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Effect of stacking fault energy and strain rate on the microstructural evolution during room temperature tensile testing in Cu and Cu-Al dilute alloys

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The effect of stacking fault energy (SFE) on the evolution of microstructures during room temperature tensile testing has been investigated at two strain rates of 8.3×10^{-4} and 1.7×10^{-1}/s in pure copper, Cu-2.2%Al, and Cu-4.5%Al alloys with SFE values of, approximately, 78, 20 and 4 mJ/m^2, respectively. The mechanism of deformation changes from simple slip leading to cell formation in the high SFE metal, Cu, to overlapping and/or intersecting deformation twins in low SFE alloy, Cu-4.5%Al. The effect of strain rate is such that it results in rather poorly defined cell boundaries in copper, with a smaller cell size at higher strain rates for similar grain sizes and strain values. The alloys deform by twinning and the propensity of deformation twins increases with both a decrease in SFE value and increase in strain rates. © *1999 Kluwer Academic Publishers*

1. Introduction

The combined effect of grain size and strain rate on the dislocation cell formation and their subsequent size refinement during room temperature tensile testing has been recently reported for pure metals, aluminum [1], nickel [2], and copper [3]. An attempt was made to relate the cell sizes to strain rates for different grain sizes. However, the stacking fault energy (SFE) values of these pure metals are 166, 128, and 78 mJ/m^2 [4], respectively, which would place them under the classification of metals that primarily deform only by slip. The mechanisms differ in them due to the various extent to which the dynamic recovery can take place. These FCC metals do not cover the SFE range in which the fundamental mode of deformation can change from slip to twinning. Thus, the purpose of this paper is to present the results of a similar study on two alloys, Cu-2.2%Al and Cu-4.5%Al (compositions are in weight percents), with SFE values of 20 and 4 mJ/m^2 [4], respectively. Copper has been also included in this study so that SFE value range may be increased besides having similar FCC crystal structure. The alloys are considered to be substitutional solid solutions.

Dynamic recovery is a factor that enhances dislocation cell formation in metals and alloys during deformation [5]. It is anticipated that metals with medium to high SFE will be able to deform by slip where the dislocations arrange themselves in a lower energy configuration of cell walls because they can undergo large amounts of dynamic recovery. The transition from cell structure to twin deformation mode is due to the limited dynamic recovery associated with low SFE metals and alloys. The addition of substitutional solute atoms to pure metals is one of the fundamental ways in which the SFE value decreases in metals. Hong and Laird [6] have indicated that a critical concentration of the solute atoms changes the wavy slip to a planar slip in copper base solid solutions. Their analysis is based on the fact that addition of solute atoms not only results in lower SFE values, but the deformation mode is also dependent on the changes in atomic misfit and shear modulus values. The experimental results on pure copper and Cu-7.5%Al alloys during fatigue confirm such analysis [7].

However, Huang and Gray [8] have shown that microbands can be developed through the formation of coarse bands in pure metals Al, Cu, Ag and in 6061 aluminum and Al-Li-Cu alloys when deformed quasi-statically to moderate strain values. The mechanism of microband formation has been explained through the formation of double dislocation walls parallel to the primary slip planes. The microband misorientation has been found to be of the order of 1–3 degrees. It has been proposed [9] that the sequence of deformation mechanism changes from pure slip to the formation of (a) dislocation cells, (b) coarse bands, (c) microbands, (d) twins within the bands, and finally, (e) only twins leading to the development of intersecting and/or overlapping twins as the SFE value decreases in copper, Cu-2.2%Al, and Cu-4.5%Al alloys during room temperature wire drawing. Song and Gray [10] have also observed the transition from slip to twinning,

as a function of temperature in zirconium, which has an HCP crystal structure. Obviously in the case of Zr, the transition is forced because of the increase in Peierls stress required for the movement of the dislocations at very low temperatures. However, the limited number of slip systems available for slip may also be partly accountable for the slip to twinning transition. They also reported the presence of secondary twins within the large primary twins in the deformed microstructures.

Thus, it can be concluded that a single system, metal and/or alloys, is capable of showing the transition from slip to twinning. In this research an attempt was made to explore the possibility of determining such a transition in Cu-Al alloys due to the differences in the SFE values only for a given grain size at two different strain rates attainable in tensile testing machines. Such transitions are quite difficult to identify in the metals, and alloys deformed in shock loading where the twinning mechanisms for deformation have been rather well established [11].

2. Experimental procedure

The chemical compositions (in parts per million by weight) of pure copper and aluminum used for fabricating the alloys are shown in Table I. The alloys were prepared by heating the pure metals in an induction heating unit, using a graphite crucible, where the system was first outgassed with the help of a mechanical pump and then filled with ultra high purity (UHP) argon gas to a pressure of 500 mm of Hg. An ingot of 101.6 mm diameter rod was cast from a liquid with a superheat of 150 °C. The diameter of the rod was reduced to 25.4 mm (one inch) by extruding the alloys at 350 °C. The finished product was a rod of 9.525 mm diameter obtained by swaging the extruded rods at room temperature.

The 9.525 mm diameter rods were annealed at 450 °C for two hours to obtain a starting material for room temperature wire drawing. Wires with 8.23, 7.34, 6.53, 5.82, and 4.60 mm diameter were produced by drawing them through a set of standard dies providing a reduction of 13% cross-sectional area per pass. Similar procedure was used for pure copper also. The wires with 6.53 mm diameter of pure copper, Cu-2.2% Al and Cu-4.5% Al alloys were heat treated at 750, 850, and 880 °C for five hours, which resulted in grain diameters of 153, 145, and 146 μm, respectively. Thus a grain diameter of 150 μm can be considered as the grain size for all

TABLE I Chemical composition of copper in atomic parts per million by weight

BI	Pb	O	P	Se	S	Te	Cu
10	10	5	3	10	15	10	Bal.

Chemical composition of aluminum in atomic parts per million by weight

O	Cu	Fe	B	Ni	Si	C	Al
23	20	7	5	4	4	3	Bal.

the three materials used in this study. The grain sizes were measured by the linear intercept method, and at least 300 intersections with the test line were counted in each case. The mean intercept lengths were multiplied by 1.68 to obtain grain diameters.

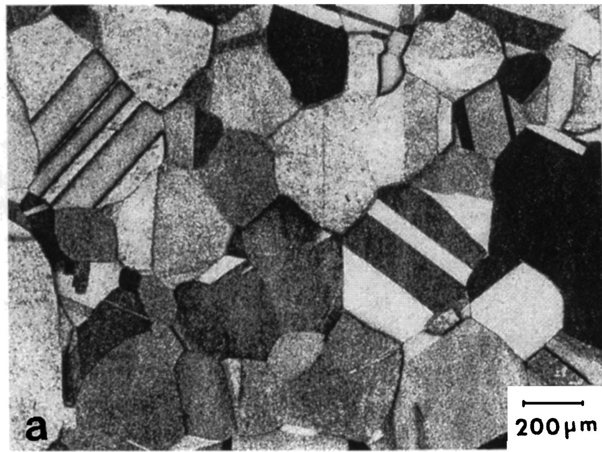
Instron model 1125 was used to deform the pure copper, Cu-2.2% Al, and Cu-4.5% Al alloys at strain rates of 8.3×10^{-4} and 1.7×10^{-1}/sec to true tensile strains of approximately 0.1 and 0.45. The optical and transmission electron microscopic (TEM) studies were done on the sections perpendicular to the tensile axis using the gage length (2 inches or 50.8 mm) portion of the tensile samples.

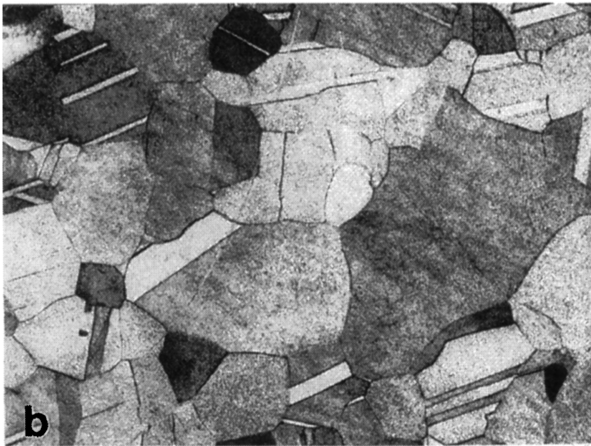
A wafer of 0.5 mm thick was cut from the deformed tensile samples with the help of a slow cutting Buehler Isomet machine. A TEM sample of 3 mm diameter was punched out from the wafer and hand-ground to a thickness of about 0.2 mm. A twin jet Struer's Tenupol 3 electroplating machine was used to polish the samples until perforation. The electrolyte consisted of a mixture of 1100 ml-distilled water, 400 ml-phosphoric acid, 500 ml-ethanol, 100 ml-propanol, and 10 grams-urea, while the polishing was performed at 2–16 °C, 10–13 volts, and a flow rate of 8. The TEM samples were observed in a Hitachi H-8000 scanning transmission electron microscope at an accelerating voltage of 200 kV. At least four to five samples were used for microstructural characterization for each deformed condition.

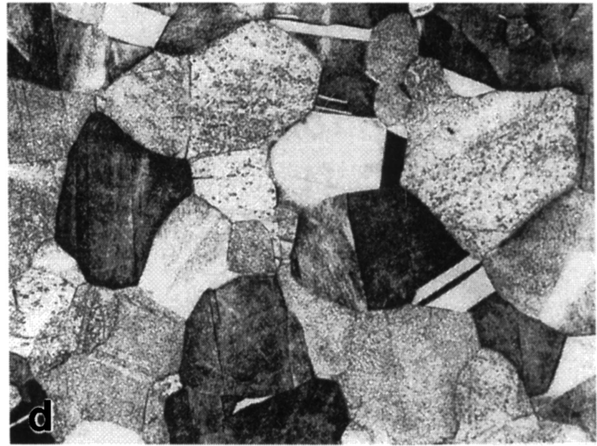
3. Results and discussion

Fig. 1 shows the microstructures developed in pure copper during tensile testing at strain rates of 8.3×10^{-4} and 1.7×10^{-1}/sec deformed to true strains of 0.1 and 0.3 using optical microscopy. The annealed microstructure shows the presence of a number of annealing twins, which can be seen in the deformed samples also. However, the important feature of the microstructures shown in Fig. 1 is the reduction in grain size at a given strain rate when the samples are deformed to higher true strains. A comparison at two different strain rates shows that more grain size refinement occurs at higher strain rates, as shown for equivalent strains in this figure. There appears to be no evidence of deformation twins developed in the deformation process. Even though the widths of the twins appear to be smaller in the deformed condition than in the annealed condition, the volume fraction of the twins are not significantly altered.

The TEM of the deformed samples is shown in Fig. 2. A well-defined cell wall develops in pure Cu even at a low strain rate of 8.3×10^{-4}/sec with a strain of 0.1. The cell walls in pure Cu are not as sharp as those generally observed in aluminum. This difference may be due to the limited extent of dynamic recovery in copper having a SFE value [4] of only 78 mJ/m^2 compared to 166 mJ/m^2 for aluminum. The cell size decreases with increase in tensile strain as shown in the micrographs in Fig. 2a and b, while Fig. 2c and d show corresponding microstructures developed at higher strain rates. A comparison of the microstructures in Fig. 2a and c shows that the effect of strain rate is such that the cell walls are not well defined at higher strain rate for similar tensile







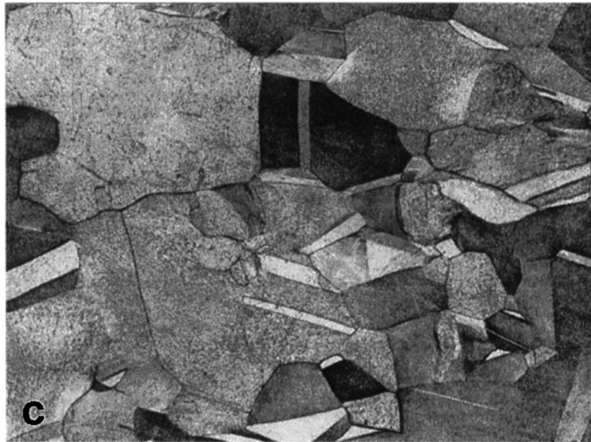


Figure 1 The microstructures developed in pure copper during room temperature tensile testing: (a) in the annealed condition, deformed at a strain rate of 8×10^{-4}/sec to tensile strains of (b) 0.11, and (c) 0.31, deformed at a strain rate of 1.7×10^{-1}/sec to tensile strains of (d) 0.11 and (e) 0.31.

strains. However, the cell size is smaller at higher strain rates at both strains of 0.1 and 0.3. The inability of the cells to form well-defined cell walls at higher strain rates, particularly those attainable in tensile testing at room temperature, has been reported in pure metals Al, Cu, and Ni [1–3] also, and was attributed to the inability of the dislocations to assume the low energy configuration in the cell walls due to the less time available at higher strain rates.

The material with the next lower SFE value (20 mJ/m^2) of this study is the Cu-2.2%Al alloy. Fig. 3 shows the optical micrographs both in the annealed condition and when deformed at the two selected strain rates, similar to those in copper, up to tensile strains of approximately 0.13 and 0.41. The annealed microstructures show similar grain size of 150 μm compared to pure copper but the volume fraction of the annealing twins is rather high in the alloy. Also, it must be noted

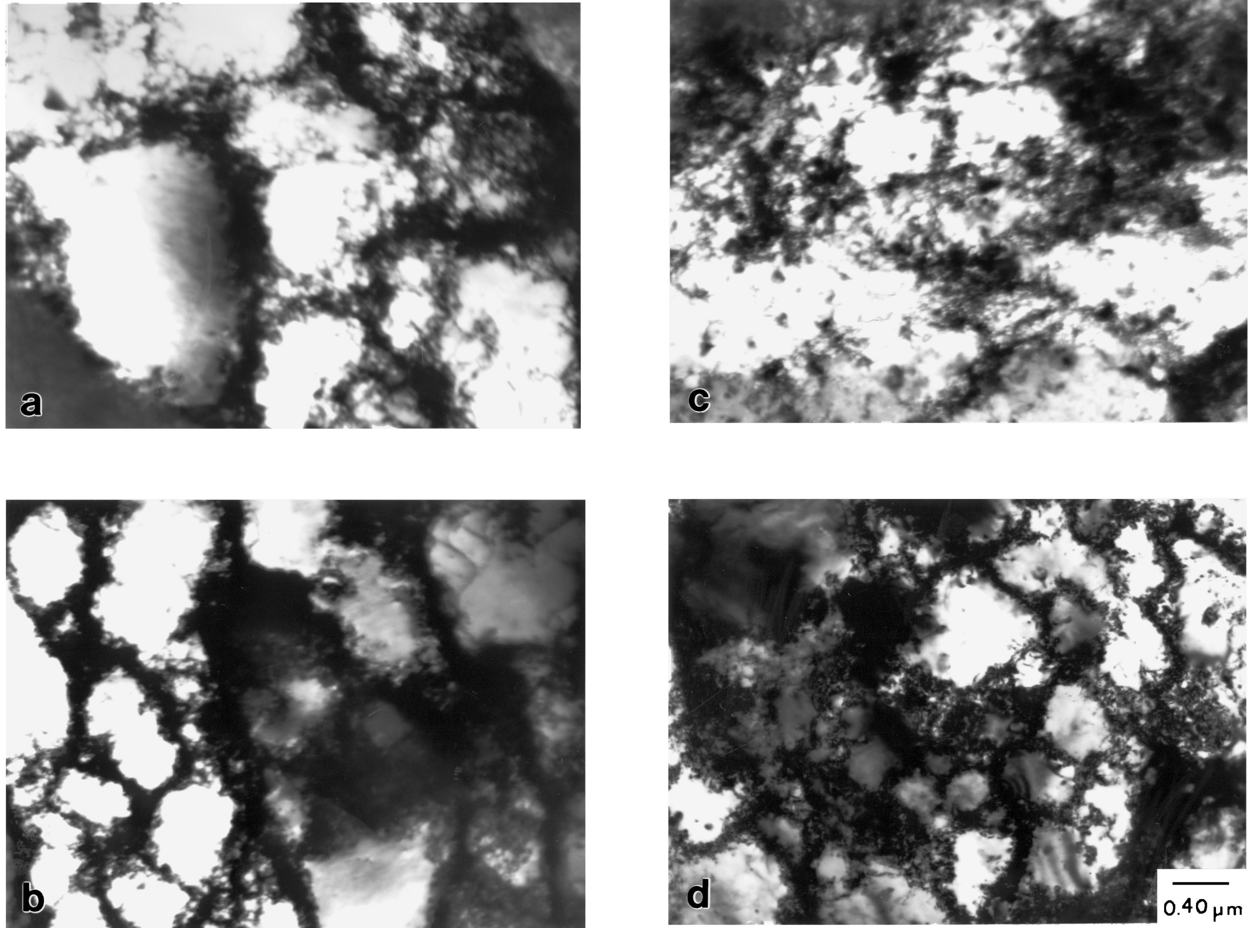


Figure 2 The micrographs showing the microstructural evolution in TEM for pure copper at a strain rate of 8×10^{-4}/sec to tensile strains of (a) 0.11 and (b) 0.31, and a strain rate of 1.7×10^{-1}/sec to tensile strains of (c) 0.11 and (d) 0.31.

that the grain size variation is relatively large, perhaps, due to the reminiscence of the drawn structure prior to annealing. There are two features in Fig. 3 that can account for the differences in the microstructures with those produced in pure copper as shown in Fig. 1: (1) presence of deformation twins and (2) higher grain size reduction with increase in tensile strain.

It can be seen in Fig. 3 that the tensile strain in the Cu-2.2% Al alloy is accommodated by the deformation twins within the grains, while the grains themselves are undergoing the size reduction at both strain rates. The volume fraction of the deformation twins increases with an increase in tensile strain at both strain rates and the grain size is smaller at higher strain rates for equivalent strain values. It must be noted that orientation of the deformation twins within a particular grain does not change in this alloy.

The details of the microstructural features in this alloy can be observed in TEM as shown in Fig. 4. Fig. 4a shows that the deformation twins at lower tensile strain rate cannot be seen; it only shows the annealing twin. The twin appears to have rather high dislocation density. At a tensile strain of nearly 0.4, the presence of deformation twins is quite evident in the microstructures. Thus, it is possible to have deformation twins at lower strain values, which the observed TEM samples did not reveal because of their large sizes. On the other hand, the deformation by slip at small strains, which changes to deformation twins at higher strains for lower strain rate process, can be rationalized from the micrographs shown in Fig. 4.

However, surprisingly, deformation twins could not be resolved in the microstructures for the samples deformed at higher strain rate as can be seen in Fig. 4c and d. These two micrographs indicate that the dislocations have a more or less uniform distribution at a strain of 0.12, while a distinct dislocation cell structure at strain values of 0.4 is the main microstructural feature at this strain rate for the alloy. It is difficult to interpret the micrographs shown at higher strain rates of this alloy unless it is assumed that dynamic recrystallization, perhaps, caused by the adiabatic heating during tensile testing, is the controlling phenomenon.

The Cu-4.5% Al alloy was the lowest SFE (4 mJ/m^2) material tested in this study. Fig. 5 shows the optical micrographs of this alloy in the annealed condition as well as after deforming them to tensile strains of 0.10 and 0.45 at the two strain rates indicated above for the other two materials. The grain size in the annealed condition is similar to the grain size in the alloy with a higher SFE value and pure copper. The volume fraction of the annealing twins in the two alloys in the annealed condition appears to be quite similar. The two characteristic features observed in the Cu-2.2% Al alloy were also identified in this alloy: the presence of deformation twins and the grain size reduction as a function of tensile strain. The presence of deformation twins at tensile strains of about 0.1 can be easily seen at both

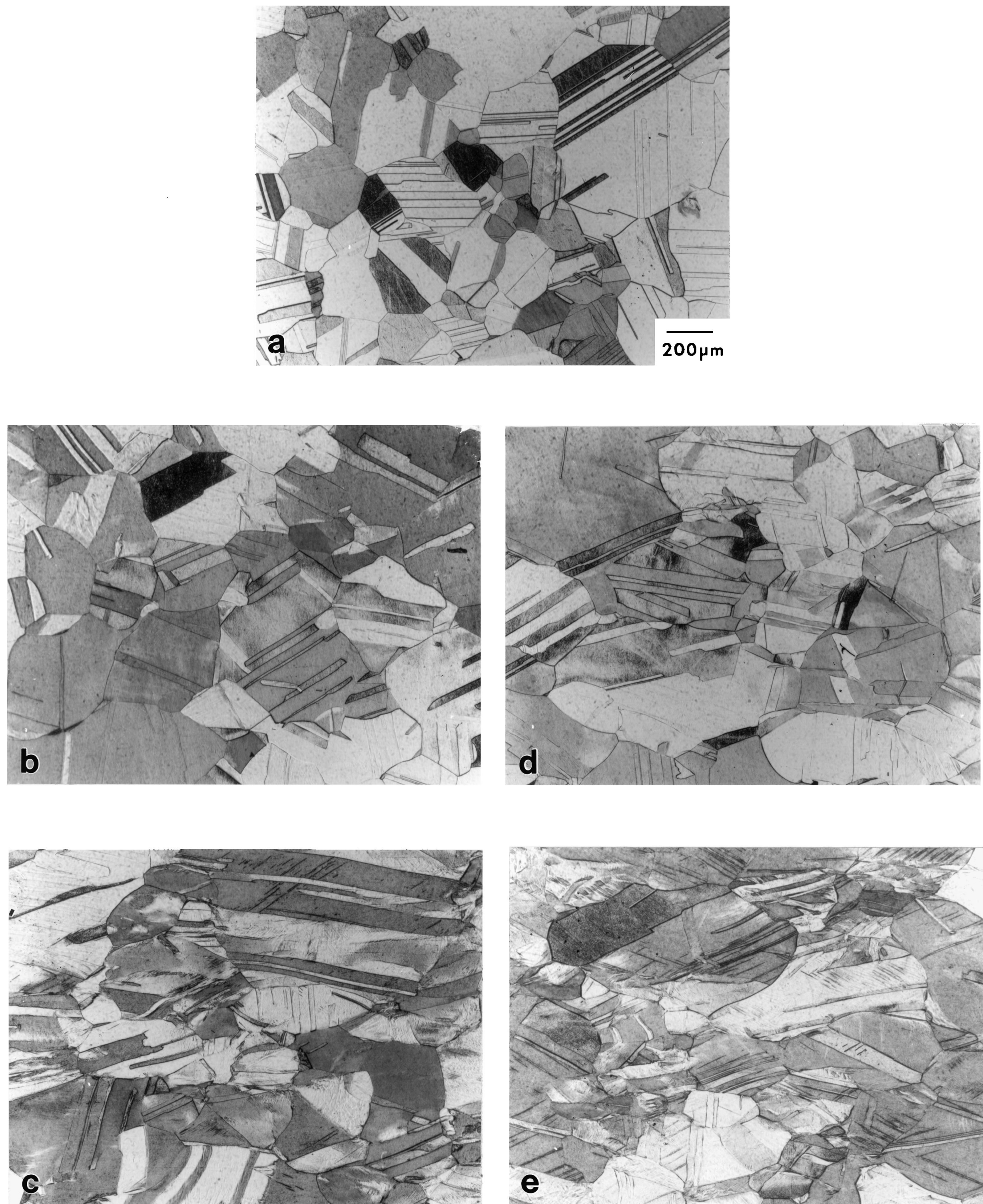


Figure 3 The microstructures developed in Cu-2.2%Al alloy during room temperature tensile testing: (a) in the annealed condition, deformed at a strain rate of 8×10^{-4}/sec to tensile strains of (b) 0.13 and (c) 0.41, deformed at a strain rate of 1.7×10^{-1}/sec to tensile strains of (d) 0.125, and (e) 0.43.

strain rates but the density of the twins is much higher at higher strain rate. Compare the micrographs in Fig. 5a and c. The most striking distinguishing feature for this alloy can be seen in the micrographs shown at tensile strains of 0.4 for the two strain rates. The deformation twins within a grain assume multiple orientations, and the density of deformation twins is so large that the identification of the grain matrix becomes a difficult task. The effect of strain rate appears to be such that the high strain rate simply produces higher deformation twin density for similar tensile strains. Note that a comparison of the ductilities of the two alloys of this study indicates higher total elongation for the alloy with lower SFE value even though the tensile data is not presented in this paper. It has been known for quite sometime now that the formation of large amounts of twins can produce higher elongations during tensile testing [12].

Fig. 6 shows the details of the microstructural evolution in the Cu-4.5%Al alloy at strains of 0.1 and 0.4 for the two strain rates of this study. The presence

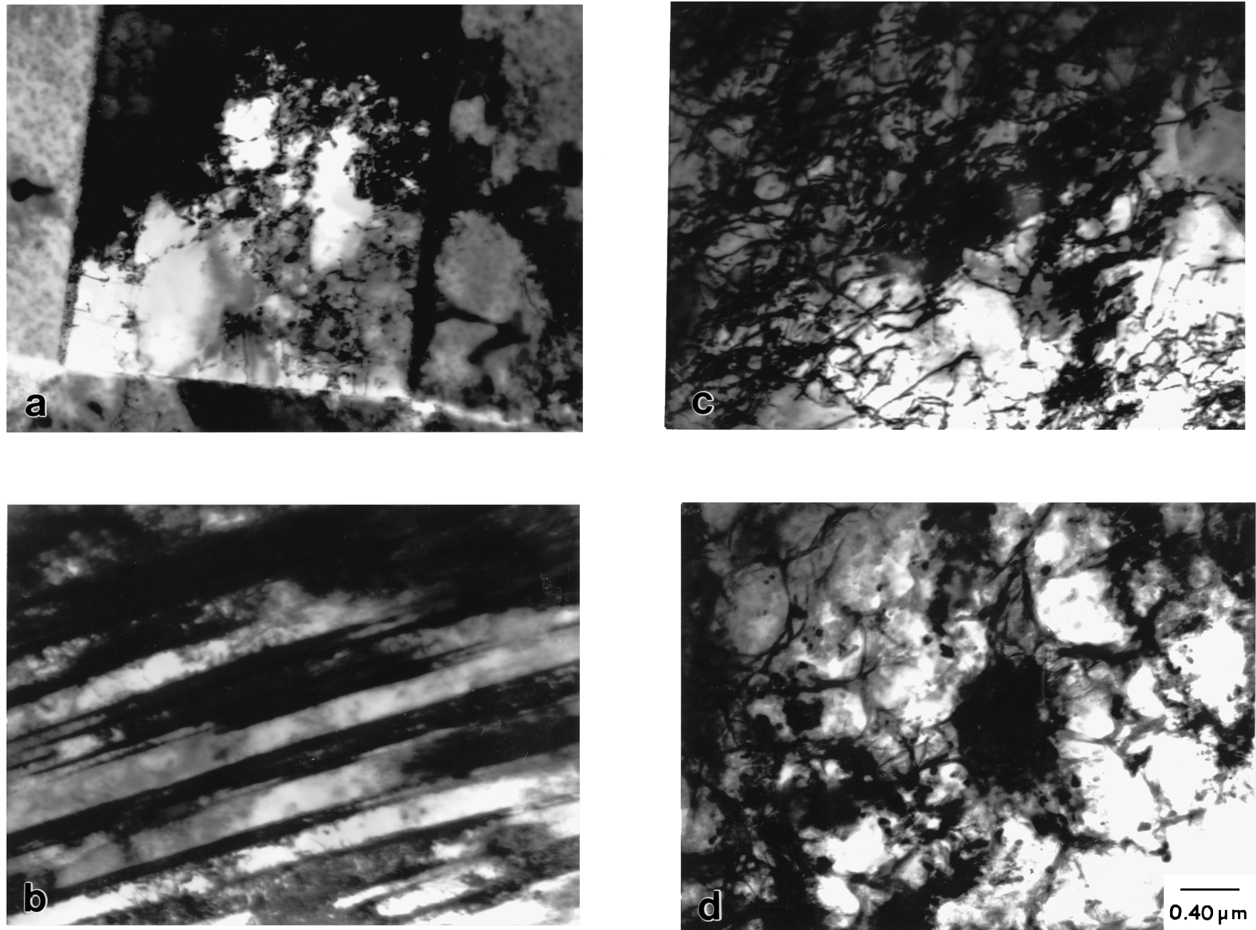


Figure 4 The micrographs showing the microstructural evolution in TEM for Cu-2.2%Al alloy at a strain rate of 8×10^{-4}/sec to tensile strains of (a) 0.13 and (b) 0.41 and a strain rate of 1.7×10^{-1}/sec to tensile strains of (c) 0.125, and (d) 0.43.

of deformation twins can be noted in all the four micrographs of this figure. More number of deformation twins can be seen in Fig. 6c than in Fig. 6a, confirming the observations of optical microscopy shown in Fig. 5. The higher strain rate produces few intersecting twins also at a strain of about 0.1 of this alloy. The effect of increasing the tensile strain at a given strain rate or increasing the strain rate for a given strain produces similar features during tensile testing based on our observations of this study. Finally, very large deformation twin density as well as higher number of overlapping and/or intersecting twins can be seen in Fig. 6d, which represents the deformation at a higher strain rate to a tensile strain of 0.4.

4. Analysis

The effect of SFE on the development of microstructures during room temperature tensile testing in pure copper, Cu-2.2%Al, and Cu-4.5%Al alloys has been observed to be such that as the SFE value decreases (a) a higher amount of grain size reduction takes place and (b) there is a transition from the slip to twin deformation mechanism. The difficulty with which the dislocations can cross slip with decreasing SFE value may be considered as one of the main factors responsible for these observations. It is not difficult to envisage the concept of higher grain size reduction for lower SFE metals and alloys on the basis of limited dynamic recovery concepts. If the dislocation density continues to increase during the tensile deformation, then the incremental increase in number of dislocations can only be accommodated in the grain boundaries due to the geometrical constraints placed on the deforming sample. This must result in an effective grain size reduction according to well-known Hall-Petch theories of grain size strengthening. However, as the effective grain size decreases, the threshold stress for twinning decreases, which would result in the transition from slip to twinning mode of deformation.

The increase in strain rate can complicate the process of microstructural evolution during the deformation process in this study. It is believed that increasing the strain rate results in the enhancement of limited dynamic recovery effects described above. However, if it is assumed that the threshold stress for twinning is lower at higher strain rates for a given grain size [13, 14], the resolved shear stress for twins of multiple orientations can be achieved rather easily. It must be noted that the microstructures observed in this study have been developed as a result of the combined effect of strain rate and SFE.

5. Conclusions

The evolution of microstructures during room temperature tensile testing in pure copper, Cu-2.2%Al, and Cu-4.5%Al alloy with SFE values of 78, 20, and

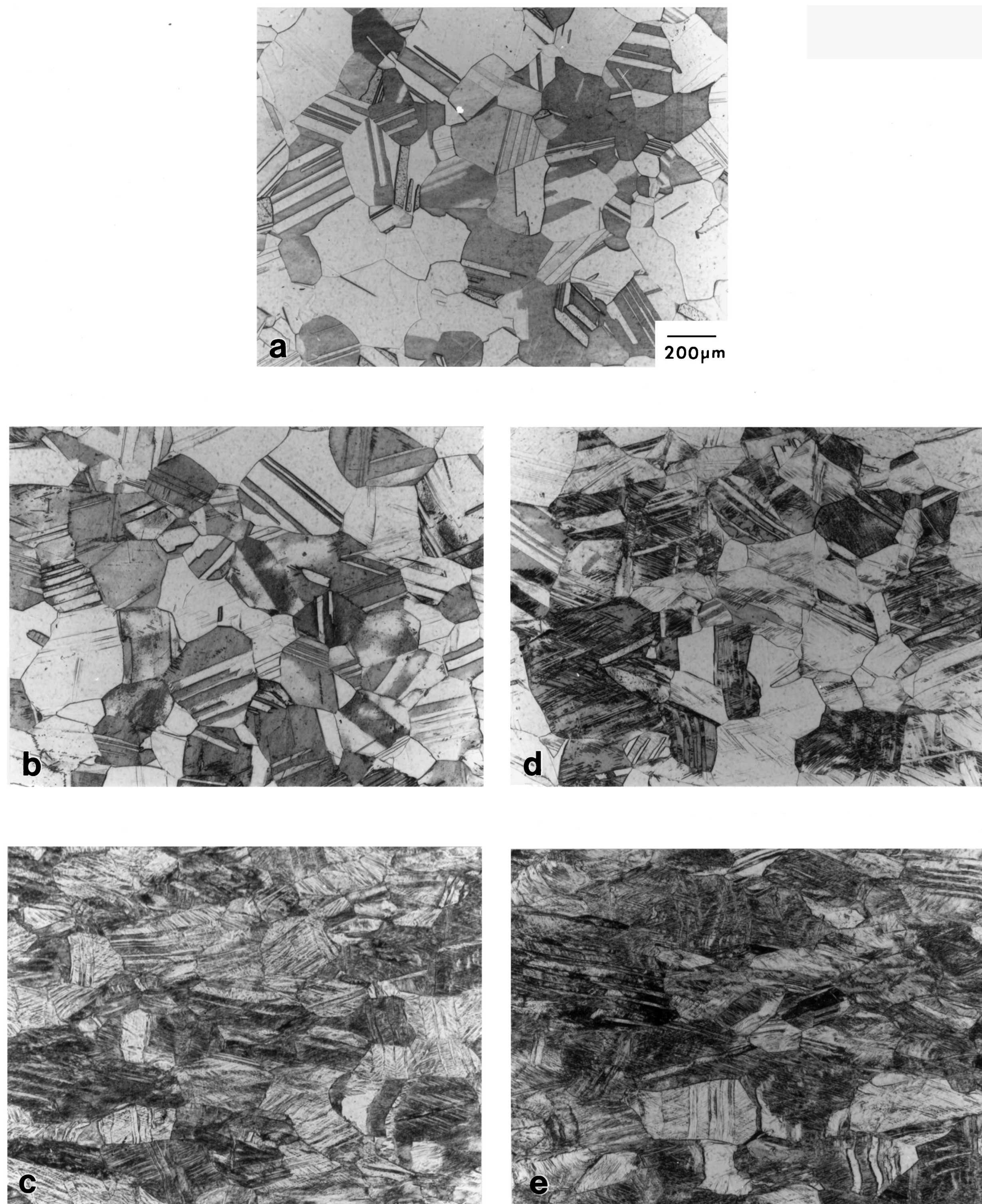


Figure 5 The microstructures developed in Cu-4.5% Al alloy during room temperature tensile testing: (a) in the annealed condition, deformed at a strain rate of 8×10^{-4}/sec to tensile strains of (b) 0.10, and (c) 0.45, deformed at a strain rate of 1.7×10^{-1}/sec to tensile strains of (d) 0.15, and (e) 0.41.

4 mJ/m^2, respectively, has been found to indicate the following features:

1. Pure copper deforms by a slip process leading to the development of dislocation cell structure, and the cell size decreases with increase in tensile strain. The effect of increasing the strain rate is to produce rather ill-defined cell walls with smaller cell sizes for similar strain values.

2. The Cu-2.2% Al alloy deforms by deformation twins, and the density of twins increases with increase in both increase in tensile strain as well as strain rate. Mainly unidirectional twins within a grain have been observed in this alloy.

3. The alloy Cu-4.5% Al also deforms by deformation twins, and the density of deformation twins increases with increase in both strain rate and tensile strain. However, the distinguishing feature of this alloy is the presence of overlapping and/or intersecting twins compared to Cu-2.2% Al alloy.

4. The effective grain size reduction (or partitioning) as a function of both strain rate and tensile strain is a

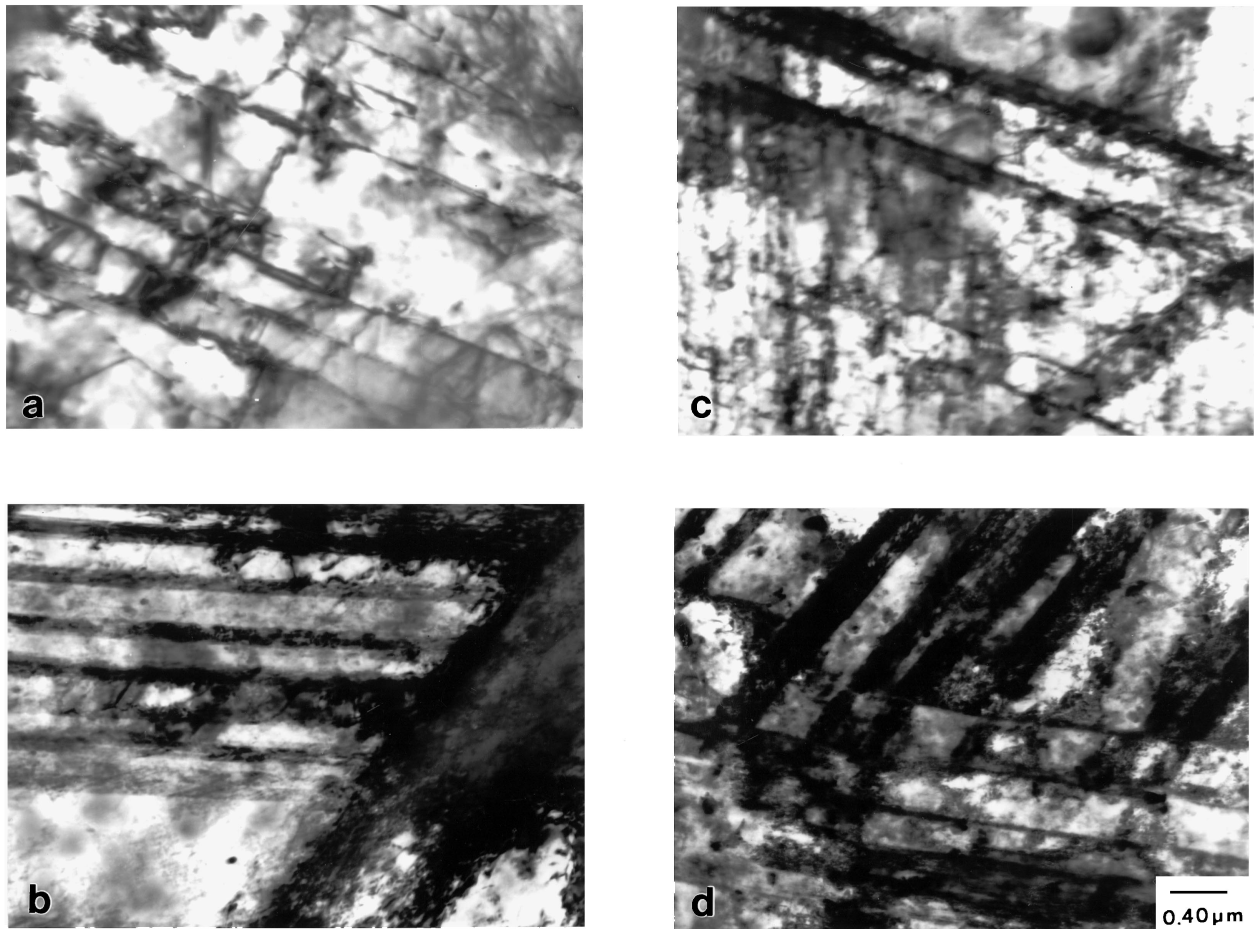


Figure 6 The micrographs showing the microstructural evolution in TEM for Cu-4.5%Al alloy at a strain rate of 8×10^{-4}/sec to tensile strains of (a) 0.10 and (b) 0.45 and a strain rate of 1.7×10^{-1}/sec to tensile strains of (c) 0.15, and (d) 0.41.

common feature for all three materials in this study. However, higher grain size reduction per unit tensile strain has been observed as the SFE decreases.

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