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Relationship between the stored energy and indentation hardness of copper after compression test: models and measurements

Mohsen Kazeminezhad

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Abstract Utilizing the differential scanning calorimetry (DSC) and Vickers hardness tests, the relationship between the stored energy and indentation hardness of copper after compression test is achieved experimentally. Three dislocation models are utilized to develop the relationships between the stored energy and hardness for justifying the experimental relationship. The relationships show that the stored energy is increased by increasing the hardness, non-linearly. By comparing the models' results with the experimental data, the validity of each model at different ranges of hardness is determined.

Introduction

The stored energy due to cold deformation of metals is an important parameter in microstructure changes of the metals after heat treatment, since it determines the number of nuclei in recrystallization phenomenon [1]. It has been reported that cold deformation causes the increase of dislocation density due to work hardening, and thus the stored energy as well as flow stress is increased. For measuring the stored energy of deformed metals some methods have been proposed, such as neutron diffraction, X-ray diffraction, and differential scanning calorimetry (DSC) [2–9]. The accuracy of the methods depends on the material type and its physical properties, and therefore the achieved values of stored energy from the methods may be 10 times lower or higher than the actual one [7]. Among the methods, the stored energy of different materials has been widely measured using the DSC [2, 8, 10, 11]. Although the stored energy can be measured from the above-mentioned methods, they are expensive and time consuming. Thus, a method with a shorter processing time and lower cost is preferred. Moreover, in many cases the deformation of different regions of a workpiece after metal forming process is different. Consequently, the stored energy of the regions is different and measuring it is difficult due to sample preparation from the different regions of the deformed workpiece. Therefore, a local mechanical property that depends on the dislocation density can be utilized to determine the stored energy. The indentation hardness can be a local mechanical property and is known as a simple test which can be performed easily at a low cost and short time.

In this study to provide a simple method for determining the stored energy, the relationship between the stored energy measured from the DSC test and Vickers hardness of the pure copper after compression test is experimentally determined and justified with the dislocation models. Then the validity of the models is discussed.

Experimental procedure

The compression test specimens with a height-to-diameter ratio of 1.5 were prepared from a 99.99% pure copper rod. The true strains in the range of 0.1–0.9 were applied to the specimens. Then utilizing the Vickers indentation test, the hardness values of the specimens were measured.

To measure the stored energy of the specimens, the DSC test was utilized. The DSC sample was cut from each specimen using a cutter machine with cooling system. To remove the deformed surface layers of the samples due to

M. Kazeminezhad (✉)
Department of Materials Science and Engineering,
Sharif University of Technology, Azadi Ave., Tehran, Iran
e-mail: mkazemi@sharif.edu

cutting, 50% nitric acid was used to dissolve the deformed layers. The samples were subsequently washed with ethanol to avoid getting any oil/grease on them. The final weight of the DSC samples was in the range of 50–100 mg.

The DSC used was a thermal analysis (TA) instruments model DSC Q100, equipped with Universal V3.9A analysis software. The calibration (temperature and cell constant) was done using an Indium standard sample with an accuracy of ±0.02 °C.

The DSC consists of two furnaces for the test and reference samples. Aluminum pans were used for both the sample and reference, and the DSC was purged with flowing nitrogen gas at a constant rate of 50 mL min^{-1}. The sample and reference pans were heated at a constant rate of 10 °C min^{-1} up to a maximum temperature of 500 °C, while the heat flow difference between the pans was monitored.

During the experiment, the instrument detects differences in the heat flow between the test sample and reference. This information is sent to an output device and results in a plot of the differential heat flow between the reference and sample cell as a function of temperature. When no thermodynamic chemical processes occur, the heat flow difference between the sample and reference varies only slightly with temperature. However, an exothermic process within the sample results in a significant deviation in the difference between the two heat flows, and a peak appears in the DSC curve. For example, the DSC curves for two of the samples are observed in Figs. 1 and 2 with an exothermic peak. Using the Universal software, the baseline was plotted, and the area bounded by the exothermic peak and baseline was measured from the initial to the final peak temperatures. The measured area is the stored energy of the sample due to the deformation.

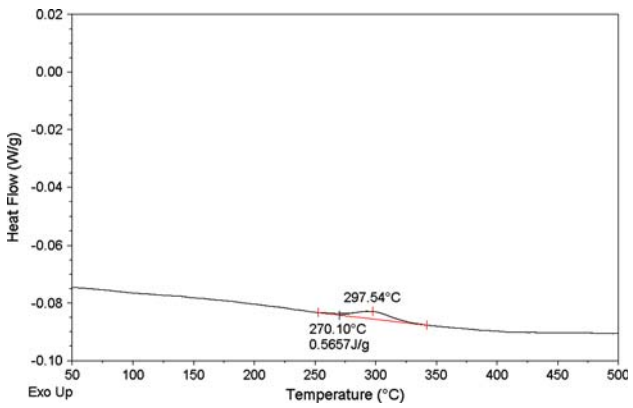

Fig. 1 DSC curve of the specimen with indentation hardness of 105 Hv

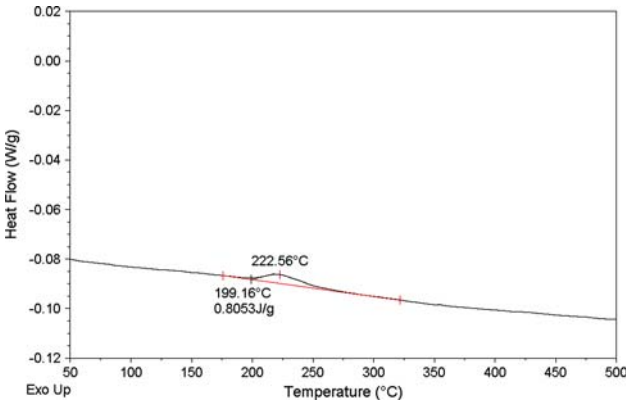

Fig. 2 DSC curve of the specimen with indentation hardness of 124 Hv

Results and discussion

Differential scanning calorimetry is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature [12]. Both the sample and reference are maintained at the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The basic principle underlying this technique is that, when the sample undergoes a physical transformation such as phase transitions, more (or less) heat will need to flow to it than the reference to maintain both at the same temperature. Whether more or less heat must flow to the sample depends on whether the process is exothermic or endothermic. When the sample undergoes exothermic processes, such as recrystallization, less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimetry is able to measure the amount of stored energy due to deformation released during recrystallization [12].

Also it can be discussed that the released stored energy due to the peaks, for example in Figs. 1 and 2, is just related to the recrystallization and dislocation density due to deformation, and the contribution of other parameters in the released energy is negligible.

It has been reported that during deformation, the point defects and deformation twins may be generated in face-centered-cubic (FCC) materials, such as copper [2, 13, 14]. They may contribute to the energy release, i.e., exothermic peak in DSC curve, while the samples are heated in DSC and may indirectly influence the measured value as the energy stored by dislocations. In fact, this may introduce an error in the dislocation models that will be developed in this study. Therefore, the fraction of energy associated with

the formation of point defects and deformation twins should be determined. In the literature, it has been noted that the energy associated with the formation of the point defects due to deformation of copper is in the range of 1.6×10^{-19} to 3.2×10^{-19} J with the concentration in the order of 10^{-5} [13–15]. Thus, the energy associated with the formation of the point defects is negligible compared with the energy values measured in the DSC tests, for example in Figs. 1 and 2.

During the deformation of copper, deformation twins are rarely observed, except at low temperature or very high strain rate deformation conditions where the dislocation slip processes are suppressed [16]. Also, it has been reported that for copper with grain size of less than 1 μm, the deformation twins at room temperature and low strain rate deformation conditions can be observed [16]. At normal deformation condition, such as compression test at room temperature, the deformation twins have not been observed in copper with grain size higher than 1 μm [17]. Since in the present research the grain size of the studied copper is 35 μm, therefore the deformation twins cannot be observed. Moreover, in the DSC curves no peak for recovery is observed before the recrystallization one. Thus, from the above descriptions, it can be concluded that all stored energy is released only in the recrystallization and relates to the dislocations density due to deformation.

In Fig. 3, the measured stored energy of the compressed specimens is plotted versus the indentation hardness, and their relationship can be given as follows:

$$E = 9 \times 10^{-5}\,\mathrm{Hv}^{2.352} \quad (\mathrm{MJ/m^3}) \tag{1}$$

where E and Hv are the stored energy and Vickers indentation hardness, respectively.

Utilizing different dislocation models, the mentioned relationship can be justified. Three models are investigated here and the discussion on their validity is carried out.

(a) Utilizing the following relationship, it is possible to find the flow stress from the Vickers hardness [18–21]:

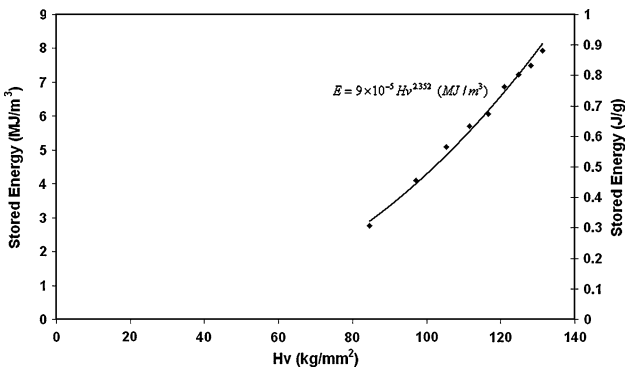

Fig. 3 The experimental measured stored energy versus indentation hardness

$$\sigma_{(\mathrm{MPa})} = \frac{Hv_{(\mathrm{kg\,mm^{-2}})}}{0.3} \tag{2}$$

where σ is the flow stress of material in a specific strain.

From the flow stress and following equations the dislocation density can be estimated, and correspondingly the stored energy is calculated [1, 8, 10, 11, 22–24]:

$$\sigma = \alpha M \mu b \sqrt{\rho} \tag{3}$$

$$E = C \mu b^2 \rho \tag{4}$$

where α is the material constant, M the Taylor factor, μ the shear modulus, b the Burgers vector, ρ the total dislocation density, and C the material constant.

By combining Eqs. 2–4, a relationship between stored energy and Vickers hardness is achieved:

$$E = \frac{C}{\mu} \frac{Hv^2}{0.09 \alpha^2 M^2} \tag{5}$$

(b) The total stored energy of cold work per unit volume is proportional to the total dislocation density [1]. Also, the total dislocation density is the sum of statistically stored dislocations and geometrically necessary dislocations [25]. Generally, the dislocation density is increased with increasing strain [25–30]. The density of statistically stored dislocations is given by [30, 31]:

$$\rho^S = \frac{M\varepsilon}{\lambda^S b} \tag{6}$$

where ρ^S is the density of statistically stored dislocations, ε the macroscopic strain of polycrystals, and λ^S the mean free path of the statistically stored dislocations. Conrad has proposed that the grain boundaries reduce the λ^S, thus [32, 33]:

$$\lambda^S = \alpha_S d \tag{7}$$

where $\alpha_S \leq 1$ and d is the grain size.

The density of geometrically necessary dislocation is also increased with increasing the strain [30, 31]:

$$\rho^G = \frac{M\varepsilon}{\lambda^G b} \tag{8}$$

where ρ^G is the density of geometrically necessary dislocations, λ^G the mean free path of the geometrically necessary dislocations which is proportional to the grain size.

Thus, the geometrically necessary dislocation density is estimated from Eq. 8:

$$\rho^G = \frac{C' M\varepsilon}{bd} \tag{9}$$

where C' is a constant.

From Eqs. 6, 7, and 9, the total dislocation density can be calculated and then the stored energy due to deformation is achieved [1, 25]:

$$\rho = \rho^S + \rho^G \tag{10}$$

$$E = C\mu b^2 \rho = \frac{C\mu bM\varepsilon}{d}\left(\frac{1}{\alpha_S} + C'\right) \tag{11}$$

where ρ^t is the total dislocation density.

Chaudhri [34] has proposed that at a specific strain, due to work hardening the Vickers hardness of copper can be estimated from the following non-linear relationship:

$$Hv = k'\varepsilon^{n'} \tag{12}$$

where k' and n' are the material constants.

Therefore, to achieve a relationship between the stored energy and indentation hardness using Eq. 12 the strain can be calculated from hardness and substituted in Eq. 11:

$$E = \frac{C\mu bM}{d}\left(\frac{1}{\alpha_S} + C'\right)\left(\frac{Hv}{k'}\right)^{\frac{1}{n'}} \tag{13}$$

(c) Jiang et al. [29] have reported that when the strain is very small, λ^S may be proportional to the grain size due to the presence of grain boundaries. As strain is increased, λ^S is decreased and very soon becomes independent of grain size, and thus the density of statistically stored dislocation may depend only on the strain. They proposed the following relationship to estimate ρ^S:

$$\rho^S = C_1\varepsilon^n \tag{14}$$

where C_1 and n are the constants.

Also, they suggested that when the strain is increased, the dynamic recovery reduces the storage rate of geometrically necessary boundaries with strain. Thus, the linear relationship between ρ^G and strain, i.e., Eq. 9, may not be valid. They proposed that the following general relationship may be more reasonable to describe the dependence of ρ^G on strain over a large strain range [29]:

$$\rho^G = \frac{C_2\varepsilon^m}{bd} \tag{15}$$

where C_2 and m are the constants.

Similar to section (b), from Eqs. 14 and 15 the total dislocation density can be calculated, and the following relationship between the stored energy and indentation hardness is concluded:

$$E = C\mu b^2\left(C_1\left(\frac{Hv}{k'}\right)^{\frac{n}{n'}} + \frac{C_2}{bd}\left(\frac{Hv}{k'}\right)^{\frac{m}{n'}}\right) \tag{16}$$

As mentioned above three Eqs. 5, 13, and 16 are achieved which describe the relationship between the stored energy and indentation hardness. From the measured Vickers hardness of the compressed specimens and utilizing the equations, the stored energy values of the specimens are calculated and presented in Fig. 4. Also, the results are compared with the experimental values of stored energy. As can be seen there is a good agreement between the results of Eq. 5 and experimental data over all values of hardness. This shows that the described dislocation model in section (a) is a general one for describing the work hardening in all studied strain as claimed by many other researchers [1, 8, 10, 11, 22–24]. Moreover, from Fig. 4 it can be observed that Eq. 13 is broken down over large values of hardness, and also the results of Eq. 16 are not valid in small ranges of hardness. Since the large values of hardness are due to the large strain, the break down of Eq. 13 in the large values of hardness can be attributed to the independence of the density of statistically stored dislocations on the grain size in large values of strain and invalidity of linear relationship between dislocation density and large strain [29], i.e., Eqs. 6 and 8 which are considered in developing Eq. 13. Also, the invalidity of Eq. 16 in small values of hardness is due to domination of the linear relationship between dislocation density and small strain [29] which is not considered in Eq. 16.

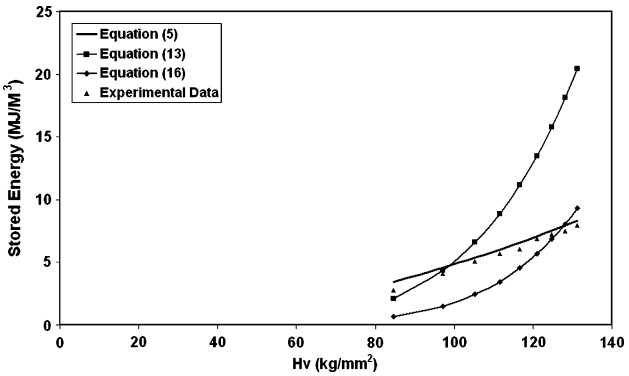

Fig. 4 Comparison between the results of different relationships and experimental data

Conclusions

The relationship between the stored energy and indentation hardness of copper after compression test is achieved experimentally. The relationship shows that the stored energy is increased by increasing the Vickers hardness value, non-linearly. Also, utilizing different dislocation models three relationships are developed that relate the indentation hardness to the stored energy. The results show that one of the models is valid over all ranges of hardness. Between the two others, it can be expressed that one is broken down over large values of hardness and the results of the other one are not valid in small ranges of hardness.

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