Change of Dislocation Density in Aluminium and Lithium Fluoride after Annealing near the Melting Point under Hydrostatic Pressure

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This paper describes an experimental investigation of the change in etch-pit density (ρ) in aluminium and lithium fluoride crystals after anneals of varying duration (up to 10 h) near the melting point, either without pressure or under hydrostatic pressures of up to 10 kbar.* It is found that, when the temperature is high enough (near the melting point), the action of pressure promotes lowering of the etch-pit density after the anneal. For instance, in the case of aluminium, the reduction in ρ after a 10 h anneal at 630° C under a pressure of 10 kbar is 100 times greater than under the same conditions without pressure. But for 550° C annealing temperature, the influence of pressure on change in ρ is already insignificant. In the case of lithium fluoride, annealing at 810° C (6 h) under a pressure of 8 kbar diminishes ρ by a factor of 30. At 750° C, the application of 8 kbar pressure reduces ρ only by a factor of 5 compared with atmospheric pressure.

The effect of pressure, and its temperature dependence, is qualitatively explained in the following way. Under pressure near the melting point, the microcracks, which always exist in solids, are healed (i.e. sintered). The inner surfaces of these microcracks serve as sources and sinks for Schottky vacancies. Therefore, during the establishment of an equilibrium vacancy concentration, when the microcracks are healed, dislocations begin to act as vacancy sources (or sinks) and this results in the acceleration of their climb and consequential annihilation (i.e. a reduction in dislocation density).

The comparison of literature data on dislocation density (*N*) in pure aluminium samples cooled (after anneal) at different rates, and of etch-pit density for similarly treated aluminium samples shows that, under our experimental conditions, ρ and *N* coincide in order of magnitude.

1. On the Stability of Dislocation in Crystals

As is well known [1], the dislocations existing in crystalline solids are thermodynamically unstable entities. Long anneals at high temperatures (near the melting point), which promote the approach to thermodynamical equilibrium, result in a diminution of the dislocation density *N*. But, in metals and in most ionic crystals, *N* only decreases to a definite value (10⁴ to $10^{5}/\text{cm}^{2}$, and sometimes only $10^{6}/\text{cm}^{2}$); after *1 bar = $10^{6} \text{ dyn/cm}^{2} = 750 \text{ torr.}$ this, any further diminution of the density, if it takes place at all, proceeds very slowly. Arrays of dislocations, such as small-angle boundaries, are also fairly stable against heating. It is supposed that the diminution of N during heating is a result of climb and annihilation of dislocations; this explains the reduction of the annihilation rate at smaller density, because the probability that a dislocation, after climb, meets another dislocation with the opposite sign is proportional to N^2 (i.e. diminishes rapidly as N decreases).

It was shown recently [2] that the dislocation density N varies with time t during an isothermal anneal according to the law:

$$1/N = 1/N_0 + At$$
 (1)

where N_0 is the initial density and A is a constant. This corresponds to the relation

$$\mathrm{d}N = -AN^2\mathrm{d}t \tag{1a}$$

which one may thus consider as experimentally confirmed.

In reference 3, it is stated that the value of N for very pure (zone-refined) aluminium may be considerably reduced by slow cooling (2 to 4° C/h), after heating to a temperature lying slightly above the recrystallisation threshold. The mechanism of this process has not been determined so far.

The influence of hydrostatic pressure on the stability of dislocations is not clear *a priori*, since pressure may, in principle, cause different and opposite effects. First of all, pressure lowers the equilibrium concentration, *c*, of vacancies by a factor of $\exp(-p\Delta v/kT)$:

$$c_{p,T} = c_{0,T} \exp(-p\Delta v/kT)$$
(2)

where k is the Boltzmann constant; T, the absolute temperature; p, the pressure; and Δv , the volume change connected with vacancy formation (Δv is approximately the volume of one vacancy).

Further, the pressure raises the activation energy for vacancy migration and thus slows down their migration. Both the noted effects slow the climb of dislocations and so promote their stability.

At the same time, the elastic energy of a dislocation is raised under hydrostatic pressure, since the formation of a dislocation is accompanied by a volume increase of the body. For this reason, the stability of dislocations is diminished under pressure. It is to be noted that, on the approximation of linear elasticity, the formation of dislocations is not connected with any volume change of the body; therefore, the elastic energy of a dislocation should not alter in the presence of pressure. The actual observed decrease of the density when dislocations appear depends on non-linear effects near the core of dislocations. Theoretical calculations [4, 5] show that non-linear deformation, appearing near the dislocation core, results in an increase in crystal volume that is equal to about

one atomic volume for each interatomic distance along the dislocation line. By Lomer's estimate [6], the volume increase, ΔV , of the deformed crystal caused by the presence of dislocations is proportional to the elastic energy of deformation, ΔE , and may be represented with the aid of the formula

$$\Delta V = 2K_{\gamma}\Delta E \tag{3}$$

where γ is the Grüneisen constant and K is the compressibility.

Comparative, precision, density measurements of annealed and deformed crystals [7-9] generally confirm the above-stated considerations. It is experimentally verified, for example, that the density of comparatively perfect monocrystals of copper and aluminium [8] and dislocation-free silicon [9] diminishes as the dislocation density in them increases. It should be noted, however, that the observed density values are partly affected by the raised vacancy concentration in the deformed crystals.

Another effect that has an influence on the stability of dislocations consists in the action of the pressure on the microcracks and microcavities always existing in solids; these defects may "heal" or sinter (after bringing together their opposite surfaces under the action of pressure). Thus, the establishment of the equilibrium concentration of vacancies at high temperatures may become difficult, because the internal (Schottky) vacancy sources, which usually are the surfaces of microcavities, fall off. This should stimulate the action of available dislocations as sources of vacancies and, accordingly, the climb of edge dislocations, followed by their annihilation.

The formation of an equilibrium concentration of vacancies exclusively from dislocation sources may diminish the dislocation density very noticeably. Thus, if, near the melting point, the vacancy equilibrium concentration cis $\sim 10^{-3}$, and if the available dislocations are represented by circular loops of radius R (the ends of a loop lying at a distance 2R in the surface of the body, the dislocation density N being $\sim 1/4R^2$ per unit surface area, or the loop density about N/R per unit volume), then the volume V(per unit volume of the body) connected with incomplete parts of atomic planes in edge dislocations is approximately represented by $V \sim$ $\pi NR^2b/2R$ (b is the atomic diameter); that is $V = \pi b/8R$. Thus, when we have $R > \pi b/8c \sim$ 10^{-5} cm or $N < 2.5 \times 10^9$ /cm², all the available

dislocations may be displaced by atoms and may disappear, if they are the only sources for the equilibrium vacancies that must be formed near the melting point.

2. Experimental Conditions

In the present work, the results of an experimental investigation of the dislocation density N in aluminium and lithium fluoride and the change in density after annealing under hydrostatic pressure are given. For this purpose, the etchpit density ρ was observed. (The relation between N and ρ is discussed below.)

Coarse-grained polycrystalline samples of aluminium with total impurity content $\leq 10^{-2}$ % and lithium fluoride monocrystals of 99.9% purity were used.

The anneal of the samples under high pressure was performed in two alternative devices. One of these, which was utilised for some of the aluminium samples, is schematically shown in fig. 1. Here each sample (1), shaped as a cylinder



Figure 1 Scheme showing how hydrostatic pressure was applied in the first apparatus.

passing to a 90° cone, was tightly fitted into a massive matrix (2) made of a refractory chromium/nickel/titanium alloy steel. The matrix had an aperture reproducing the shape of the sample. With the aid of a plunger (3) made of refractory alloy steel, the necessary pressure was applied to the sample. In the working (conical) part of the sample, the stress system approximated to pure hydrostatic compression [10, 11]. The whole assembly (the matrix with the samples and the lower part of the plunger) was surrounded by an electrical oven that allowed the temperature to rise. In the same matrix, a control specimen (4) was located, which was annealed without pressure. The anneal proceeded in a vacuum enclosure, described elsewhere [12], at pressures up to 4.5 kbar (the load applied to the plunger was 50 kg, the diameter of the working part of the plunger being 1.2 mm; for samples with a 2 mm diameter, loads of up to 150 kg were employed). After an anneal of the required duration at the desired temperature (as a rule, at 630° C), the matrix with the samples was carefully cut along a plane passing through the working part of the sample annealed under pressure.

The alternative apparatus consisted of a press-mould that was mounted between the cheeks of a hydraulic press. The pressuretransmitting medium was pyrophillite. The heating was produced by a current passing through a graphite heater. With a pyrophillite holder of diameter 0.8 cm, a pressure up to 10 kbar could be achieved and a temperature of $\sim 1000^{\circ}$ C could be reached. In this apparatus, all the experiments with lithium fluoride crystals were performed, also some of those (at the higher pressures) with the aluminium samples.

The preparation of a surface for microscopic investigation (before and after anneal) included, in the case of the aluminium samples, mechanical polishing, chemical dissolution of the deformed layer, and electropolishing (the bath was a mixture of acetic and perchloric acids, the current density ~ 0.8 A/dm²). To reveal the dislocations, chemical etching was used. The etching reagent had the following composition*: HCl, 45 wt %; HNO₃, 18 wt %; HF, 10 wt %; methyl alcohol, 27 wt %. The samples investigated were previously treated with a 40% aqueous solution of KOH and, after thorough washing, they were etched to determine the dislocation density. The etching took 10 to 15 sec.

The dislocations in lithium fluoride were revealed by etching with an aqueous solution of iron chloride (5 to 10%); the time of etching was 5 to 15 sec.

Since the N (ρ) values show some scatter in different volume elements of the body, we applied the following procedure for finding the average value of ρ . For each sample, the ρ quantities were measured, as a rule, on three flat sections that had been cut ~ 300 μ m apart.

*This composition was proposed by E. E. Badyan in our laboratory. The difference between the action of the indicated etching reagent and others described in the literature [15] is mainly in the speed of dissolution.

On each section, there were investigated, under the microscope, three to four different areas at the magnification 200 to 400 in the case of aluminium, and 70 to 240 in the case of lithium fluoride. The average values of ρ are given in tables I and II (averaged for all the sections and areas on them); the scattering limits, $\Delta \rho$, of ρ are also given.

It has been found in some investigations [14] that the number of observed etch pits depends not only on the dislocation density but also on the impurity content and distribution. Only the so-called "decorated" dislocations are revealed; thus, $\rho = NW$, where the "decoration probability" W is always less than unity. The exact values of W could not be determined in this work. But, under constant conditions of heat-treatment, the value of W should not alter,

and so the observed values of ρ will be proportional to the N values. Moreover, as is shown below, in our case the observed ρ values and the present N values are of the same order of magnitude, while the changes in ρ caused by the application of pressure exceed by an order of magnitude.

3. Results of Annealing Aluminium Samples under Pressure

In fig. 2, four photomicrographs are shown of aluminium samples that had been annealed for 10 h at 630° C under different pressures. A comparison of etch-pit density in these pictures distinctly shows that the pressure acting during the annealing considerably influences (other conditions being equal) the observed magnitude of ρ , which diminishes as the pressure rises.*

TABLE I Etch-pit density ho in aluminium samples after annealing. (The initial etch-pit density was \sim 6 \times 10⁶/cm².)

Sample No.	Pressure applied during			р	T	t	U	Average	Scatter
	Heating	Anneal	Cooling	(kbar)	(° C)	(h)	(° C /h)	ρ (cm ⁻²)	limits of ρ^{\dagger}
1	Yes	Yes	No	4.5	630	10	200	2×10^4	1 to 4×10^4
2	,,	,,	,,	10	,,	,,	,,	$3 imes 10^{3}$	2 to 4 $ imes$ 10 ³
3	,,	,,	Yes	4.5	,,	,,	,,	$7 imes 10^4$	3 to 8 $ imes$ 10 ⁴
4	No	,,	No	,,	,,	,,	,,	$1 imes10^{5}$	0.8 to 2 $ imes$ 105
5	,,	No	,,	0	,,	,,	,,	$4.5 imes 10^5$	3 to 6 $ imes$ 10 ⁵
6	,,	,,	,,	,,	500	,,	,,	$5 imes 10^6$	3 to 6 $ imes$ 106
7	Yes	Yes	,,	4.5	550	1	,,	$4 imes 10^5$	3 to 5 $ imes$ 10 ⁵
8	No	No	,,	0	,,	,,	,,	$6 imes 10^5$	4 to 8 $ imes$ 10 ⁵
9	Yes	Yes	,,	,,	630	10	40	$5.5 imes10^4$	4 to 7×10^4
10	,,	,,	,,	4	,,	,,	"	1×10^4	0.8 to 2 $ imes$ 104
11	,,	,,	,,	8	,,	,,	,,	$3 imes 10^{3}$	2 to 5 $ imes$ 10 ³

[†]The ρ values were measured for each sample, as a rule, in three plane sections that were $\sim 300 \,\mu\text{m}$ apart. Each section was investigated in several (three to four) different points, under the microscope at a magnification of 200 to 400.

TABLE II Etch-pit density ρ in lithium fluorid	crystals after annealing. (The initial	∣ etch-pit density was ~106/c	:m².)
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Sample	<i>p</i> ‡	T	t	U	Average	Scatter
No.	(kbar)	(°C)	(h)	(° C /h)	ρ (cm ⁻²)	limits of ρ
1	0	750	6	100	$5 imes 10^5$	3 to 7×10^5
2	4	,,	,,	,,	$1.5 imes10^{5}$	0.9 to 3 $ imes$ 10 ⁵
3	8	,,	,,	,,	$1 imes 10^5$	0.8 to 2 $ imes$ 10 ⁵
4	0	810	,,	,,	,,	,,
5	2	,,	,,	,,	$5 imes 10^4$	3 to 6 \times 10 ⁴
6	4	,,	**	,,	$1 imes10^4$	0.9 to $2 imes 10^4$
7	8	,,	,,	,,	$4 imes 10^{3}$	2 to 5 $ imes$ 10 ³
8	0	,,	**	40	4×10^4	4 to $7 imes 10^4$
9	4	,,	,,	,,	$6.5 imes10^{3}$	5 to 8 $ imes$ 10 ³
10	8	,,	,,	,,	3×10^3	2 to 5 $ imes$ 10 ³

‡In all these experiments, the pressure was applied during the heating and annealing, and was taken off before cooling began.

*A repeated heating to the same temperature (630° C) without pressure does not change the decreased etch-pit density (if the cooling rate to room temperature remains the same).



Figure 2 Photomicrographs of aluminium samples annealed for 10 h at 630° C under different pressures (\times 375); (a) 1 bar; (b) 1.5 kbar; (c) 3 kbar; (d) 4.5 kbar.

The quantitative data of this dependence are given in the graphs of figs. 3, 3a, and 4, and also in table I.

In fig. 3, the dependence of $\log \rho$ on \sqrt{t} is demonstrated, where t is the annealing duration at 630° C for various pressures, p. As is seen, ρ diminishes sharply in the early stages of annealing, later approaching a limiting value (which is dependent on the pressure). In fig. 3a, the same data are plotted to show the dependence of $1/\rho$ on t for three values of p, It is proved here that, under pressure, $1/\rho$ varies linearly with t (i.e. an equation like (1) is correct for unpressed material also). If the above-expressed considerations concerning the pressure action are right, we may expect that the constant A in (1) is proportional to $\exp(+p\Delta v/kT)$. Plotting log A (as determined from the experimentally found straight lines of $1/\rho$ versus t, at constant values of p) against p, we found a linear dependence with slope $\simeq 7 \times 10^{-10}$ cm²/dyn. From this, we obtain $(\Delta v)^{1/3} = 4.5 \times 10^{-8}$ cm, which has the right order of magnitude, although it is too high by a factor ~2.

The values of ρ_0 (for t = 0) for the different straight lines in fig. 3a do not coincide. This corresponds to a different change of etch-pit (or dislocation) density during heating to 630° C under the action of different pressures. Assuming that the heating rate was practically the same



Figure 3 Etch-pit density ρ of aluminium samples versus (time of anneal)¹/², at 630° C, under different pressures: (1) 1 bar; (2) 1.5 kbar; (3) 4.5 kbar.



Figure 3a Values of $1/\rho$ versus time of anneal *t* (*T* = 630° C) for different pressures: (1) 1 bar; (2) 1.5 kbar; (3) 4.5 kbar.

in all experiments, one may expect the dependence of ρ_0 on p to be given by the factor $\exp(-p\Delta v/kT)$. The value of $\Delta \log \rho_0/\Delta p$ found from the experimental data of fig. 3a is $\sim 8.6 \times 10^{-10} \text{cm}^2/\text{dyn}$. This corresponds to the quantity $(\Delta v)^{1/3} = 5 \times 10^{-8}$ cm, which, as mentioned above, is about two times higher than the



Figure 4 Etch-pit density ρ in aluminium samples versus pressure p during annealing at 630° C: (1) t = 30 min, $U = 200^{\circ}$ C/h; (2) t = 10 h, $U = 200^{\circ}$ C/h; (3) t = 10 h, $U = 40^{\circ}$ C/h.

expected value for the linear dimension of a vacancy.*

The dependence of ρ on p (for different, constant, annealing times) is given in the graph in fig. 4.

The data given in figs. 3 and 4 show that annealing under a pressure of 4.5 kbar allows us to obtain, in aluminium, an etch-pit density value $(2 \times 10^4/\text{cm}^2)$ that is less, by almost an order of magnitude, than that appearing after an anneal without pressure. Taking into account the character of the pressure dependence of ρ , one could expect to obtain aluminium crystals with a still smaller etch-pit (dislocation) density. And, indeed, in an experiment, carried out in the second apparatus, in which the pressure reached 10 kbar (annealing temperature 630° C and duration 10 h), a value of ρ of only 3 × 10³/cm² was found.

4. Supplementary Experiments with the Aluminium Samples

The above-stated results contradict the data of Hilliard *et al* [13]; they checked the dislocation density in aluminium of 99.99% purity, after annealing for 1 h at 550° C under a pressure of 40 kbar, and found no change in N under pressure (within a factor of 2 or less). The distinction between our experiments and those of Hilliard *et al* [13] is apparently connected with the lower temperature and shorter annealing duration in the earlier work. The different method of loading of the samples may also have

^{*}In the last calculation, T was taken as $T_{\text{max}} = 630^{\circ} \text{ C} \sim 900^{\circ} \text{ K}$, but the actual average "effective" temperature for dislocation absorption (for the whole heating period) is $T_{\text{eff}} = aT_{\text{max}}$, where a < 1, so the real $(\Delta v)^{1/3}$ value must be $a^{1/3}$ times smaller.

played a role. Hilliard et al increased the pressure proportionally to the temperature rise, and so the pressure was not supported constant at the maximum level throughout the time of heating and annealing. This was done with the aim of avoiding crystallisation of mineral oil serving as a pressure-transmitting fluid. As was stated above, dislocations may disappear if they are the only available sources of equilibrium vacancies (i.e. if the microcracks and microcavities, the free surfaces of which customarily serve as sources of vacancies, are fully "healed" or sintered under the action of high temperature and high pressure*). But if the sample is heated without pressure, and the pressure begins to act when the maximum temperature is reached, at first there will arise the normal vacancy concentration $c_{0,T}$ (on account of the customary sources). After pressure has been applied, there would be a surplus of vacancies, which (if other sinks are absent) should settle on the dislocations, promoting climb and annihilation of the latter.

Thus the formation of the equilibrium vacancy concentration may be realised with participation of dislocations not only when the pressure begins to act before heating is started, but also if it does not act until after the heating is finished and the maximum temperature is reached. But the change of the dislocation density will be different in the two cases and, in general, will depend on the moment in which the pressure starts to act.

To check the above considerations, we carried out some supplementary experiments, annealing aluminium samples under pressure (4.5 kbar for 10 h at 630° C), differing in respect of the stage when the pressure started and finished acting.

(a) The sample, as above, was exposed to pressure before heating was started; the pressure was supported during the heating to the maximum temperature (630° C), then also supported during the time of annealing at 630° C (10 h), and it was taken off before the start of cooling. (b) The samples were heated to 630° C without pressure; then the pressure (4.5 kbar) was applied and acted throughout the time of the anneal until the start of cooling.

(c) The action of the pressure on the sample (which was annealed at 630° C for 10 h) began before heating and was continued until the end of cooling to room temperature.

(d) This was a control sample heated and annealed without pressure.

The experiments showed (see table I) that: (d) gave the greatest etch-pit density $(4 \times 10^5 \text{ to } 10^5/\text{cm}^2)$; (a) gave the minimum $(1 \times 10^4 \text{ to } 2 \times 10^4/\text{cm}^2)$; (c) gave $7 \times 10^4/\text{cm}^2$; and (b) gave $1 \times 10^5/\text{cm}^2$. Thus, it was experimentally verified that the moment of application of pressure plays an appreciable role. The minimum final dislocation density is achieved when the full pressure is applied before heating begins and taken off before cooling begins.

For comparison with the results of reference 13, we also carried out a supplementary experiment in which an aluminium sample was annealed, both under 4.5 kbar pressure and without pressure, at 550° C for 1 h. We found etch-pit density values of $4 \times 10^5/\text{cm}^2$, under pressure, and $6 \times 10^5/\text{cm}^2$, without pressure. Therefore, at an annealing temperature of 550° C, and a duration of 1 h, the influence of pressure on the etch-pit (dislocation) density change proved to be very small (as in reference 13) and is within the range of scatter of the values of ρ .

5. The Results of Annealing Lithium Crystals under Pressure

In fig. 5, photomicrographs are shown of etched lithium fluoride samples after annealing for 6 h at 810° C under different pressures (up to 8 kbar). The corresponding data on change of ρ with p are given in fig. 6 and in table II, where the figures for samples annealed at 750° C are also presented.

As in the case of aluminium samples, it is observed that the etch-pit density ρ (after annealing) falls as the applied pressure p rises. This phenomenon is intensified when the temperature approaches the melting point. When 8 kbar pressure is applied, annealing at 810° C for 6 h diminishes the etch-pit (dislocation) density in lithium fluoride by 1.5 orders of magnitude more than in the case when pressure is not applied (and all other conditions remain the same). At an annealing temperature of 750° C (other conditions unchanged), the difference is only 5 times.

Our results on lithium fluoride also contradict the conclusions in reference 13. But in that work [13] the anneal of lithium fluoride samples (under a pressure of 40 kbar for 1 h) was carried

^{*}In so far as we observed a continuous fall of ρ with rising p, it is necessary to suggest that new microcavities are healed continuously, step by step, as p rises.



Figure 5 Photomicrographs of lithium fluoride crystals annealed for 6 h at 810° C under different pressures (×225): (a) 1 bar; (b) 4 kbar; (c) 8 kbar.

out at 500° C, which is too far from the melting point of lithium fluoride (842° C at p = 1 bar) to expect any sintering of the microcavities.



Figure 6 Etch-pit density ρ in lithium fluoride crystals versus pressure ρ during annealing: (1) $T = 750^{\circ}$ C, $U = 100^{\circ}$ C/h; (2) $T = 810^{\circ}$ C, $U = 100^{\circ}$ C/h; (3) $T = 810^{\circ}$ C, $U = 40^{\circ}$ C/h.

6. The Correlation between Etch-Pit Density and Dislocation Density

The above data unambiguously testify that hydrostatic pressure applied during annealing near the melting temperature promotes a reduction of dislocation density N. But the absolute values of N in the described experiments remain unknown, since the values of "decoration probability" W are not found. The determination of these last values is a difficult task in the region of the observed ρ data, as is the direct measurement of N. In references 2 and 3, to measure N, use was made of Lang's X-ray diffraction topography technique. But this technique is only satisfactory for comparatively perfect crystals with low N values ($N \sim 10^2$ to 10^{3} /cm²). For greater N, the resolution of the photographs becomes insufficient.

For evaluation of W, if only in order of magnitude, we made use of an indirect method; namely, we compared some literature data [3] for N in pure (99.999%) aluminium samples, cooled at different rates after annealing, with ρ values determined in our laboratory for similar aluminium samples of different purities. It was noted above that low cooling rate promotes diminution of dislocation density.

In fig. 7, data from reference 3 are given for N versus cooling rate U (=dT/dt) for aluminium samples that had been annealed at 510° C for 7 h and then cooled to room temperature. These data are approximately represented by the straight line A, which is drawn in fig. 7 exactly as





Figure 7 (A) dislocation density *N* and (B) etch-pit density ρ versus cooling rate *U* for aluminium samples. (1) – data from reference 3. (2–5) samples with different aluminium content: (2) 99.999%; (3) 99.84%; (4) 99.7%; (5) 99.0%.

in reference 3. In the same figure, data on etchpit density ρ are given which were obtained in our laboratory for aluminium samples (of different purities, including one metal like that used in reference 3) that had been cooled from 500° C at various rates (200 to 10° C/h). The ρ values are somewhat scattered, but they are satisfactorily grouped near the straight line B, which is located in the neighbourhood of A and may be very approximately considered as its continuation. This shows that the ρ values, measured as average etch-pit densities for a range of purities, coincide in order of magnitude with the N values, the two differing by a factor rarely exceeding 2. This gives reason to consider that, to a first approximation, the ρ data quoted (in the tables and in the text) characterise the N values also.

It is to be noted that the connexion between the cooling rate and etch-pit (dislocation) density given in fig. 7 corresponds only to the case when heating and cooling proceed without pressure. After an anneal under pressure, the relative reduction of ρ values is lower when U is decreased, as shown by several results given in tables I and II. The reason is not yet clear, since the origin of the reduction of ρ with decreasing cooling rate U is not understood.

7. Conclusion

All data given above show unambiguously that, under hydrostatic pressure, the reduction of dislocation density, during annealing near the melting point, is considerably intensified the greater the pressure and the nearer the temperature to the melting point.

So far this has been observed only in aluminium and lithium fluoride crystals, but it is probably a general rule for all crystalline bodies.

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