–15Cr in situ composites, the micro hardness of Cr phase in Cu–15Cr–0.5Fe in situ composites decreases with increasing drawing strain when drawing strain exceeds 1.6. In order to investigate the mechanism of the Cr softening caused by drawing, TEM (transmission electron microscopy) observations were made on the Cr fibre obtained at different drawing strains. It was found that the dislocation density in the deformed Cr phase in Cu–15Cr–0.5Fe in situ composites decreases with drawing strain increasing from 4.5 to 6.9, which is the result of the dynamic recovery, and the reason of the softening of Cr.

(Received December 9, 1999; In Final Form March 15, 2000)

Keywords: composites, dislocation, microstructure, recovery

1. Introduction

The Cu based in situ composites, which are reinforced by b.c.c. metals such as Fe, Cr, Ta and Nb have drawn a lot of attentions because of the excellent combination of the high strength and high electrical conductivity. The low cost and good performance make these so-called in situ composite materials more competitive in industry application than the artificial composites.

The 5 mass%Cr reinforced Cu (Cu–15Cr) in situ composites have been developed in National Research Institute for Metals in Japan for decades, the record of tensile strength of 143 MPa and conductivity of 54% IACS (International Annealed Copper Standard) has been obtained from Cu–15Cr cold drawn wires with drawing strain at 5.3, and strength of 600 MPa and conductivity of 80% IACS have been acquired after aged at 425°C for 1 h.

A new goal has been set to produce Cu–15Cr based in situ composite wire with strength higher than 1000 MPa and conductivity higher than 75% IACS. Small amount of third elements, such as Zr, Ti and C, have been added into the base alloy to achieve extra hardening, the tensile strength has been modified without a large sacrifice of conductivity.

0.5 mass%Fe has been selected as the third element to modify the properties of Cu–15Cr in situ composites in this study, the effect of drawing strain on dislocation density and micro Vickers hardness in Cr phase has been examined.

2. Experimental Procedures

The mixture of highly pure Cu, Cr and Fe pellets (the nominal composition of the mixture is 0.5 mass%Fe, 15 mass%Cr and the balance being Cu, and the chemical analysis is shown in Table 1) was molten at 1550°C and held for 3 min before cooling in the vacuum furnace. The ingot was hot forged at 1000°C with the 75% total reduction and solid solution treated at 1000°C for 1 h followed by water quenching. The billet was sliced into the required pieces, which were then rolled and drawn at room temperature with total deformation strain (referred to as drawing strain, \( \eta \), which is defined as the natural logarithmic form of the ratio of initial cross section area to the final cross section area, i.e., \( \eta = \ln(A_0/A) \)) at range of 0 to 6.9. The final diameter of the as-drawn wire is 1 mm.

The morphology of the Cr fibre has been observed with scanning electron microscopy (SEM) after the surface of the composite wire has been slightly etched in 40% diluted nitride acid. The thickness of the deformed Cr phase was measured on the SEM images, which were taken from the cross section of the as-drawn composites. The final thickness is the average of 30 measurements.

Micro Vickers hardness testing for Cr phase was carried out with the DMH-1 micro hardness testing machine with loading at 4.9N for 2s, the testing was performed on the polished longitudinal section of the as-drawn composites. The presented results are the average of 10 testings.

The deformed Cr fibres have been extracted by dissolving the cold drawn wires (the drawing strains are 4.5 and 6.9 respectively) in 40% diluted nitride acid and the substructure of the Cr fibre was observed directly with transmission electron microscope (JEM 2000FXII) with accelerating voltage at 200 KV.

3. Results and Discussion

3.1 The Effect of Drawing Strain on the Microhardness of Cr Fibre

Figure 1 shows the relationship between cold drawing strain and micro hardness of the Cr fibres in binary Cu–15Cr and ternary 15 mass%Cr and 0.5 mass%Fe reinforced Cu (Cu–15Cr–0.5Fe) in situ composites respectively. The

<table>
<thead>
<tr>
<th>Elements</th>
<th>Cr</th>
<th>Fe</th>
<th>C</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elements</td>
<td>14.3</td>
<td>0.41</td>
<td>0.0012</td>
<td>0.003</td>
<td>0.0014</td>
<td>bal.</td>
</tr>
</tbody>
</table>

Table 1 Chemical composition of Cu–15Cr–0.5Fe in situ composites.
hardness of Cr fibre in binary Cu–15Cr \textit{in situ} composites increases with drawing strain because of the cold work hardening, whereas the hardness of Cr fibre in ternary Cu–15Cr–0.5Fe \textit{in situ} composites increases with increasing drawing strain from 0 to 1.6, and decreases with increasing drawing strain from 1.6 to 6.9.

The difference of the hardness of Cr phase between Cu–15Cr and Cu–15Cr–0.5Fe \textit{in situ} composites is caused by the addition of the 0.5 mass\%Fe, the Fe content in Cr phase (2.4 mass\%, as shown in Table 2) is higher than this in the whole materials (0.5 mass\%) because the chemical potential of Fe in Cr is larger than that of Fe in Cu. The higher content of Fe in Cr phase provides solid solution hardening to Cr fibre and changes the substructure and deformation behavior of Cr fibre.

The SEM back-scattering electron (BE) and secondary electron (SE) images of Cu–15Cr–0.5Fe \textit{in situ} composites are shown in Fig. 2, the Cr phase (the darker phase) has not been deformed effectively when drawing strain is 4.5, there still are a lot of large Cr phase (indicated by A in Fig. 2(a)) remained while some have been deformed (marked by B in Fig. 2(a)). The further deformation makes Cr deform uniformly (shown in Fig. 2(b)) because of the heavier deformation and resulting in the softening of Cr phase.

### 3.2 TEM observation of Cr fibre

The variation of the hardness of Cr with drawing strain is a result of the change of the substructure of Cr fibre, because the hardness of Cr reaches its peak when drawing strain is 1.6 and decreases afterwards, it would be useful to observe the substructure of the Cr fibre at the peak hardness ($\eta = 1.6$) and

![Image](image-url)

**Table 2** Chemical composition of Cr phase extracted from Cu–15Cr–0.5Fe \textit{in situ} composites.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Cr</th>
<th>Fe</th>
<th>C</th>
<th>S</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>bal.</td>
<td>2.46</td>
<td>0.007</td>
<td>0.011</td>
<td>0.43</td>
</tr>
</tbody>
</table>

![Image](image-url)

![Image](image-url)
lowest hardness ($\eta = 6.9$). However, because the Cr fibre is too thick to be transparent to electron beam when $\eta = 1.6$, we chose the Cr fibre at $\eta = 4.5$ instead, the thickness (or diameter) of which is within the transmission electron microscopy (TEM) limit.

The substructures of Cr fibres are shown in Fig. 3, the axial direction of the Cr fibre is parallel to the (110) because the {001}{$\{110\}$ texture has been developed in bcc Cr after cold drawing. Dislocations have been produced in Cr because of the plastic deformation.

Under the Cr [001] incident electron beam, lined-up screw dislocations have been found in the Cr fibre (as shown in Figs. 3(a) and (b)), the number of dislocations in fibre drawn at 4.5 (average thickness is about 2.03 $\mu$m) is much higher than that in the fibre being drawn at 6.9 (average thickness is 1.08 $\mu$m).

The dislocation density in Cr can be obtained by counting the dislocation number in a number of TEM photographs and divided by the counting area and correlated with the thickness of the Cr fibre. The results in Cr fibre of $\eta = 4.5$ and $\eta = 6.9$ are listed in Table 3, which are the averages of at least 20 countings on 5 TEM images.

4. Analysis

4.1 Dislocation density

During cold drawing, both Cu and Cr are deformed under the joint effects of compressive stress in radial direction and tensile stress in axial direction, the work of the cold drawing will increase the boundary area and dislocation density.

During the deformation, slip takes place on the most densely packed planes and the most densely directions, which define the slip systems {110}{$\{111\}$, {112}{$\{111\}$ or {123}{$\{111\}$} in bcc metal. The slip system in Cr has been found to be {110}{$\{111\}$ at room temperature. Adachi et al.\textsuperscript{13} has proposed the formation of screw dislocations and there jogs in Cr fibre of Cu–15Cr in situ composites during cold drawing. The screw dislocation is created by the Frank-Read type source and moved by plastic deformation.

During the cold deformation, Cr slips on its preferred slip system {110}{$\{111\}$, the dislocations on different slip planes and directions meet each other and form the jogs.

The trapping of the newly formed mobile dislocations by the existing dislocations will lead to the increase of dislocation density, which results in the hardening of Cr in the binary composites.

The addition of 0.5 mass%Fe may change deformation

![TEM bright field images and the SAD patterns under Cr [001] of Cr fibres obtained from Cu–15Cr–0.5Fe in situ composites at $\eta = 4.5$ (a) and $\eta = 6.9$ (b).](image-url)
characteristics of Cr, the higher stacking fault energy of Fe makes the dynamic recovery possible during the cold deformation. At the initial stages of the deformation, the hardness (or flow stress) increase because the dislocations interact and multiply, however, the driving force (or stored energy) for recovery increases with the increasing dislocation density, the dynamic recovery occurs.

When the dislocations are unlocked, the mobile dislocations can glide on the glide plane. The edge dislocations of opposite signs on the same glide plane may annihilate by gliding towards each other, which leads to lowering the dislocation density. Dislocations of opposite Burgers vectors on different glide planes can annihilate by glide and climb, the latter requires thermal activation thus only can occur at high temperature. The annihilation of screw dislocations takes place by cross-slip, which occurs at low temperature in a materials with high stacking fault energy.

### 4.2 Relationship between dislocation density and hardness for Cr

Assume the flow stress ($\sigma$) of Cr is a function of the dislocation density ($\rho$) as follows:\(^{14}\)

$$\sigma = kG\bar{b}\rho^{1/2}$$  \hspace{1cm} (1)

where $k$ is a constant of the order of 0.5, $\bar{b}$ is the Burgers vector in Cr, and $G$ is the shear modulus of Cr, and the flow stress of Cr is also directly proportional to the hardness ($H_V$):\(^{15}\)

$$\sigma = 3.27^*H_V(0.1)^n$$  \hspace{1cm} (2)

where, $n$ is work-hardening exponent for Cr. The relationship between dislocation density and hardness for Cr can be written:

$$H_V = K G \bar{b} \rho^{1/2}(0.1)^{-n}$$  \hspace{1cm} (3)

where $K = (k/3.27)$, a constant. The relationship between hardness and dislocation density in Cr fibre at $\eta = 4.5$ and $\eta = 6.9$ can be expresses as follows:

$$\frac{H_V(\eta = 4.5)}{H_V(\eta = 6.9)} = \left(\frac{\rho(\eta = 4.5)}{\rho(\eta = 6.9)}\right)^{1/2}$$  \hspace{1cm} (4)

The ratio obtained by hardness measurement is 1.12, which agrees with the 1.13 of the ratio of square root of the dislocation density. The good agreement between the ratio of the hardness and the ratio of the square root of the dislocation density indicates that the decrease of the hardness of Cr phase with increasing drawing strain from 4.5 to 6.9 is caused by the decrease of dislocation density, which is the result of dynamic recovery of the deformed Cr due to higher work hardening and stacking fault energy by the Fe addition.

### 5. Conclusion

In contrast to continued hardening of the Cr in Cu–15Cr binary in situ composites, the softening of Cr fibre in Cu–15Cr–0.5Fe in situ composites occurs when the drawing strain is over 1.6. TEM observation of Cr fibre suggests the dislocation density in Cr fibre at higher drawing strain ($\eta = 6.9$) is lower than the dislocation density in Cr fibre at lower drawing strain ($\eta = 4.5$). The softening of Cr at higher drawing strain is attributed to the annihilation of dislocation, which results from dynamic recovery of Cr by the addition of Fe.

### REFERENCES

8) Sun Iig Hong: Scripta Mater., 39 (1998), 1685.