phonon wavelength as well as the glide plane spacing of the dipole. Considered as a line (phonon wavelength comparable to glide plane spacing), a dipole is both a center of lattice rotation and of mechanical polarizability (low shear modulus). For shorter phonons it is two separate dislocations with modified strain fields. Also, it may have some point character at its ends; and at any orientation it junctions along its length.

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An Experimental Study on Electrical Resistivities of Vacancy, Dislocation and Stacking Fault in Aluminum

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The change in electrical resistivity of super pure aluminum due to the lattice defects introduced by quenching and by deformation is correlated with the kinds and concentrations of defects obtained by transmission electron microscopy. The resistivity of an ordinary dislocation does not come from the stacking fault but mainly from the dislocation itself. The specific resistivities of vacancy and dislocation and the resistivity of stacking fault are found to be $10^{-26}\Omega$ cm, $10^{-19}\Omega$ cm and $10^{-13}\beta\Omega$ cm^2 respectively, for stacking fault density β cm^{-1}.

§ 1. Introduction

There has been considerable interest in the discrepancy between the calculated and experimental values for the increment in electrical resistivity due to dislocations in deformed metals. Seeger[1] and Howie[2] have explained this discrepancy by the resistance due to the stacking faults separating the partial dislocations.

The decrement of electrical resistivity by the disappearance of tetrahedra containing the stacking fault in quenched gold foil was measured by Cotterill[3] and this resistivity decrement was interpreted as the annealing out of the stacking fault.

Recently it has been found that almost all dislocation loops in quenched pure aluminum contain a stacking fault in spite of its high stacking fault energy[4]. Using this fact, the resistivity of the stacking fault would be separated from that of dislocation itself. With the aim to determine which is mainly effective to the dislocation resistivity, dislocation itself or stacking fault, the electrical resistance measurements have been carried out on quenched and on deformed aluminum, together with the direct observations of the defects by transmission electron microscopy.

§ 2. Deformation Experiments

Single crystals of 99.998% pure aluminum, which were prepared by Fujiwara's stress-annealing method, were used for the deformation experiments. Those single crystals had their lengthwise axis in the direction shown in Fig. 1 and are considered to be deformed

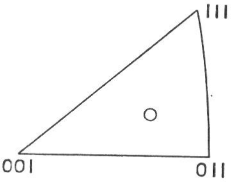


Fig. 1. Crystallographic orientation of lengthwise direction of crystals for deformation experiment.

predominantly by the single glide in the early stage of elongation. The specimens for resistivity measurement were single crystal wires of 1.6 mm in diameter, on which four pieces of fine wires of the same material were spot-welded, two for potential leads and two for current leads. The distance between potential leads was about 7 cm.

The specimens were deformed in tension using a tensile testing machine specially designed and built in our laboratory. The load-extension curves were recorded during testing by a load cell and counters which were geared to the machine. The electrical resistance measurements were made at liquid nitrogen temperature using a potentiometer, and the wire of the same material was used as a dummy in order to avoid the fluctuation of the measurement due to the temperature change.

The crystals were extended at liquid nitrogen temperature 3.7%, 10% and 14%, and their resistance changes were measured just after unloading. Assuming that the cross-section of the crystals change uniformly, the change in the electrical resistivity $\Delta\rho$ due to the deformation was calculated and given in the second column in Table I. After these cold-worked crystals were kept at room temperature for about one day, the resistance changes were measured again, and the resistivity decrease $\Delta\rho_1$ by this room temperature annealing was also given in the third column.

Table I

percentage of elongation (%)	$\Delta\rho$ ($10^{-9}\Omega$ cm)	$\Delta\rho_1$ ($10^{-9}\Omega$ cm)	$\Delta\rho_2$ ($10^{-9}\Omega$ cm)	N (10^9 cm^{-2})	ρ_d ($10^{-19}\Omega$ cm)
3.7	1.45	0.34	1.11	1.7	6.5
10	4.06	1.73	2.33	3.8	6.1
14	6.34	3.05	3.29	5.0	6.6

$\Delta\rho$: Total resistivity increase. $\Delta\rho_1$: Resistivity decrease by room temperature annealing. $\Delta\rho_2 = \Delta\rho - \Delta\rho_1$. N: Dislocation density. ρ_d: Specific dislocation resistivity.

The transmission electron microscopy was used for the determination of the density of dislocations introduced by the deformation using single crystal plates of 0.5 mm thick. The specimens which were extended by similar method described above were electro-thinned in a mixture of methyl alcohol and perchloric acid (4:1). The typical dislocation configuration for the specimen extended to 14% is shown in Fig. 2. Using the method of Smith and Guttman[5] or direct counting method, the dislocation densities N were estimated and given in the fifth column in Table I.

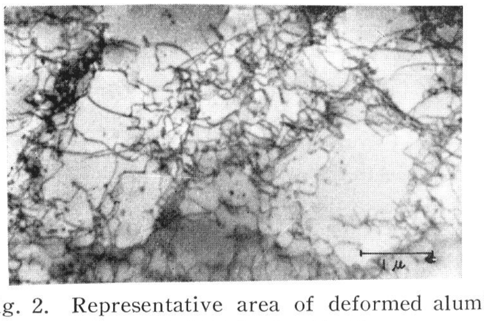
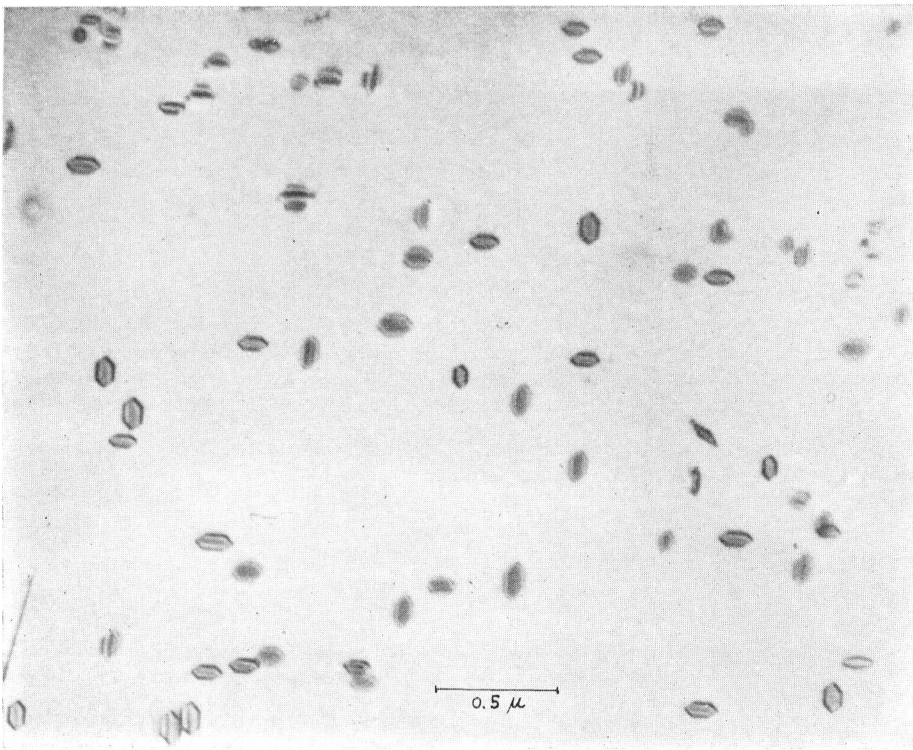


Fig. 2. Representative area of deformed aluminum. Elongated 14% in liquid nitrogen.

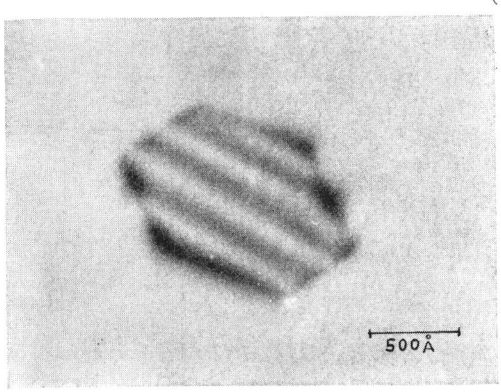
§ 3. Quenching Experiment

The material used was super pure aluminum zone-refined in vacuum, whose ratio of electrical resistivity at room temperature to that at 4.2°K was 6000 or more. The specimen used for transmission electron microscopy were thin films of 100μ in thickness which were annealed for ten hours at 630°C. The specimen was kept at 550°C for one hour in a vertical furnace and then quenched into ice water at 0°C. The specimens, which were aged for ten hours at room temperature after the quenching, were electro-thinned in the same mixture as used in the deformation experiment. The typical electron micrographs are given in Fig. 3 (a) and (b). It should be noted here that the defects generated by the clustering of quenched-in vacancies were the hexagonal Frank sessile dislocation loops which contain a stacking fault. The average size and the density of the loops were 1300Å and 3×10^{13} cm^{-3} respectively.

The specimens which were aged at room temperature after quenching from 550°C, furthermore, were bulk annealed at several temperatures for 15 minutes. Frank sessile dislocation loops shrunk still preserving the stacking fault in them by the annealings and finally they disappeared at about 200°C. The change of the average size of the remaining loops and their density for those isochronal annealings are shown in Table II. The density of stacking faults was calculated from this table and plotted in Fig. 4 against the annealing temperature.



(a)



(b)

Fig. 3. (a) Representive area of quenched aluminum from 550°C to 0°C. All dislocation hexagons contain stacking faults. (b) A dislocation loop containing stacking fault. Note the fringes caused by stacking fault.

Table II

annealing temp. (°C)	average size (Å)	density (cm^{-3})
(not annealed)	1300	3×10^{13}
140	1200	2×10^{13}
160	1200	7×10^{12}
170	1100	2.5×10^{12}
180	950	1×10^{12}

The change of size and density of dislocation loops by bulk annealing.

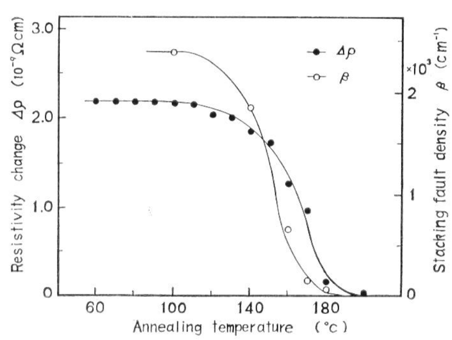


Fig. 4. The changes of stacking fault density and resistivity by isochronal annealing.

The resistance changes due to the quenching were measured on wires of 0.3 mm in diameter of the same material as above. The measurement apparatus of electrical resistance and the methods of quenching and aging were described in detail in a previous paper by T. Kino and S. Kabemoto[6], so described briefly here. The heating of specimen before quenching was made by direct current through the specimen, and the resistivity measurements were carried out in stirred oil bath regulating the fluctuation of temperature within 0.01°C. Aging at room temperature was made in the same oil bath, and the annealing above room temperature was carried out in a silicon oil bath. The resistivity change was calculated from the resistance difference between a specimen and dummy.

The decrement in resistivity during the aging at room temperature was about $30\times 10^{-9}\Omega$cm on the specimen quenched from 550°C, but this value is ambiguous because of the rapid rate of resistance decrease immediately after quenching.

Specimens fully aged at room temperature subsequent to the quenching from 550°C were annealed at higher temperature. The decrease in resistivity in the isochronal annealing for 15 minutes at several temperatures is given in Fig. 4. The total amount of resistivity decrease in this annealing stage was $2.2\times 10^{-9}\Omega$ cm.

§ 4. Discussion

a) *Dislocation Resistivity and Stacking Fault Resistivity*

The decrease in resistivity $\Delta\rho_1$ of the deformed specimens by the annealing at room temperature is considered to be due to the annihilation of vacancies which were introduced by the deformation. The part $\Delta\rho_2(=\Delta\rho-\Delta\rho_1)$ which is not annealed out by this room temperature annealing should be attributed to the dislocations generated by deformation including stacking fault separating the dislocations into two partials.

On the other hand, the resistance decrease of the quenched specimen by the annealing at about 200°C may correspond to the annihilation of dislocation loops containing stacking fault, because there is a close similarity between the annealing out of the loops in the direct observation and the resistivity decrease as shown in Fig. 4.

Dislocation introduced by deformation is considered to be separated into two partial dislocations containing a stacking fault between them. In aluminum, it may be reasonable to assume that the separation of two partial dislocation is the order of 10Å, because aluminum is thought to have a relatively high stacking fault energy. The stacking fault density β thus estimated is given in Table III together with the dislocation density N and the corresponding resistivity increase for the deformation of 14%.

Table III

	resistivity change ($10^{-9}\,\Omega$ cm)	N (10^9 cm^{-2})	β (cm^{-1})
deformation	3.29	5.0	500
quenching	2.2	0.78	2200

N: Dislocation density. β: Stacking fault density.

Hexagonal stacking faults in quenched aluminum have a Frank sessile dislocation surrounding them, and the stacking fault density β and the Frank sessile dislocation density N are also given in Table III with the resistivity increase. The values in Table III show that resistivity change by deformation is larger than that by quenching even though the area of stacking fault is smaller in deformation. It is concluded, therefore, that the contribution of ordinary dislocation to the electrical resistivity is mainly due to the dislocation itself and not to the stacking fault. Resistivity contribution from dislocation itself and that from stacking fault can be separated using Table III, and the specific resistivity ρ_d of dislocation and the resistivity ρ_{sf} of stacking fault can be estimated as $6\times 10^{-19}\Omega$ cm^2 and $4\times 10^{-13}\,\beta\Omega$ cm^2 respectively, where β is the stacking fault density. Using these values of resistivity, the contribution of stacking fault to the resistivity of ordinary dislocation is less than several per cent of the total. Here the contributions to the resistance from the different kinds of dislocations are assumed to be the same, because square of the Burgers vector of Frank sessile dislocation is the same as the sum of that for Shockley partial dislocation. The values of specific resistivity here are not decisive and more accurate experiments are desired.

Clarebrough *et al.*[7] showed recently that the dislocation resistivity estimated from their experimental results differs very little in copper, silver, and gold, and arrived to the same conclusion as the present work that stacking faults do not make an important contribution to the resistivity of deformed metals. In the experimental data by Cotterill in quenched gold foil, the resistance change due to the disappearance of the tetrahedra is roughly proportional to the total length of stair-rod dislocations at the edge of tetrahedra but not to the total area of stacking fault. Therefore his data can be also explained by the same idea as the present work.

b) *Vacancy Resistivity*

The increase in resistance just after the quenching, which is equal to the sum of the resistance decreases by the room temperature annealing and by the annealing near 200°C, is considered to be due to the vacancies which were introduced by the quenching. The concentration of this quenched-in vacancies can be calculated from the density and the average size of dislocation loops which are thought to be formed by the vacancy condensation. From the resistance increase above and this vacancy condensation, the specific resistivity of vacancy in aluminum is about $0.9\times10^{-26}\Omega$ cm. This value is large compared with the result of Simmons and Balluffi[8] and might be due to the underestimation of vacancy concentration. Actually uncertain defects, of higher concentration than that of dislocation loops, were observed and they might be three dimensional aggregates of vacancies.

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DISCUSSION

Saada, G.: What was the smallest radius of loop you could measure?

Yoshida, S.: It was about 200Å.

Saada, G.: Did you measure the stress strain curves in your tensile experiments and will you publish them?

Yoshida, S.: Yes, we will publish them elsewhere.

Quéré, Y.: In the figures given for the resistivity of dislocations, do you take into account the existence of point defects produced by cold work?

Yoshida, S.: Deformed crystals were kept at room temperature for about one day to eliminate point defects and, then, the resistivity was measured.

Seeger, A.: I should like to mention that there is very satisfactory agreement between your results and our recent work at Stuttgart. Seeger and Statz (Physica status Solids (July 1962)) and Statz (to be published) have reconsidered the electrical resistivity of stacking faults and have found it to be the same order of magnitude as reported here on aluminium. A discrepancy exists with Cotterill's measurements on gold, which give a larger stacking-fault resistivity than can be accounted for by the theory, which is believed to be rather well formed.

The conclusion that the electrical resistivity of dislocations in aluminium comes mainly from the strain field and not from the stacking fault ribbon, agrees well with the calculation of Seeger and Bross (Z. Naturfor. (1960)) who showed that the strain field scattering from edge dislocations is at least one order of magnitude larger than was supposed by Hunter and Nabarro. The theory shows the fact that for an isolated edge dislocation the resistivity would be logarithmically divergent.