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DEFORMATION MECHANISMS OF METALS HARDENED BY DISPERSED INCOHERENT SECOND PHASE

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ABSTRACT

The hardening by incoherent precipitates is analyzed in the f.c.c. metals. The precipitates determine indirectly the rapid multiplication of the dislocations in the matrix. The hardening is related to the so formed dense tangles. The recovery of these tangles is made by cross-slip, climb and probably by some other complex thermally activated mechanisms.

I. INTRODUCTION

In recent years the investigation of dispersion strengthening has shown that the hardening of the metal depends mainly on whether the discrete second phase particles are sheared by the dislocations moving in the matrix. In this respect the classification of hardening by a coherent or incoherent second phase can be adopted since the coherent phase but not the incoherent phase is usually sheared.

Nevertheless, other factors, such as the size of the particles, influence the shearing. When a dislocation confined to slip in its glide plane encounters particles, it may expand between them, leaving residual loops around each particle or it may shear them. The stress necessary in the first case, known as the Orowan stress,[1] is $\tau_c \approx \mu b/d$, where μ is the shear modulus of the matrix, b the Burger's vector, d the interparticle distance in the slip plane. For a given volume fraction of precipitate, τ_c decreases as the size of the particles increases. In the second case the stress increases with the size of the particle. An upper limit for the size of the particles to be sheared, $r\ell$, can be obtained by equating the two stresses. The higher the interfacial energy the lower is the upper limit. Only very small incoherent particles, which have a high interfacial energy γ, can be sheared. For the aluminum-alumina system, taking $\gamma = 1000$ ergs/cm^2, an approximative calculation gives $r\ell = 15\mathring{A}$.

Therefore a glide dislocation encountering such incoherent particles will generally expand between them, or alternately bypass them by some more complicated mechanisms, like cross slip or climb. The strain

hardening of these alloys, at least for small enough deformations, is much higher than that of the pure matrix and intimately related to the substructures of the alloy matrix. It will be shown that the hardening can in effect be explained in terms of the elastic interactions of the dislocations in the matrix. The second phase particles play the indirect role of accelerating the increase in dislocation density with the strain, leading to steep strain hardening.

On the other hand, direct dislocation-particle interactions are expected to control the hardening by coherent particles.

It is the purpose of this paper to give a review of the results obtained on crystals containing uniformly distributed incoherent precipitates. The true elastic limit will be briefly considered, and emphasis will be placed on the variation of the flow stress with temperature for macroscopic plastic deformation.

II. ELASTIC LIMIT

The precipitates intersecting the glide plane are anchoring points for the dislocation. As mentioned before, according to Orowan,[1] the increase in the elastic limit relative to the pure matrix is $\approx 2t/bd$, where t is the line tension of the dislocation. The Orowan bowing applies provided the dislocations are confined in their motion to the slip plane. Depending on whether the precipitates are elastically harder or softer than the matrix, the dislocation is repelled or attracted by the precipitates and the anchoring is somewhat different in each case.[2] The Orowan criterion holds in both cases.[3]

The Orowan relationship has been verified for single crystals containing uniformly distributed small particles.[4-6] It must be pointed out that this law is expected to apply when the initial dislocation density is low enough for the particles to play the role of first obstacles.

Ashby,[7] using an idea of Hirsch,[8] proposed a bypass of particles by double cross-slip, as schematically shown on Fig. 1 for an edge dislocation. When the dislocation begins the Orowan bowing, the resulting screw components cross slip out of the slip plane under the action of the stress concentration near the particle. The cross slipped segments attract each other, resulting after annihilation in a prismatic loop behind the particle and jogs in the dislocation line. Ansell[11] has shown that restrictive geometrical condition, namely when $2r \leq d/10$ where r is the radius of the particle, affect the bypass by double cross slip. Experimental evidence of this mechanism is limited to the prismatic loops observed by electron microscopy.[7,9,10] Because this cross slip has to be thermally activated, the true elastic limit, if controlled by such a mechanism, has to vary more strongly with temperature than the elastic constants, which is in contradiction with experiment.[9] Nevertheless it will be shown later that cross-slip occurs extensively after some plastic straining.

III. TEMPERATURE VARIATION OF THE FLOW STRESS

We summarize here the results obtained by the author on the polycrystalline Al_2O_3-Al system (SAP)[12] and by Mitchell et al. on the

Al/Al_2Cu system.[14] The flow mechanisms involved can probably be generalized to alloys with incoherent precipitates in a f.c.c. matrix of high stacking fault energy. Electron microscope observations by Ruedl, confirming the plasticity mechanisms will be given later.

The composition and the structural parameters, as determined by electron microscopy, of the alloys are given in Table 1. While the Al_2Cu particles are roughly spherical in shape, the alumina particles in SAP are platelets with linear size between 200 and 2000Å, and a thickness of about 200Å; the values of r given in Table 1 for the mean planar particle radius, are in this case the radius of the circle of the same area as that of the mean intersection area of platelets by a random plane, i.e. $r = (2Le/\pi)^{1/2}$ where L is half of the mean linear size and e the thickness.[12] The temperature dependence of deformation mechanisms controlling the flow stress are now discussed.

3.1 Flow stress at low temperature (ABC in Fig. 1)

3.1.1 Forest process

In the range of low temperatures, and under small stresses for creep (or at low strain for tension at constant strain rate), the mobile dislocations cannot leave the glide plane; their glide, in pure f.c.c. metals, can be hindered both by long range internal stresses and by short range obstacles provided by the "trees", dislocations piercing their glide plane. The deformation is then controlled by the thermally activated jog formation during the cutting through those trees and by the movement in long range stress field.

The tensile curves for both Al/Al_2O_3 and $Al/CuAl_2$ systems, especially

for small strains, present a strain hardening rate which is much higher than that for polycrystalline aluminum[12-14] and largely depending on the temperature.

The variations of the tensile flow stress for a given strain rate, with temperature (for three strains increasing from 1 to 3), are shown in Fig. 2.

The curves of type (1) or (2) are similar to those of pure metals whose hardening is controlled by the forest process as mentioned before. The AB part on Fig. 1 corresponds to the thermally activated jog formation, whereas in the BC part, the forest opposes the development of loops only by long range internal stresses τ_i. In f.c.c. structures, the long range stresses are principally due to the attractive trees giving junction reactions with the mobile dislocations.[15]

An estimate of the energy of formation of a pair of jogs, $2U_{fc}$, can be deduced from the measurement of T_c,* according to Friedel's theory[2] as described in reference twelve. The results so obtained are gathered in Table 2, for Al/Al_2O_3, Al/Al_2Cu alloys, and for two purities of Al. Applying the forest theory to the thermal decrease of τ at low temperatures, we thus obtain for both alloys and aluminum a common value of $2U_{fc}$ namely $2U_{fc} = 0.37 \pm 0.05$ e.v. A better estimate of $2U_{fc}$ is given by a direct measurement of the activation energy beyond T_c;[2] the value so obtained, for SAP 11.7 is ≈ 0.35 e.v.,[12,13] in good

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*T_c is next to 173°K for SAP, for a strain rate of $2.7 10^{-4}$ s^{-1} and at small strains ($<2.10^{-3}$).

agreement with the former mean value, and of a reasonable order of magnitude for the energy of formation of a pair of jogs in aluminum. Thus, the activation energy is compatible with the forest process controlling the plastic deformation in these alloys as in pure metals.

The measurement of the activation volume from strain rate change tensile tests, allows the determination of the force-distance diagram, showing the elastic interaction between a repulsive tree (not giving a junction reaction) and a mobile dislocation. The results obtained by Mitchell et al. for Al/Al_2Cu and pure aluminum[14] and by the author for SAP and impure aluminum[12,13]* are shown on Fig. 3. The fact that these F-x curves differ but little for the SAP, Al-Cu, Al, gives an added evidence for the presence of the intersection mechanism for all materials.

3.1.2 Cross slip and self diffusion

For large strains (curve (3) in Fig. 1), the shape of τ/μ - T curve can no longer be explained solely with the mechanism of section 3.1.1. The absence of a plateau in the τ/μ - T curve seems to be due to a strongly thermally activated mechanism. In fact, measuring the activation energy, ΔH, at constant strain rate as a function of temperature, we establish that the change in shape of the τ/μ - T curves is reflected by the ΔH - T curves, Fig. 4 for SAP 11.7.[12,13] At small strains, ΔH flattens out at $\approx 2U_{fc}$ for $T > T_c$, suggesting a forest process; but for large strains, ΔH increases beyond T_c and reaches a plateau, at about 1.6 e.v. for high enough temperatures, clearly corresponding solely to the operation of a diffusion process. Below, the variation of ΔH

*The SAP matrix is in impure aluminum (99.5%).

with T probably corresponds to ill defined cross slip processes complicated by the effect of internal stresses. These phenomena should determine the relaxation of the internal stresses leading to the thermal decrease of τ. The observation by electron microscopy of plastically deformed specimens shows an homogeneous distribution of dislocations when the deformation is low, while after high deformations the dislocations are tangled and have a cell structure.[10,16,17] Cells in SAP were only observed for high deformations and beyond the plateau $\tau(T)$.

As we previously saw, the cross slip of screw dislocations should lead to a decrease in τ with increasing temperature, and for high strains, we can reasonably conclude that our observations agree with the idea that the cell formation is related to:

(a) the cross slip of screw dislocations,

(b) the appearance of stage III of the stress strain curve,

(c) the tensile stress decrease with temperature after the plateau.

Thomas et al.[18] suggested that the tangles and cells result from the multiplication of dislocations by operation of Frank-Read sources left by cross-slip of dislcoations adjacent to particles. This could explain for instance why the cells observed in TD-Ni appear to have corners located at the largest particles.[18] This mechanism of formation of cells is slightly different from the above--which is in fact the normal cell formation by cross-slip in pure metals--because requiring the further operation of sources resulting from cross-slip. Until more evidence is obtained, it can not be considered as firmly established. As will be shown in the next paragraph, the role of particles as multiplication

centers is evident but not well defined.

3.1.3 Strain hardening

The measurement of the athermal stress τ_i (part BC on Fig. 2) directly leads to an estimate of the density of dislocations with $\rho = \left(\frac{\mu b}{\beta \tau_i}\right)^{-2}$ after Saada's relations,[15] where $\beta \approx 4$ in f.c.c. structures.

On the other hand, when the forest process is controlling the deformation, the measurement of the activation volume v^* gives an other possibility of determination of ρ, namely $\rho \approx (1.5\ b^2/v^*)^2$.[12]

Obviously, a direct determination of ρ is feasible by transmission electron microscopy. The variations of ρ with the strain, as determined from the activation volume and electron microscopy by Ruedl, are given in Table 3, for SAP 4.5 deformed at room temperature.

For strains smaller than about 2%, it can be seen that the agreement between both methods is quite good. For high strains, when cross slip becomes operative and introduces a cell structure, the density as determined from the activation volumes, is on the whole close to the density as determined by electron microscopy in the cell walls (the density in the walls is between 2 and 4 times higher than the mean density). Thus, it seems that the activation volume is characteristic of a forest process for which the mobile dislocations have to cut through the cell walls.

The absolute values of the dislocation densities are high, such as are only achieved in pure metals after severe cold working without subsequent annealing. The high initial value of this density in SAP, prior to tensile strain is probably due to the fabrication process

(sintering and extrusion).

From the preceding study we can conclude that the hardening of these alloys has the same origin as in pure f.c.c. metals. (Dispersions in b.c.c. structures have not been seriously studied. The limited number of mechanical tests made by Sell et al.[19] on dispersions of oxides, nitrides and carbides in tungsten are not conclusive.)

Their larger strain hardening is due to a more rapid increase of the dislocation density with plastic strain, as shown on Fig. 5, after Mitchell et al. Figure 5 represents the variation of the distance between trees as a function of strain. Similar results were obtained by the author for SAP,[12,13] and Heimendahl and Thomas for TD-Ni.[18] For Al/Al_2Cu, $(d\tau/d\varepsilon)_T$ decreases with increasing particle spacing d, for a given volume fraction f; for SAP the work hardening rate increases with increasing f or decreasing d. Although no quantitative relation has been proposed, these results show that the particles are thus playing an indirect role of multiplying the dislocation density. Nevertheless the exact mode of multiplying is not well understood. The particles can be active by creating prismatic loops when dislocations by-pass them by cross-slip or by volume indentation effect. The particles can also act as barriers to moving dislocations nucleated at other sources, such as Frank-Read sources, grain boundaries and twin boundaries in TD-Ni. This explains why in both cases the cell size seems to be governed by the particle spacing.

An interesting feature appears when studying the grain size influence. As shown by Heimendahl and Thomas for TD-Ni,[16] and the author for SAP,[12]

the grain size has no influence on the flow stress at 0.1 or 0.2 pct tensile stress as well as on the ultimate tensile stress. Transmission electron microscopies taken by Ruedl on tensile strained recrystallized SAP show that for the same plastic strain, the dislocation density is about the same for as-extruded and recrystallized specimens. Thus in recrystallized speciemens, the dislocation density, initially low, increases very rapidly with deformation, reaching after a few tenths pct of strain the same dislocation density, and therefore the same flow stress, as in the as-extruded material.

Furthermore, the Fisher-Hart-Pry theory[20] of strain hardening, based on the result of planar dislocation loops around the dispersed particles is invalid, since as we saw, the role of the particles is to multiply the dislocations and enhance the cross-slip processes leading to the cell structures.

Ashby[17] proposed a somewhat more realistic model to account for the strain hardening of these alloys, based on forest intersection. The strain hardening was calculated assuming that the glide dislocations have to cut through the cell walls set up by the residual prismatic loops left by cross slip on the particles (c.f. paragraph 2). Nevertheless the assumed substructure is peculiar; even if such prismatic loops are observed it is difficult to ascribe the work hardening due to their presence only. On the other hand, the formation of cell configurations takes place only at high enough strains. At small strains, when the dislocations are uniformly distributed in the matrix this theory is questionable. A pure forest process has to be considered. Unfortunately

in this case the rate of increase of dislocation density with plastic strain is not clear.

3.2 Flow stress at high temperatures (Above C* in Fig. 2)

The internal stresses, related to the dislocation tangles are relieved at high temperatures by mechanisms common to pure metals (cross-slip and climb) and by some more complicated mechanism. This recovery is, however, strongly limited by the presence of the second phase, and the material will maintain high strength. The stability of the dislocation substructure is obviously a function of the dispersion stability; in this respect, oxide dispersions, which are very stable, will assure a good alloy dislocation substructure at high temperatures, and therefore high strength.

These recovery processes can be analyzed as a function of temperature from the spectrum of the activation energy as determined by tensile tests for different grades of SAP and polycrystalline impure aluminum (99.5%), Fig. 6.[21]

3.2.1 Cross-slip and self-diffusion

Between 100 and 350°C, for a strain of 2.10^{-3}, the movement of dislocations in the matrix is controlled by cross slip and climb, as in pure aluminum (the cross-slip--at same stress level--and self diffusion energies in pure aluminum are respectively 1.2 and 1.5 e.v.). The creep of TD-Ni (Ni + 2 vol.% ThO_2) can be also satisfactorily explained, below 0.5 Tm, by cross-slip,[22] and the high temperature creep of several recrystallized Ni-ThO_2 (1 to 3 vol.% ThO_2) by climb.[23] The creep of Al/Al_2Cu at high temperatures has also an activation energy close to

the self-diffusion energy in pure aluminum. The general equation for the strain rate resulting from the climb of edge dislocations by diffusion of jogs has to be applied.[2]

$$\dot{\varepsilon} = \text{cte}\ \frac{1}{h} \exp\left(-\frac{U_D}{kT}\right) \text{sh}\left(\frac{nb^3\tau}{kT}\right)$$

where U_D is the self-diffusion energy, τ the applied stress, n a stress concentration factor, and h the mean height that a dislocation has to climb before escaping by glide or annihilation. If the escape takes place at the head of a piled-up group on a second phase particle, h has to be of the order of the half height of the particle;[24] for a pile-up on intracrystalline barriers, h has to vary like τ^{-2}.

Measurements of the activation volume made by the author on SAP[21] are in reasonable agreement with the values expected from the previous equation. Cross-slip and climb can be operative at lower temperature (paragraph 3.1.2) for higher strain and therefore higher stress level.

3.2.2 Other processes

The high activation energies measured above 350°C show that the high temperature deformation mechanisms in SAP are radically different from that in aluminum, since in the latter the climb of dislocations is the controlling process up to the melting point, with a constant activation energy closed to the self diffusion energy.[25] The variation of the activation energy with the temperature is apparent, because the tensile tests were conducted under low stress at high temperature and vice versa. So the activation energy steeply increases with decreasing stress, as reported in references twelve and twenty one. Similar

activation energies have been observed by other investigators in SAP.[26,27] In fact, high values of activation energy, reaching at high enough temperatures ten times or more the self diffusion energy seem to be a general feature for dispersion hardened alloys; indium-glass composite,[28] TD-Ni,[23] Ni-Al_2O_3 alloys.[29] Nevertheless there is no general agreement concerning the stress dependence of the activation energy. Different models have been suggested depending on whether the apparent activation energy is a function of the stress.

a/ Grain boundary sliding

Wilcox and Clauer[23] explained qualitatively, in terms of grain boundary sliding, the independent of stress activation energy for creep in TD-Ni. The presence of impurities or particles in the boundaries would increase the activation energy for sliding to the observed value. Nevertheless the large stress exponent (n = 40) in the preexponential term of the strain rate remains difficult to explain.

b/ Dislocation generation from grain boundaries

Ansell and Weertman[27] developed a semiquantitative theory based on the generation of dislocations from grain boundaries. The activation energy of such a process is obviously strongly stress dependent, but the expected grain size dependence of the flow stress is not observed experimentally.[12,16]

c/ Particles by-pass by glide

The model, based on the Orowan mechanism, consists of thermally activated glide by-passing the particles intersecting the slip plane, for stresses lower than the critical Orowan stress. Calculations have

been made by Coulomb[3] for precipitates softer than the matrix (or bubbles) and the author[21] for precipitates harder than the matrix. In the last case, for small enough precipitates and for applied stress very near the critical Orowan stress τ_c, the activation energy is strongly stress dependent:

$$\Delta H \simeq 2td\ (1 - \tau/\tau_c)^{3/2}$$

The numerical values agree well enough with the experimental ones for SAP, but the calculated activation volumes are between 10 and 100 times greater than the experimental ones.[12]

d/ Thermal activation of junction reactions

In alloys deformed at high temperatures, the particle limited recovery can still lead to large dislocation densities. Hirsch type junction reactions[30] between attractive dislocations are frequently observed in this case.[10] The author has studied the thermal dissolution of such reactions.[31] The calculated activation energy and volume, which again vary rapidly with the applied stress, agree reasonably well with experimental data in SAP.

Even if the various models presented here are partially satisfactory, it is doubtful that a single model can at high temperatures uniquely describe the flow mechanism of this type of alloy. In any case the large value of the activation energy indicates a highly restricted dislocation mobility. Such alloys will therefore retain an appreciable flow stress, even at high temperatures.

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Table 1. Structure parameters of the alloys

alloy type	dispersed phase	f(%)	r(μ)	d(μ)	grain size (μ)
SAP 4*	Al_2O_3 γ	2.75	0.0253	0.208	
SAP 7	"	4.85	0.0293	0.182	2 - 5
SAP 10	"	7.00	0.0314	0.162	
4M	Al_2Cu	4.5	0.298	2.8	430
4C	"	4.5	1.08	11.4	410
5F	"	5.9	0.158	1.3	430

f = volume fraction of dispersed phase.

r = mean planar particle radius.

d = mean planar interparticle distance

*The number is the weight fraction of alumina.

Table 2. Energies of formation of a pair of jogs.

Alloy	SAP 4.5	SAP 11.7	4M	4C	5F	Al 99.5%	Al 99.9%
$2U_{fc}$ (e.v.)	0.43	0.42	0.35	0.41	0.34	0.33	0.37

Table 3. Dislocation densities as a function of tensile strain at room temperature. SAP 4.5

Strain ε%	mean density, cm^{-2} (electron microscopy)	mean density, cm^{-2} (activation volume)	distribution
0	$(5 \pm 2)10^9$		homogeneous
0.05	$(7 \pm 2)10^9$		"
0.2	$(1.05 \pm 0.3)10^{10}$	$(1.9 \pm 1.2)10^{10}$	"
1	$(1.2 \pm 0.3)10^{10}$	$(2.3 \pm 1.1)10^{10}$	"
2		$(3.6 \pm 1.0)10^{10}$	"
3	$(1.6 \pm 0.3)10^{10}$	$(4.9 \pm 0.9)10^{10}$	cell structure
5		$(6.2 \pm 1.3)10^{10}$	"
6	$(2 \pm 0.3)10^{10}$	$(6.9 \pm 1.1)10^{10}$	"

FIGURE CAPTIONS

Figure 1. Double cross-slip for an edge dislocation.

Figure 2. Variations of the tensile flow stress, for different strains, as a function of temperature (the flow stress is corrected for the variations of the shear modulus μ with temperature).

Figure 3. Force-distance diagram.

Figure 4. Variations of the activation energy function of temperature, at constant strain rate ($\dot{\varepsilon} \simeq 2.7.10^{-4}\ s^{-1}$) and for different strains. SAP 11.7.

Figure 5. Variation of 1/L with true strain.

Figure 6. Activation energy at constant strain rate as a function of temperature for SAP 3.6, 8.3, 11.7 and Al-99.5%. $\dot{\varepsilon} = 2.7.10^{-4}\ s^{-1}$. $\varepsilon = 2.10^{-3}$.

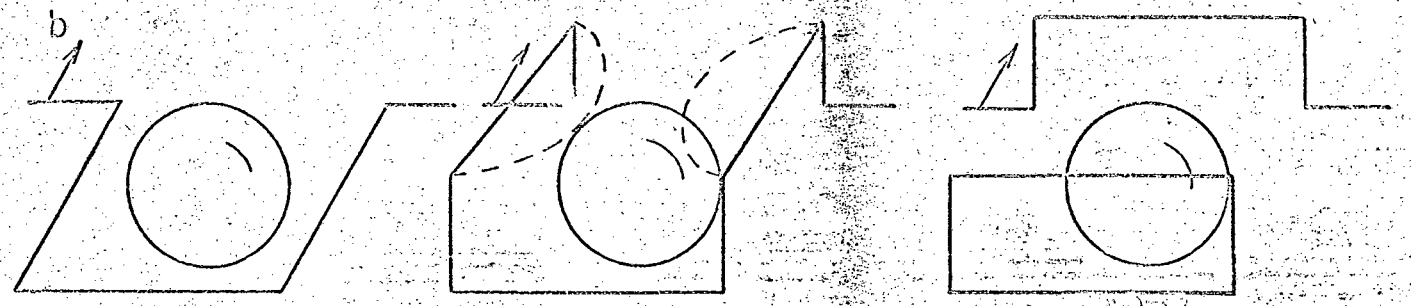

FIG. 1

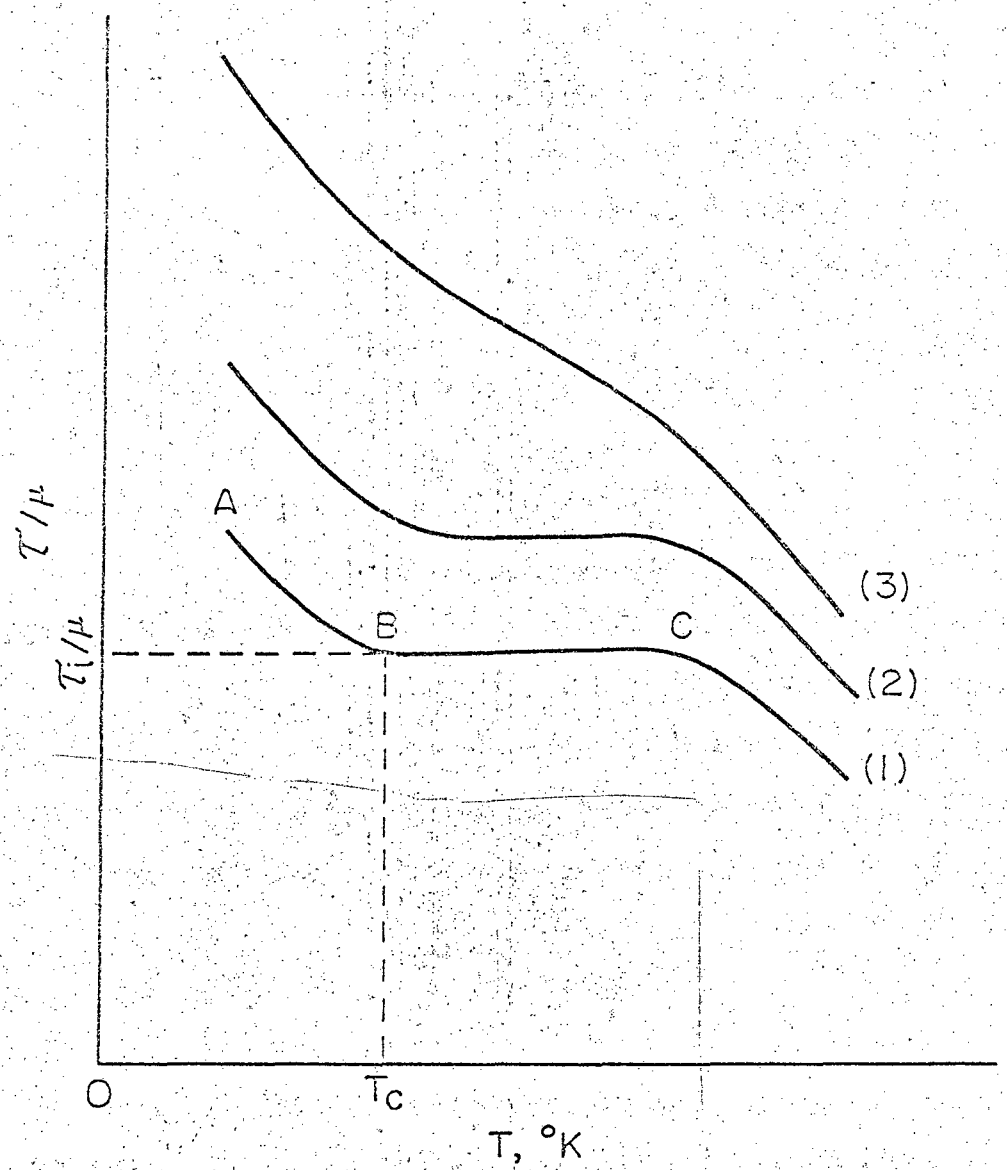

FIG. 2

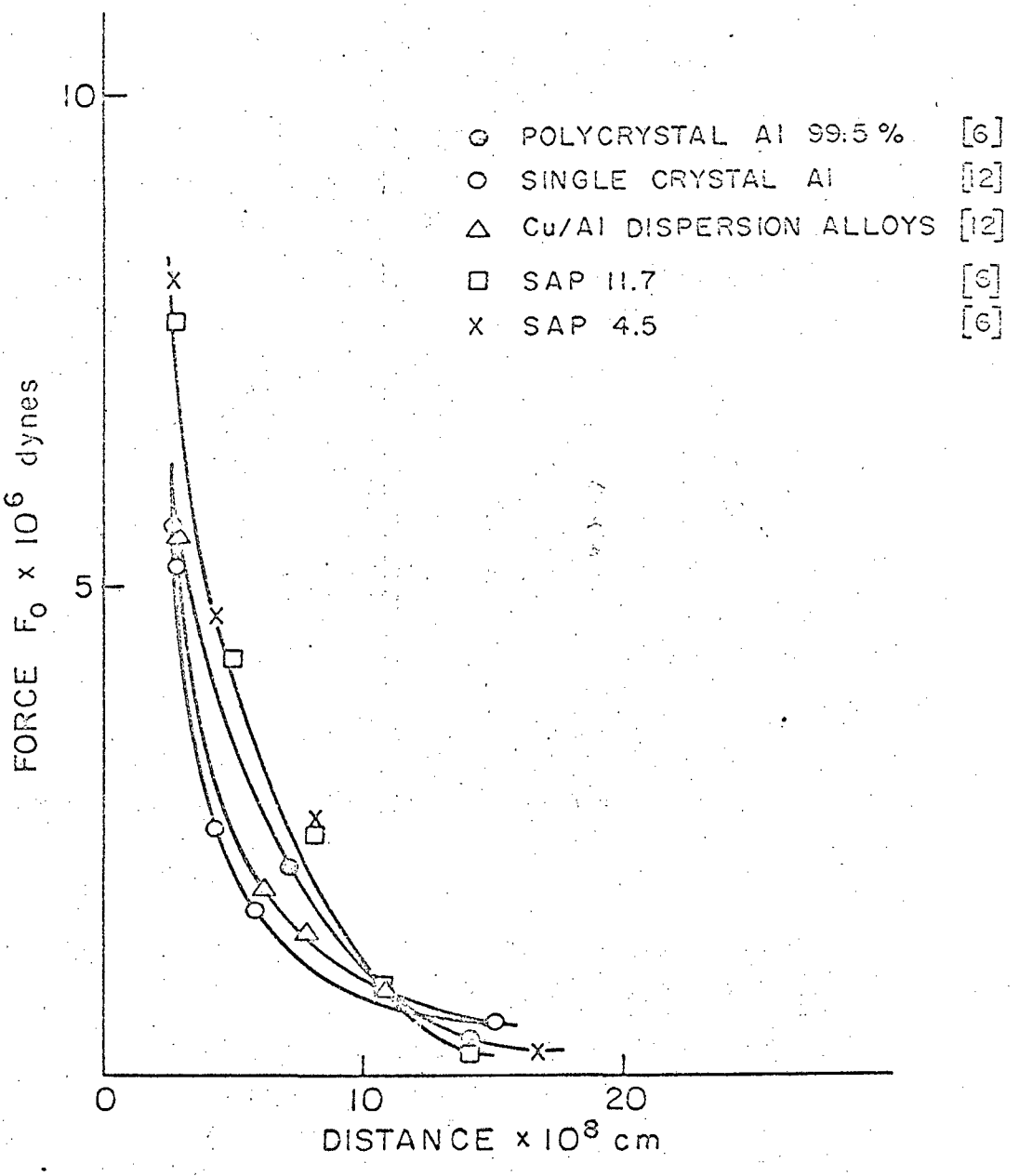
POLYCRYSTAL Al 99.5 % [6]
SINGLE CRYSTAL Al [12]
Cu/Al DISPERSION ALLOYS [12]
SAP 11.7 [6]
SAP 4.5 [6]
FORCE $F_0 \times 10^6$ dynes
DISTANCE $\times 10^8$ cm
10
5
0
10
20

FIG. 3

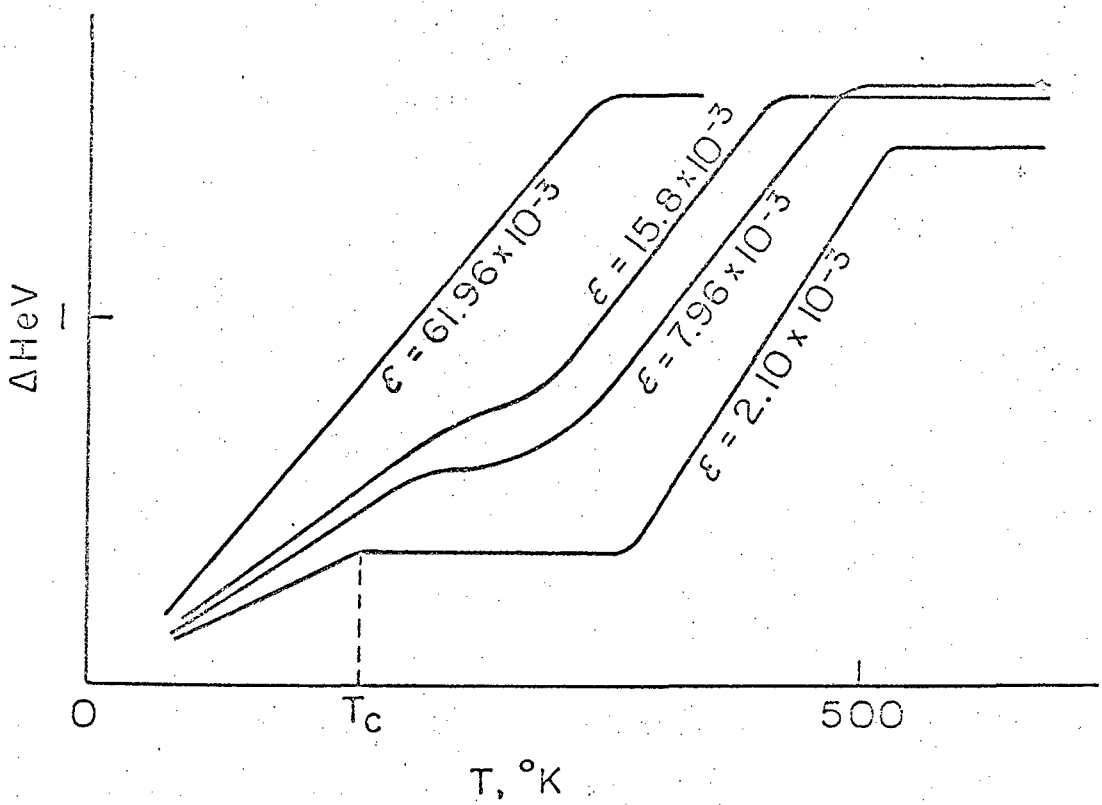

FIG. 4

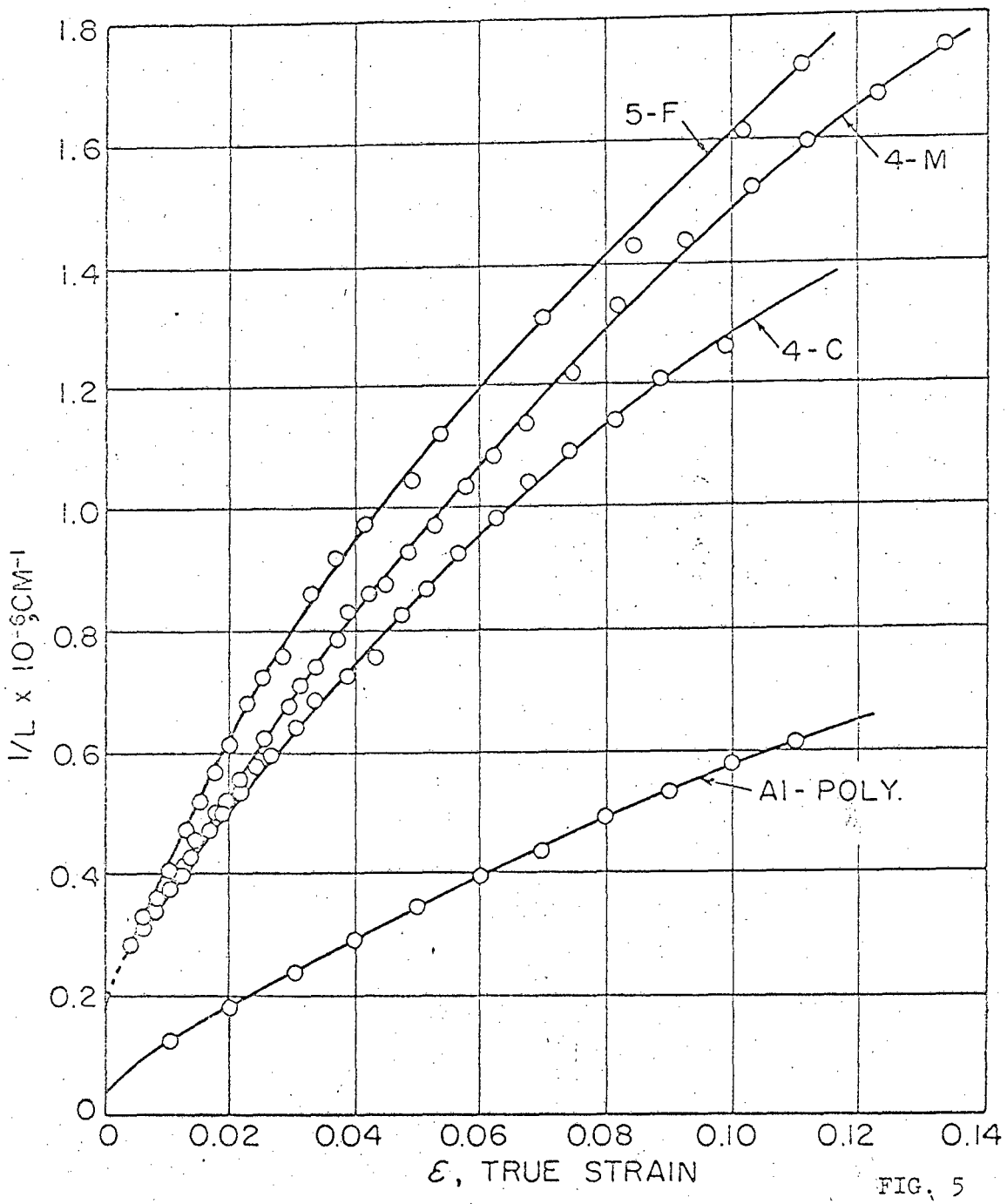
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0
1/L x 10⁻⁶CM⁻¹
0
0.02
0.04
0.06
0.08
0.10
0.12
0.14
ε, TRUE STRAIN
5-F
4-M
4-C
Al-POLY.

FIG. 5

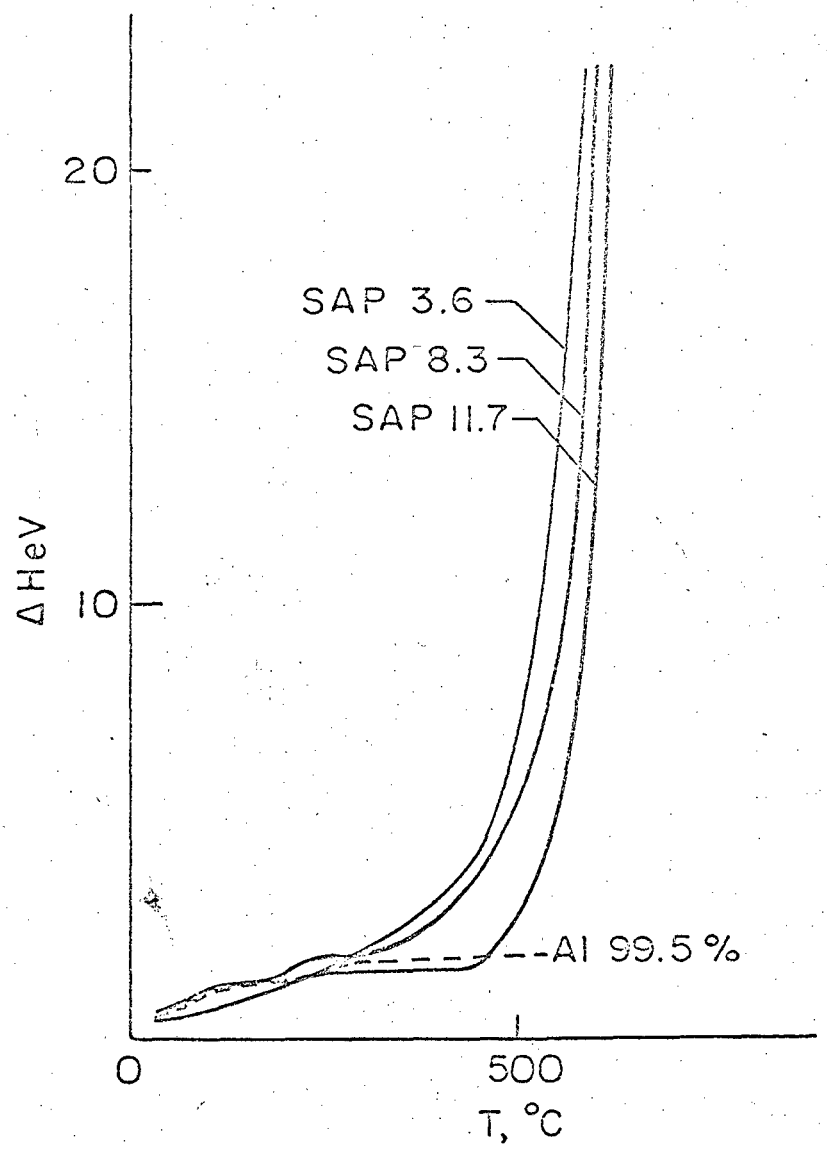

FIG. 6

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