PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON CRYSTAL LATTICE DEFECTS, 1962, CONFERENCE
JOURNAL OF THE PHYSICAL SOCIETY OF JAPAN VOL. 18, SUPPLEMENT II, 1963

Stacking Faults of Copper-Germanium Alloys

Shigeyasu KODA, Koji NOMAKI AND Minoru NEMOTO
Research Institute for Iron, Steel and Other Metals
Tohoku University, Sendai, Japan

Stacking-fault energies of solid solution Cu-Ge alloys were obtained by means of X-ray diffraction method and electron microscopy, and also the segregation of solute atoms into stacking faults (Suzuki effect) was examined experimentally. The stacking-fault energy of a Cu-9.13%Ge alloy was given to be 0.88 erg/cm² by X-ray method, and 10 erg/cm² by electron microscopy. This discrepancy was discussed.

In the present report, stacking-fault energy of copper solid solution alloys containing germanium from 3 to 9% was obtained by means of X-ray diffraction method and electron microscopy. Moreover, the structure of a cold-worked alloy with 9% germanium was observed by electron microscopy, and also the segregation of solute atoms into stacking faults (Suzuki effect was examined from the experimental results.

1. Results Obtained by X-ray Method

The specimens used were pure copper and four copper-germanium alloys, which contain germanium of 3.09, 5.32, 6.48 and 9.13 wt.%, respectively.

When an alloy has many stacking faults, that is, the stacking-fault probability of the alloy is high, a peak shift of diffraction lines occurs according to the theory of Warren *et al.*[1] As a result, for example, the difference between (111) and (200) diffraction lines decreases as the stacking-fault probability increases. However, as the stacking-fault probability of an alloy in the annealed state is too small and cannot affect the line shift, the alloy was filed into powder to increase the probability by increasing the density of dislocations. This method has been applied by many authors, and also applied in the present experiment.

As-filed powder of the alloys and their annealed ones were examined by a G-E high angular goniometer equipped with a Geiger counter. The peak positions of X-ray diffraction lines were obtained by point counting across each peak. The measurement of counts in a constant time was made at every 0.01° by handling. Therefore, the error did not exceed above 0.01° for 2θ, where θ is a diffraction angle of a diffraction line. Thus, the difference between $2(\theta_{200}-\theta_{111})$ was obtained for each alloy, and the stacking-fault probability was calculated by the same method as Warren *et al.* did.

The result obtained was shown in Fig. 1.

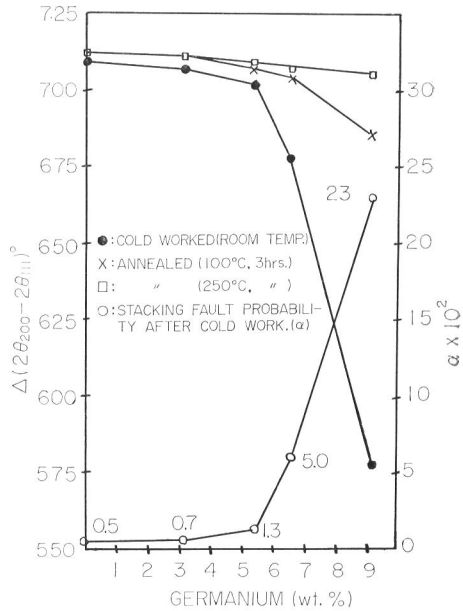

Fig. 1. Difference between (200) and (111) diffraction lines and stacking-fault probability of various alloys.

In order to obtain the stacking-fault energy from the probability, it is necessary to know the density of dislocations. However, in the present experiment, it is assumed that the density was the same for all specimens, because the cold working by filing is so severe that the density may be considered to be a constant value of 10^{12}/cm² for all specimens. Assuming the stacking-fault energy of copper as 40 erg/cm², the stacking-fault energies of the alloys were estimated as shown in Fig. 2.

As seen from Fig. 2, the stacking-fault energy of this alloy system decreases rapidly with approaching to the solubility limit of the solid solution. It is due to the fact that the neighbouring phase of the copper solid solution in the copper-germanium system is Cu_5Ge and the structure is hexagonal.

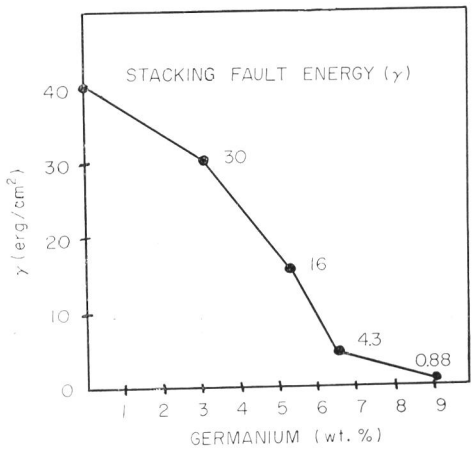

Fig. 2. Stacking-fault energy obtained by X-ray method.

2. Result Obtained by Electron Microscopy

A Cu-9.13%Ge alloy was used for electron microscopy. The alloy was cold rolled to 0.05 mm thick and annealed at 800°C.

In the first experiment, specimens taken from this sheet were deformed by tension to the elongation of 2, 5, 10, or 20%, and then thinned by electropolishing to observe them by the transmission electron microscope.

Fig. 3. Electron micrograph of Cu-9.13%Ge alloy after 2% deformation.

In the annealed state of this alloy, any long extended dislocation could not be seen in the electron micrographs, but in cold worked state. both long extended and not extended dislocations were observed together (Fig. 3). When the degree of working was small, the arrangement of dislocations was rather simple, and sometimes piled-up dislocations were observed at grain boundaries. As the deformation proceeded, the dislocations became to tangle each others (Fig. 4) and, in some cases, form a cell structure (Fig. 5), or tangle to grain boundaries or twin boundaries. More-

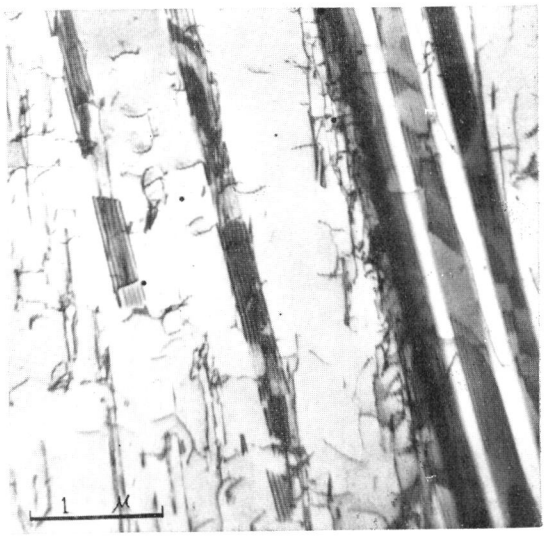

Fig. 4. Electron micrograph of the same alloy after 5% deformation.

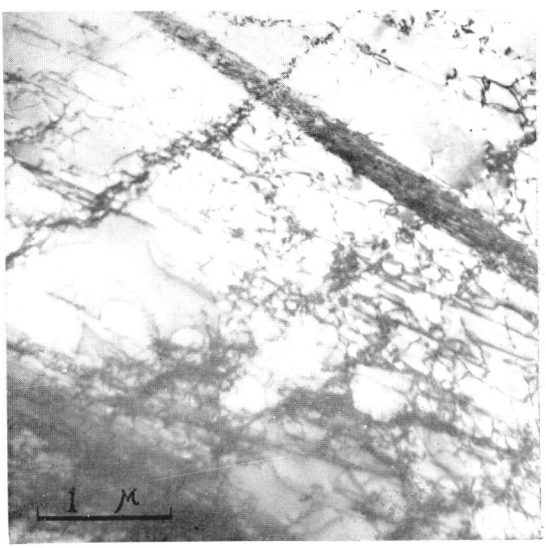

Fig. 5. The same conditions as for Fig. 4.

over, hexagonal dislocation networks on a slip plane, which were formed by crossing of dislocations on two slip planes, were also observed. (Fig. 6) In this case, as Whelan[2] described, the extension of dislocation nodes was observed. Furthermore, striations that were due to small twins, were observed in some cases (Fig. 7).

The stacking-fault energy can be obtained by two methods from the results of the electron microscopy. The first method is to measure the curvature of dislocation nodes of the hexagonal networks and to obtain the energy by a calculation using the formula given by Whelan[2]. The second is to obtain the energy from the width of an extended dislocation which is in an equilibrium state of forces, that is, in a state that a repulsive force acting between two half dislocations is in equilibrium

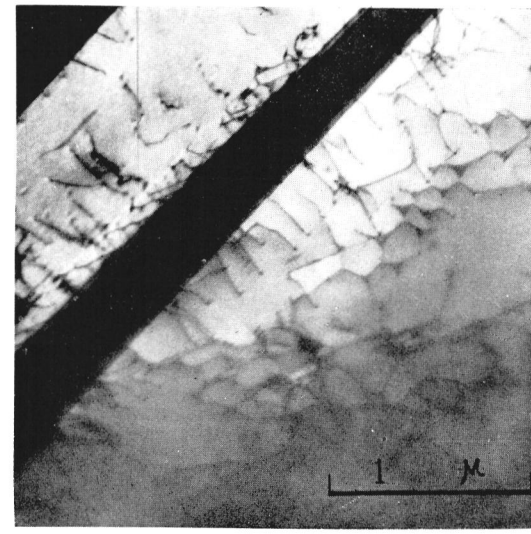

Fig. 6. The same conditions as for Fig. 4.

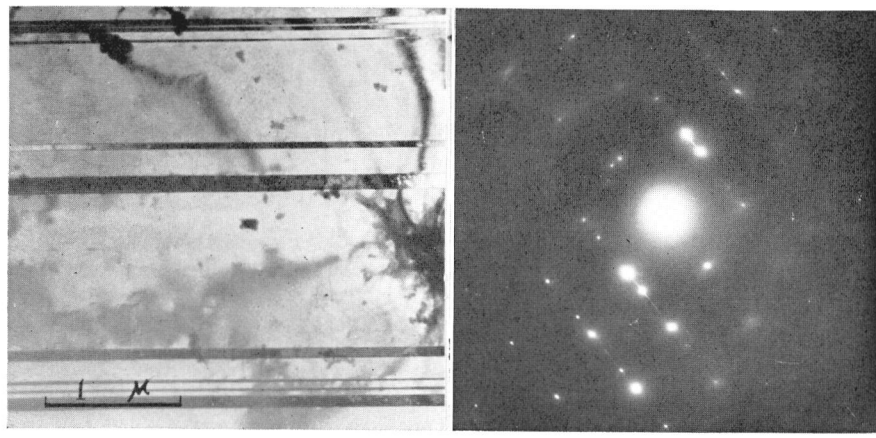

Fig. 7. Electron micrograph and selected area diffraction pattern of the same alloy after 5% deformation.

with a contractive force due to the stacking fault. In the present case, it is hardly considered that the very long extended dislocations were in equilibrium, because they are too wide, sometimes having a width of several microns, and at the same time very narrow and undissociated dislocations were also observed with them. However, if there are some criteria to ascertain their equilibrium, this method might be applied.

Then, the stacking-fault energy was obtained by the first method and given as about $10\pm0.8\,\mathrm{erg/cm^2}$ for a Cu-9.13%Ge alloy. The mean value of the curvature of dislocation nodes was about 1,200 A.

In the second experiment, the observation of the change in the structure of dislocations and the measurement of stacking-fault energy when annealing a specimen deformed 5% by elongation, were carried out.

When annealing any remarkable change in the arrangement of dislocations could not be seen before recrystallization, but some extension of dislocation nodes could be observed as shown in Fig. 8. Table I shows the stacking-fault energy obtained by the same method as mentioned above. In this table, the bracketed figures show rather inaccurate value, because the number of measurements was a few.

Considering from these results, it can be concluded that decrease of stacking-fault energy occurred by low temperature annealing. This change of stacking-fault energy may be

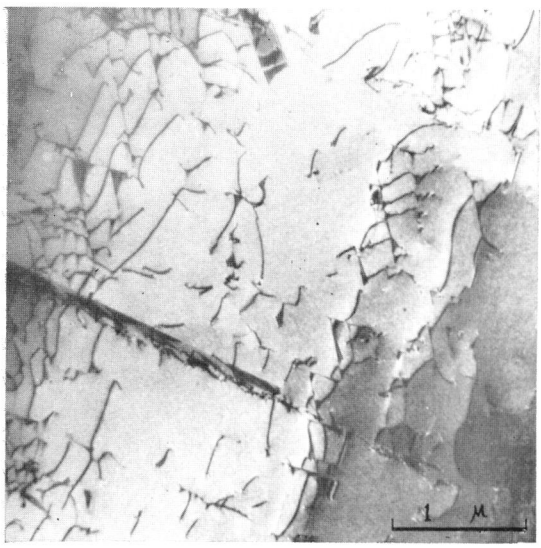

Fig. 8. Electron micrograph of the same alloy after 5% deformation and annealing for 1,000 min at 350°C.

due to the segregation of solute atoms into stacking faults by low temperature annealing. This fact can be thought as an evidence of Suzuki effect.

3. Discussion

There may be two questions. The one is the nature of long extended dislocations observed in electron micrographs of cold worked specimens, and the other is the discrepancy between values of stacking-fault energy obtained by X-ray and electron microscopy.

It is an unsolved question at the moment why such long extended dislocations appear. However, these long extended dislocations could not be considered to be in equilibrium. The reason is as follows. The width of the stacking fault of these dislocations is as wide as several microns, so the stacking-fault energy becomes to be an order 1/100 erg/cm^2, if these extended dislocations are in equilibrium. If this value is true, the co-existence of unextended dislocations cannot be explained. On the other hand, the value obtained from the curvature of dislocation nodes is too large to explain the extension of some dislocations.

Table I. Change of stacking-fault energy of Cu-9.13%Ge alloy by annealing. (erg/cm^2)

Cold worked state	10.0±0.8			
Annealed state	300 min	1,000 min	3,000 min	15,000 min
130°C		7.3±1.0		(5.2)
200°C		6.2±0.8	7.0±1.1	
250°C	(8.7)		6.2±0.3	
300°C	(7.0)	7.8±0.4	(6.7)	
350°C		6.7±0.3	6.4±1.1	
400°C			8.7±0.8	
450°C	7.0±0.1			
500°C	recrystallized			

Table II. Stacking-fault energy obtained by other authors on Cu-Ge alloys.

Ge wt.%	stacking-fault probability	stacking-fault energy (erg/cm^2)	reference
0 (Cu)	3.3×10^{-3}	40 (assumed)*	(5)
3.0	5.0×10^{-3}	27	
6.0	14.5×10^{-3}	9.1	
8.6	$31.7\text{-}37\times10^{-3}$	4.2—3.6	
0 (Cu)		(165)—102	(6)
1.76		97—117	
6.00		45— 58	
7.94		2— 9	

Calculated by the present authors, assuming the energy of pure copper as 40 erg/cm^2.

Another suggestion made by S. Weissmann[3] and also P. R. Swann[4] is that these long extended dislocations are an early stage of twin formation. This idea is very attractive, but its proof is difficult at present.

The stacking-fault probability and the energy of Cu–Ge alloys were obtained by two authors[5),6)] as shown in Table II. In these values, Haasen and King's result[6] is hardly plausible, because their work is mainly concerned with the stress-strain curves of Cu–Ge alloys and the estimation of stacking-fault energy from these curves were not their important object. On the other hand, Smallman and Westmacott[5] tried to obtain the stacking-fault probability by X-ray diffraction method. Their result is almost consistent with the present result obtained by X-ray method. Then it seems that the value of stacking-fault energy depends upon the technique used. At present, the authors cannot conclude which is the most reliable technique to get stacking-fault energy. Therefore, some only tentative considerations will be made.

In the X-ray method, the authors assumed the stacking-fault energy of copper as 40 erg/cm^2. If this value was taken to be 120 erg/cm^2, as Seeger[7] described, the calculated values become three times larger. But such a large value for copper is not plausible, because some ambiguities are present in his method. However, the X-ray method itself also has following ambiguities. The first is concerned with the existence of long extended dislocations and the second is the existence of deformation twins. These two may affect the peak shift of diffraction lines and some errors will be introduced to the calculation of stacking-fault probability. Even if the existence of twins does not affect the peak shift, as Wagner[8] proved theoretically, and also the authors could not observe an asymmetry of the profile of diffraction lines due to twins predicted from his theory, the existence of non-equilibrium extended dislocations in cold worked state may affect the stacking-fault probability to become larger. Then the result obtained by X-ray method can be considered to give a lower limit of the stacking-fault energy of the alloy.

On the other hand, the deduction of the energy from the curvature of dislocation nodes is very simple, and the formula used is very clear. However, there is a following question. As the networks observed are very small and the distance between nodes is also generally very small, so stresses from the neighbouring dislocation lines might give an effect to nodes. It may constrict the expansion of nodes. Therefore, the value of stacking-fault energy obtained by this method might have a tendency to become too large, and will be considered to show an upper limit.

In conclusion, the true value is supposed to be between two values obtained by two methods.

References

1 B. E. Warren and E. P. Warekois: Acta Met. **3** (1955) 473.
2 M. J. Whelan: Proc. Roy. Soc. **A249** (1958) 114.
3 S. Weissmann: Private communication.
4 P. S. Swann: Private communication.
5 R. E. Smallman and K. H. Westmacott: Phil. Mag. **2** (1957) 669.
6 P. Haasen and A. King: Z. Metallk. **12** (1960) 155.
7 A. Seeger, R. Berner and H. Wolf: Z. Phys. **155** (1959) 247.
8 C. N. J. Wagner: Acta Met. **5** (1957) 427.