This paper reports on the relationship between the mechanical properties and the grain size of Cu microwires modified by Joule heating. The increase in yield strength as the grain size of a metal or alloy decreases is known as the Hall-Petch relation. Because the crystal grain size in thin metallic wires is fine, these have higher strength compared to their bulk counterparts. To improve the formability of 25 µm-thick Cu microwires, the wires were heat-treated at various temperatures by Joule heating, and the grain size of the wires was evaluated quantitatively by cross section method. Larger crystal grains grew at higher temperatures, and the wire heat-treated at the highest temperature of 600°C had a bamboo structure, in which the grain boundaries were only in the radial direction of the wire. Small-span, three-point bending tests were performed on the heat-treated Cu microwires to determine their mechanical properties. The Young’s modulus of the wires was found to be independent of grain size, with an average value of 86.4 ± 2.4 GPa. On the other hand, the yield stress of the wires clearly depended on the grain size. The yield stress of a Cu microwire that had not been subjected to Joule heating was 311 MPa, and this decreased to 75 MPa after heat treatment at 600°C. Finally, we confirmed that the Hall-Petch relation was applicable to the Cu microwires, except for those that, due to insufficient heat treatment, had crystal grain structures in which the grains were highly elongated in the axial direction of the wire.

Key words: Cu microwire, Heat treatment, Joule heating, Grain size, Yield stress

1 Introduction

Metallic micro- and nano-materials have excellent physical properties and unique geometrical features; thus, it is expected that these small-scale materials will be very important elements in future materials systems. The validity of the Hall-Petch relation, which describes the relationship between the yield strength of a metal or alloy and its grain size, has been verified for various kinds of metals through experiment1)-15) and analysis16-19). Related to this, the abovementioned small-scale materials show higher strength compared with their bulk counterparts due to the fine crystal grain structure, and therefore, it is expected that these materials will be used as high strength components in future advanced materials systems. On the other hand, the strength of a material usually has an inverse relationship with its ductility6),8), and small-scale materials with fine polycrystalline structures tend to have inferior formability compared to the bulk material.

Generally, the physical properties of metals and alloys are changed by structural modification, and heat treatment is widely used for this purpose. Because they have smaller heat capacity, the temperature of small-scale materials can easily be increased by passing a current through it, and therefore, various techniques to modify the microstructure of thin wires or films by Joule heating have been reported20)-26). Because Cu has superior formability and excellent electrical conductivity, Cu microwires are widely used in various industries, especially in the electronics industry. The physical properties of Cu microwires are sensitive to the crystalline structure27)-32), and thus, controlling the structure of the wire is a useful way of enabling the wires to be used for various purposes. It has been reported that Cu microwires can be softened by Joule heating25), and that the Cu grains grow faster by Joule heating compared with the heat treatment with a conventional furnace26).

In this paper, 25 µm thick Cu microwires were heat-treated at various temperatures by Joule heating. The heat treatment caused the growth of the crystal grains, and the relationship between the grain size and the heat treatment temperature was investigated. Furthermore, small-span, three-point bending tests were performed on the heat-treated Cu microwires, and the mechanical properties of the wires, i.e., Young’s modulus and the yield stress, were determined. Finally, we discuss whether or not the Hall-Petch relation is applicable to the Cu microwires under test in this study.

2 Temperature of a Thin Wire with a Current Passing through It

To determine the amount of current needed in a thin wire in order to heat treat it, we consider the electro-thermal
Therefore, the current required to cut the wire with Joule heating by the difference in the thermal boundary conditions between the middle of the wire. On reaching the steady state, where the temperature of the wire remains fixed with time, the maximum temperature, $T_C$, is given by:

$$T_C = \frac{1}{8K\sigma} \left( \frac{I^2 L}{A} \right)^2 + T_0,$$

where $K$ and $\sigma$ are the thermal and electrical conductivities. $L$ is the segment to which the current is supplied, and $A$ is given by $\pi d^2 / 4$. The current to cut the wire by Joule heating under the thermal boundary conditions shown in Fig. 1(a), $(I_C)_0$, can be determined analytically by equating $T_C$ with $T_M$, the melting point of the wire, and rearranging Eq. (1), as follows:

$$(I_C)_0 = \sqrt{(T_M - T_0) 8K\sigma \frac{A}{L}}.$$

On the other hand, as shown in Fig. 1(b), in a realistic situation, the temperature at each end might be greater than $T_0$, and heat transfer from the wire surface cannot be ignored. Therefore, the current required to cut the wire with Joule heating examined experimentally, $(I_C)_{\text{EXP}}$, might be different to $(I_C)_0$. The difference between $(I_C)_{\text{EXP}}$ and $(I_C)_0$ represents the difference in the thermal boundary conditions between the cases shown in Figs. 1(a) and Fig. 1(b), and can be expressed by $f$ defined as:

$$f = \frac{(I_C)_0}{(I_C)_{\text{EXP}}}.$$

With the use of $f$, the maximum temperature of the wire under the thermal boundary conditions shown in Fig. 1(b) is given by:

$$T_{HT} = \frac{1}{8K\sigma} \left( \frac{I_{HT} L}{A} f \right)^2 + T_0,$$

where $T_{HT}$ is the heat treatment temperature and $I_{HT}$ is the current required to realize this. In this study, prior to the heat treatment, we performed an experiment to cut the wire, and the value of $f$, which is needed to calculate $I_{HT}$, was determined; see the Appendix for further details.

### 3 Experimental Procedure

#### 3.1 Heat Treatment by Joule Heating

The samples were the polycrystalline Cu microwires, which are used as bonding wires in the electronics industry, and the diameter was 25 µm. The experimental setup for the heat treatment is shown in Fig. 2(a). This was performed in air ($T_0 = 22^\circ$C) while under observation with a microscope. The current was supplied via 0.2 mm thick Ag terminals, and the value of $L$ was fixed at 1 mm for all samples. The value of $f$ was determined experimentally prior to heat treatment, and with $L = 1$ mm was found to be 4.36; see the Appendix for further details.

Figure 2(b) shows the relationship between $T_{HT}$ and $I_{HT}$ determined form Eq. (4). The physical constants of Cu used were $K = 401$ W m$^{-1}$K$^{-1}$ and $\sigma = 5.96 \times 10^7$ S m$^{-1}$. The values of $T_{HT}$ were set to be 100, 150, 200, 250, 300, 350, 400 and
600°C, and the corresponding values of $I_{HT}$ were determined to be 0.44, 0.56, 0.66, 0.74, 0.82, 0.89, 0.96 and 1.18 A, respectively. In all cases, the current was increased at a rate of 0.02 A s$^{-1}$ until it reached the prescribed value ($I_{HT}$), and on reaching this, the current was maintained for 3 minutes. It has previously been verified that the crystal grain growth saturates within 3 minutes of the current being supplied\(^{20}\).

With current $I_{HT}$ the temperature at the middle of the wire reaches $T_{HT}$, and the temperature either side of this is lower than $T_{HT}$. Therefore, in this study, we used a scanning ion microscope (SIM) to examine a 0.1 mm section (10% of $L$) at the middle of the wire to evaluate the grain size.

3.2 Small-Span Bending Test

Small-span, three-point bending tests were performed under microscope observation to determine the mechanical properties of the heat-treated Cu microwires. As mentioned in the previous section, the middle part of the wire was heat-treated at $T_{HT}$, which is where the maximum value of the bending moment is applied in the three-point bending test, so the middle part of the wire makes a significant contribution to the bending stress. Thus, the three-point bending test was selected to determine the mechanical properties of the microwires.

The test platform consists of a sample holding part and a load part comprising a piezo stage and a force sensor, see Fig. 3(a)\(^{31}\). An expanded image of A in Fig. 3(a), which shows the test part, is shown in Fig. 3(b). The Cu microwire is simply supported by knife edges as shown in Fig. 3(b), and, in this study, the distance between the supports, $J$, was set to 0.93 mm.

![Fig. 3](image-url) (a) Photograph of the mechanical test apparatus. (b) Detail of A in (a). (c) Detail of the similar types of force sensor used.

3.3 Grain Growth

Grain growth to $T_{HT}$ was determined to $I_{HT}$ of $L$ and $K$ were set to be 100, 150, 200, 250, 300, 350, 400 and 450, respectively. In all cases, the current was increased at a rate of 0.02 A s$^{-1}$ until it reached the prescribed value ($I_{HT}$), and on reaching this, the current was maintained for 3 minutes. It has previously been verified that the crystal grain growth saturates within 3 minutes of the current being supplied\(^{20}\).

A loading probe was used to apply a load at the midpoint between the supports, and the force acting on the loading probe was recorded. Here the displacement of the wire at the loading point is given by

$$\delta = \delta_S - \delta_C,$$

(5)

where $\delta_S$ and $\delta_C$ are the displacement of the piezo stage, and the deflection of the double-beam cantilever, respectively. The force on the wire was determined from

$$P = k\delta_C,$$

(6)

where $k$ is the spring constant of the double-beam cantilever, which was set to 0.192 mN / \(\mu\)m in this study.

4 Results and Discussion

4.1 Grain Growth

An example of a scanning ion microscope (SIM) image of a Cu microwire that has not been heat-treated is shown in Fig. 4(a). Because the Cu microwires were prepared by the wire drawing process, those had characteristic crystal grain structures, with highly elongated crystal grains in the axial direction of the wire. Although the widths of the grains are of the order of nanometers, the lengths of the grains are of the order of micrometers. SIM images of examples of Cu microwires heat-treated at 200°C, 350°C and 600°C are shown in Figs. 4(b) to (d), respectively. It is clear that the crystal grains have grown due to the heat treatment, and this tendency is more remarkable in Cu microwires treated at higher $T_{HT}$. The crystal grains have grown in the radial direction, and a bamboo structure in which there are grain boundaries only in the radial
Because the middle part of the Cu microwire was treated in the software (QuickGrain, Inotekku), where cross section method with heat treatment was evaluated using image analysis, the middle part of the Cu microwire was considered to be heat treated at \( T_{\text{HT}} \), the evaluation was performed on a 0.1 mm long section at the middle of the wire.

Above 150\(^\circ\)C, the value of \( D \) for the untreated Cu microwire was determined to be 2.1 \( \mu \)m. On the other hand, the value of \( D \) for the Cu microwire heat-treated at 600\(^\circ\)C was 13.7 \( \mu \)m, which is 6.5 times greater than the untreated one.

### 4.2 Mechanical Properties

Figure 6(a), (b) and (c), show examples of load \((P)\)-displacement \((\delta)\) curves for the untreated Cu microwire, and those for wires heat-treated at 200\(^\circ\)C and 400\(^\circ\)C, respectively. In all these cases, \( P \) initially increases linearly with increasing \( \delta \), and the relationship becomes nonlinear at greater values of \( \delta \). From the linear part of the \( P-\delta \) relationships, the Young’s modulus of the Cu microwire can be determined using the following equation,

\[
E = \frac{J^3}{48I} \frac{P}{\delta}, \tag{7}
\]

where \( I = \pi d^4 / 64 \) is the moment of inertia of cross sectional area. It is not easy to determine the yield points in the \( P-\delta \) curves shown in Fig. 6, which were obtained from the three-point bending test, and moreover, the proof stress at a specific strain is difficult to define as in tensile tests. Therefore, in this study, we determined the yield stress as follows. The yield point, \( P_Y \), at which the \( P-\delta \) relation becomes nonlinear, was determined to be at the point where the coefficient of determination for a linear fit became less than 0.99. These points are indicated by the arrows in Figs. 6(a) to (c). Here we assume that all the whole cross section yields at \( P_Y \). Under this assumption, the yield stress, \( \sigma_Y \), can be found from

\[
\sigma_Y = \frac{6P_Y J}{d^3}, \tag{8}
\]

The values of \( E \) and \( \sigma_Y \) thus determined are summarized in Table 1 together with the heat treatment conditions for the wires. In each condition, three microwires were tested. Figure 7(a) shows \( E \) plotted against \( T_{\text{HT}} \). Although there was crystal grain growth at higher \( T_{\text{HT}} \), \( E \) was found to be independent of \( T_{\text{HT}} \), in other words independent of \( D \). The average value of \( E \) of all the microwires was determined to be 86.4 ± 2.4 GPa. This value of \( E \) is smaller than the value for bulk Cu (110~130 GPa). Related to this, It has been reported that rolled Cu having the crystalline anisotropy show lower values of \( E \).

### Table 1 Results of three-point bending tests done on the Cu microwires. The heat treatment conditions for the wires are also included in the table.

| Condition # | \( T_{\text{HT}} \) (\(^\circ\)C) | \( I_{\text{HT}} \) (A) | \( D \) (\( \mu \)m) | \( E \) (GPa) | \( P_Y \) (mN) | \( \sigma_Y \) (MPa) |
|------------|----------------|----------------|----------------|--------------|--------------|----------------|
| C1         | w/o            | n/a            | 1.60 ± 0.2     | 86.2 ± 1.6   | 3.51 ± 0.08  | 311 ± 7.1     |
| C2         | 100            | 0.44           | 1.79 ± 0.2     | 86.4 ± 1.9   | 3.51 ± 0.06  | 310 ± 4.9     |
| C3         | 150            | 0.56           | 2.21 ± 0.4     | 89.8 ± 3.7   | 2.16 ± 0.06  | 190 ± 5.7     |
| C4         | 200            | 0.66           | 2.69 ± 0.2     | 88.3 ± 0.7   | 1.82 ± 0.10  | 160 ± 11.9    |
| C5         | 250            | 0.74           | 4.95 ± 0.3     | 86.3 ± 4.1   | 1.31 ± 0.10  | 116 ± 12.9    |
| C6         | 300            | 0.82           | 5.37 ± 0.3     | 86.4 ± 1.2   | 1.16 ± 0.08  | 102 ± 7.2     |
| C7         | 350            | 0.89           | 7.54 ± 0.1     | 81.2 ± 7.4   | 1.15 ± 0.20  | 102 ± 16.5    |
| C8         | 400            | 0.96           | 12.3 ± 0.5     | 84.3 ± 4.9   | 1.08 ± 0.08  | 95 ± 7.4      |
| C9         | 600            | 1.18           | 13.4 ± 0.9     | 88.4 ± 8.3   | 0.85 ± 0.06  | 75 ± 5.6      |
In all these cases, and those for wires heat-treated at 200 and 400°C, respectively. The modulus of the Cu microwire can be determined using the equation:

\[ E = \frac{1}{2}k' \rho \frac{T_{HT} - T_{HT}}{m} \]

where \( E \) is the Young's modulus, \( k' \) is a constant, \( \rho \) is the density of Cu, \( T_{HT} \) is the heat treatment temperature, and \( m \) is the number of microwires. The value of \( E \) was applied. Because the middle part of the Cu microwire was determined to be 2.1 ± 0.2 GPa after heat treatment at 600°C was 13.7 ± 0.8 GPa after heat treatment at 400°C for 3 minutes. The Young’s modulus of the heat-treated Cu microwires examined in this study. As described in Section 4.1, the untreated Cu microwire and the one heat-treated at 100°C were confirmed as having a characteristic crystal grain structure with the grains highly elongated in the axial direction of the wire, and therefore, the value of \( D \) determined by cross section analysis was not representative of the average grain size of the wires. This is the most likely reason why the values of \( \sigma_y \) for these two microwires were larger than expected.

The two microwires excluded from the linear fit have higher yield strengths than the others. Why is this so? These microwires have crystal grains elongated in the axial direction, so the physical properties of these wires must be extremely anisotropic. In the present study, the force was applied in the radial direction of the wire, and the grain boundaries parallel to this direction were much shorter than \( d \). Therefore, it would be difficult for dislocations to accumulate in such short grain boundaries, and this contributed to the realization of higher yield strength.

### 4.3 Yield Strength and Grain Size

The Hall-Petch relation indicates that there is an inverse relationship between \( \sigma_y \) and \( D \), as follows

\[ \sigma_y = \sigma_0 + \frac{k}{D^{0.5}} \]

where \( \sigma_0 \) is the friction stress, and \( k \) is the Hall-Petch coefficient. Both of these are constants and independent of \( D \). Figure 7 shows the relationship between \( \sigma_y \) and \( D^{-0.5} \). A regression line fitted to all the data (C1 to C9 in Table 1) has a coefficient of determination of 0.856. To search for the best linear fit for the relationship between \( \sigma_y \) and \( D \), the fitting was performed on selected data. The coefficient of determination for fits to 8 points (C2 to C9), 7 points (C3 to C9) and 6 points (C4 to C9) was determined to be 0.807, 0.930 and 0.915, respectively, and the maximum coefficient of determination is for the line fitted to 7 points. In this case, \( \sigma_0 = 9.00 \times 10^6 \) Pa and \( k \) was found to be independent of \( T_{HT} \) and tends to saturate at higher \( T_{HT} \). The value of \( \sigma_y \) for the untreated Cu microwire was 311 ± 7.1 MPa, and decreased to 24% of this value (75 ± 5.6 MPa) after heat treatment at 600°C.

Kallend and Davies investigated the effect of the crystalline structure of the rolled Cu on its yield strength with Taylor model and Sachs model, and reported that the yield strength determined by both models fluctuated around 10% depending on the crystalline anisotropy. In the present study, the values of standard deviation for the determined values of \( \sigma_y \) for almost conditions were less than 10%, and therefore, the determined values of \( \sigma_y \) were considered to be valid.

### 5 Conclusions

Thin Cu wires with a diameter of 25 μm were heat-treated by Joule heating, and the mechanical properties of these wires were examined by conducting small-span, three-point bending tests. Initially, the Cu microwires had characteristic crystalline structures, where the crystal grains are elongated in the axial direction of the wire, and became uniform by supplying a constant direct current into the wire. The grain size of an untreated Cu microwire, evaluated by cross section method, was 2.1 μm, and this became 13.7 μm after heat treatment at 600°C for 3 minutes. The Young’s modulus of the heat-treated microwires was independent of temperature with a value of 86.4 GPa. On the other hand, the yield strength of the...
The authors would like to acknowledge Professor M. Saka for valuable discussions throughout this work. This work was supported by JSPS KAKENHI Grant Number 18H01331.

Appendix: Experiment to Cut the Wire to Determine the Thermal Boundary Conditions around the Wire

To determine the term $f$ that is used to account for realistic thermal boundary conditions around the Cu microwire, we performed an experiment to cut the wire. The experimental setup was the same as that used for the heat treatment, see Fig. 2(a), and the values of $(I_{\text{EXP}})$ were measured for various values of $L$.

Figure A1 shows the relationship between the current required to cut the wire by Joule heating experimentally, $(I_{\text{EXP}})$, and the length of the segment, $L$, through which current passed. $(I_{\text{EXP}})$ was found to decrease with increasing $L$. The values of $f$ were determined from Eqs. (2) and (3), and these are displayed in Fig A1(b) as a function of the slenderness of the wire, $d / L$. By fitting the data, we obtain

$$f = m \left( \frac{d}{L} \right)^2 + n \left( \frac{d}{L} \right).$$

where $m = -1.47 \times 10^3$ and $n = 2.11 \times 10^2$ are dimensionless constants. From Eq. (A1), the value of $f$ for $L = 1$ mm, which was the fixed value of $L$ used in this study, was determined to be 4.36, and this was used to determine $I_{\text{EXP}}$ from Eq. (4).

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