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DISPERSED PARTICLE STRENGTHENING AT LOW TEMPERATURES

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ABSTRACT

The low temperature thermally activated process of deformation for the dispersion alloys of CuAl_2 in Al has been shown to be the intersection mechanism. The variation of the mean separation between the forest dislocations and the change of back stresses with strain has been obtained. The increase in strength and strain hardening for the dispersion alloys has been concluded to arise from the high initial dislocation density and its rapid increase with strain.

DISPERSED PARTICLE STRENGTHENING AT LOW TEMPERATURES

Jack B. Mitchell,¹ Sandip K. Mitra,² and John E. Dorn³

Inorganic Materials Research Division
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Introduction

Early investigations on dispersion strengthened alloys were principally concerned with correlating their mechanical behavior with particle size and degree of dispersion. More recently several theories have been proposed that attempt to relate the plastic behavior of dispersion hardened alloys with interactions between the dispersed particles and dislocations. As indicated in the very recent review of this subject by Guard,¹ major evidence now favors Orowan's² model for initiation of yielding and the Fisher, Hart, Pry³ extension of Orowan's theory for the subsequent strain hardening.

Orowan² demonstrated that the shear stress, τ_y necessary to force a dislocation through the matrix between incoherent particles of precipitate a mean distance λ apart on the slip plane is given by

$$\tau_y - \tau^* = \frac{\Gamma}{b\lambda/2} \quad (1)$$

where τ^* is the local long range back stresses, $\Gamma = \frac{1}{2}Gb^2$ is the dislocation line tension, G is the shear modulus of elasticity, and b is

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the Burger's vector. This concept is in qualitative agreement with the usual observations that the yield strength, τ_y , is greater for the finer dispersions which have smaller values of λ . Furthermore, Ashby and Smith⁴ have shown that the yield strength of internally oxidized Al alloys of Cu agrees closely with predictions based on Orowan's theory.

According to Orowan's theory, each dislocation that moves past a particle leaves a dislocation loop about it. When the local shear stress arising from the stress fields due to a series of concentric loops becomes sufficiently great, the particle or the matrix will fracture. Fisher, Hart and Pry³ extended Orowan's theory by showing that long range back stresses arise from these loops. Such long range back stresses which must be surmounted by the moving dislocations, therefore, contribute to strain hardening. They show that the extra hardening stress τ_p^* arising from the loops about the particles is given by

$$\tau_p^* = \frac{3\pi G b f^{3/2}}{\tau} \quad (2)$$

where π , the average number of loops about a particle, depends on the strain, τ is the mean particle radius and f is the volume fraction of the dispersed phase. The concept presented suggests that following a small amount of strain hardening, the flow stress is no longer given by Eq. 1 but by the sum of τ_p^* , given by Eq. 2, and all other sources of hardening.

Recent electron microscopic observations of deformed dispersion hardened alloys, however, have shown that the dislocation-particle interactions are much more complicated than assumed in the Fisher-Hart-Pry model. Thomas, employing thin film transmission electron microscopy, has observed that in contrast to Orowan's model, dislocations

are frequently generated at the interface between the hard dispersed phase and the matrix. Using replica techniques, Thomas, Nutting and Hirsch⁵ have shown that slip in over-aged Cu alloys of Al does occur in the matrix without deformation of the particles. Whereas the Fisher-Hart-Pry model was based on the operation of slip on a single plane, as might be expected from the von Mises⁶ requirement for polyslip, slip was observed to take place on several slip systems in the dispersion hardened alloys. Transmission electron microscopic observations by Nicholson, Thomas and Nutting⁷ and the discussion by Hirsch⁵ have shown that cross slip takes place as the dislocations approach the dispersed particles, a prominent feature of the substructure being the resultant complex tangles of dislocations in relatively high density that are localized in the vicinity of the particles. Furthermore, Ashby and Smith⁴ have noted that cross slip leads to prismatic dislocation loops that do not lie on the original slip plane.

Although the complexity of details so far observed by electron microscopy in dispersion hardened alloys had not yet been marshalled to provide a detailed model for quantitative rationalization of dispersion strengthening, they nevertheless show that the Fisher-Hart-Pry theory is, at best, based on a model that is only remotely in agreement with the presently known facts. The apparent agreement that was obtained between predictions based on the theory and several isolated experimental facts on the plastic behavior of dispersion hardened alloys must have been largely fortuitous. This conclusion is further emphasized by the now mounting evidence that the strengthening of pure metals and alpha solid solutions is largely associated with the formation of dislocation entanglements. It is the major thesis of this investigation that the strain

hardening that is achieved in a dispersion strengthened alloy is primarily associated with the dense entanglements of dislocations that takes place as a result of the enhanced cross slip, particle induced polyslip, and other cooperative interactions between dislocations that are induced by the dispersed particles rather than as a result of planar dislocation loop formation about the dispersed particles as assumed in the Fisher, Hart and Pry model.

The experimental procedure that will be used in this investigation is based on the method of analysis used by Mitra, Osborne and Dorn⁸ and Mitra and Dorn⁹ for uncovering the magnitude of the local interactions and long range back stresses operative during thermally activated intersection of dislocations. This analysis is based on extensions of Basinski's¹⁰ modifications of the Seeger¹¹ theory for thermally activated intersection. It will be shown that the thermally activated mechanism for deformation of dispersion hardened alloys is intersection just as it is in the case of single crystals, polycrystals and alpha solid solution alloys. Furthermore, it will be shown that the greater hardening of the dispersion strengthened alloys arises from the greater density of dislocations in the entanglements. The effects of local interaction stresses and the long range back stresses will also be evaluated.

Preparation of Specimens

The aluminum-copper system was selected to develop dispersions of CuAl_2 precipitated particles in an Al solid solution matrix for this investigation. This system has received considerable study and it is known that good control of auxiliary variables can be achieved while developing a uniform dispersion of the precipitated particles. Also, the

results of the analysis of the low temperature mechanical tests could be compared with previous work on pure aluminum and aluminum-magnesium solid solution alloys. In addition to the degree of dispersion, the following factors were considered to be important in interpreting the properties of the alloys.

1. Grain size of the alpha phase.
2. Composition of the alpha phase.
3. Uniformity of dispersed particles, particularly the absence or only nominal presence of grain boundary precipitates.
4. A single type of incoherent equilibrium precipitate.

Alloys containing 4 and 5% copper by weight, as shown in Table I, were prepared from high purity aluminum-copper alloys cast into an ingot, homogenized, hot and cold rolled to 12-in. wide sheets, 0.100 in. thick and finally annealed.

The heat treatments required to obtain the desired features noted above had been previously determined.¹² The heat treatments selected for the test specimens were as follows:

A. The as-received alloys were solution heat treated and homogenized for 72 hrs. at 540°C and water quenched.

B. The alloys were then cold worked 30% preliminary to a recrystallization treatment at 540°C to provide a constant final grain size of the alpha phase. Since additional grain growth occurred during subsequent precipitation treatments, the time at the recrystallization and the grain growth temperature was adjusted for each of the various alloys and dispersions so that the final alpha solid solution grain size was about the same.

Table I
Chemical Composition of Alloys*

Percent Weight Alloying Components							
Nom. % by wt. Cu	Chemical Analysis	Spectrographic Analysis					
		Cu	Fe	Si	Mg	Ca	Na
4%	4.080	.002	.002	.0004	<.0006	<.0006	.001
5%	5.240	.001	.001	.000	.000	.000	.000

*

All alloys and analyses were furnished by the courtesy of the Aluminum Company of America Research Laboratories.

C. The recrystallization treatments were followed by appropriate aging treatments. The aging treatments were selected to avoid excessive grain boundary precipitation.

D. Finally, the alloys were given a preliminary aging treatment after which they were furnace cooled to 305°C and aged for two days. Specimens were then machined and an additional two days of aging at 305°C was allowed to remove the effects of machining and to establish the composition of copper in the alpha solid solution near the limit of solubility of copper in aluminum at 305°C. The actual heat treatments which were used are shown in Table II.

The resulting solid solutions were established by previous studies to contain about 0.19 atomic percent copper.¹² Furthermore, subsequent room temperature aging or strain aging during testing were observed to be insignificant. The specimens were etched in a solution of 6 HNO₃, 9 HCl, 2 HF and 15 H₂O parts volume to establish identical surface conditions and to determine the average alpha grain size. The grain size determinations made by the intercept method are recorded in Table II.

The average number of particles observed per unit area of polished surface, N , were established for each group of specimens by metallographic examination of a number of specimens of each of the degrees of dispersion. Calculations of the mean planar particle radius and mean planar interparticle spacing were made assuming the particles to be spherical and uniformly distributed throughout the matrix. Neither of these assumptions, however, is perfectly valid in these alloys. Assuming also that each particle of radius r on the plane of polish has a circle of influence of radius R , then $\frac{r^2}{R^2} = f$ where f is the volume fraction of particles in the matrix. The number of particles per unit area of

Table II
Heat Treatments for Dispersions of CuAl_2

Description of % Cu dispersions	Recrystallization		Preliminary Aging Treatments				Grain size grains/mm. 10^{-4}	$N \times 10^{-4}$	f	r cm^{-5} $\times 10^{-5}$	λ cm^{-4} $\times 10^{-4}$
	Time min.	Temp °C	Time hrs	Temp °C	Time hrs	Temp °C					
4-M	2-1/2	540 W.Q.	1/2	440	A.C. & 3	350	2.3	1631	.0453	2.98	2.8
4-C	2-3/4	540 W.Q.	24	465			2.4	98	.0453	10.8	11.4
5-F	2-1/2	540 W.Q.	.025	525	W.Q. & 1	400	2.3	7620	.0595	1.58	1.3

Symbols: C = coarse, M = medium, F = fine, W.Q. = water quench, A.C. = air cooled, N = number of particles per unit area of plane of polish, r = mean planar particle radius, λ = mean planar distance between particles, f = volume fraction of precipitate

matrix is $N = \frac{1}{\pi R^2}$ and the mean planar particle spacing, λ , was taken approximately equal to $2R$. The volume fraction of precipitate is obtained from the aluminum-copper phase diagram together with values of the density of the alpha solid solution and the θ particles. Taking a density of 4.35 for CuAl_2 , a density of 2.73 for the alpha solid solution and the solid solution to contain 0.194 atomic percent copper, the values of f , r and λ obtained are shown in Table II.

Theory

As shown by Seeger,¹¹ the strain rate, $\dot{\gamma}$, ascribable to thermally activated intersection of dislocations is given by

$$\dot{\gamma} = N A b \nu e^{-U/kT} \quad (3)$$

where

- N = the number of points per unit volume where intersection will occur under an appropriate thermal fluctuation,
- A = the average area swept out per unit intersection,
- b = the Burger's vector,
- ν = approximately the Debye frequency,
- U = the smeared average activation energy for all permissible thermally activated intersection mechanisms,
- k = Boltzmann's constant,
- T = the absolute temperature.

The force-displacement diagram from intersection at the absolute zero first suggested by Basinski,¹⁰ is shown schematically as the $F_0 - x$ curve in Fig. 1. At higher temperatures, the force becomes

$$F = F_0 G/G_0 \quad (4)$$

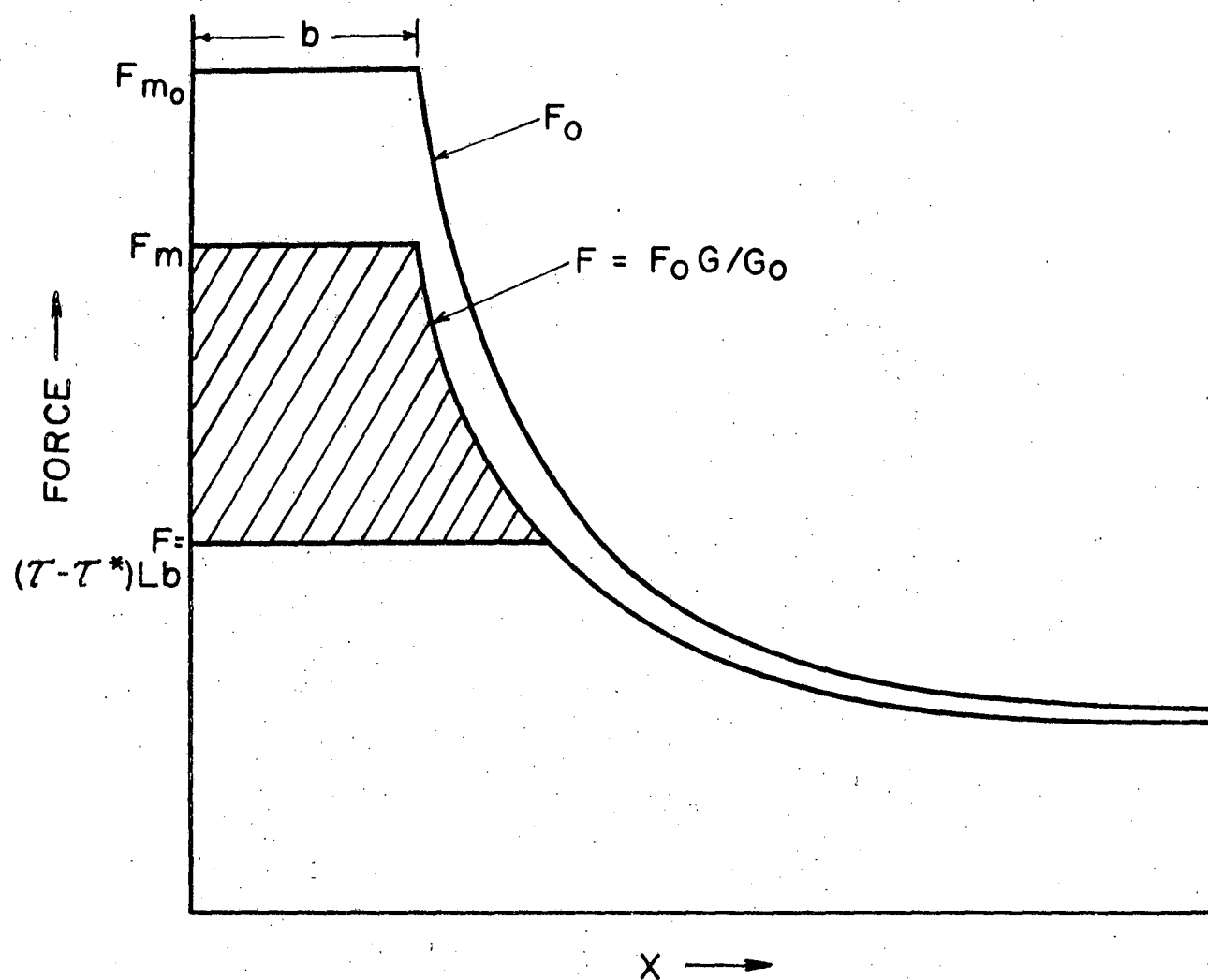


FIGURE I. SCHEMATIC REPRESENTATION OF THE AVERAGE $F-X$ CURVE FOR INTERSECTION.

where G and G_0 are the shear moduli for the test temperature and the absolute zero, since all restraints to intersection vary linearly with the shear modulus of elasticity. When a resolved shear stress τ is applied, the force assisting intersection is given by

$$F = (\tau - \tau^*)Lb \quad (5)$$

where τ^* is the back stress field, and L is the mean spacing between dislocations in the critical region where thermally activated intersection occurs. Consequently, the energy that must be supplied by a thermal fluctuation to complete intersection is given by the cross-hatched area of Fig. 1 which is

$$U = \int_{F=(\tau-\tau^*)Lb}^{F_m} x dF \quad (6)$$

An important factor in determining the $F_0 - x$ curve is the experimentally determinable value of β , which is defined as

$$\beta = \left(\frac{\partial \ln \dot{\gamma}}{\partial \tau} \right)_T = \frac{xLb}{kT} \quad (7)$$

as can be deduced from Eqs. 3 and 6.

Analyses

The objectives of the following analyses are the determination of the $F_0 - x$ curve, and the experimental evaluation of the two parameters L and τ^* that determine the strain hardened state. In order to illustrate how this was done, we will consider one example, namely that of the 5-F alloy which was prestrained at 77°K and $\dot{\epsilon} = 1 \times 10^{-4}$ /sec to five arbitrarily chosen states corresponding to stresses 3.38, 4.48, 5.44, 6.03 and 6.94×10^8 dynes/cm² and strains 0.010, 0.015, 0.021, 0.027

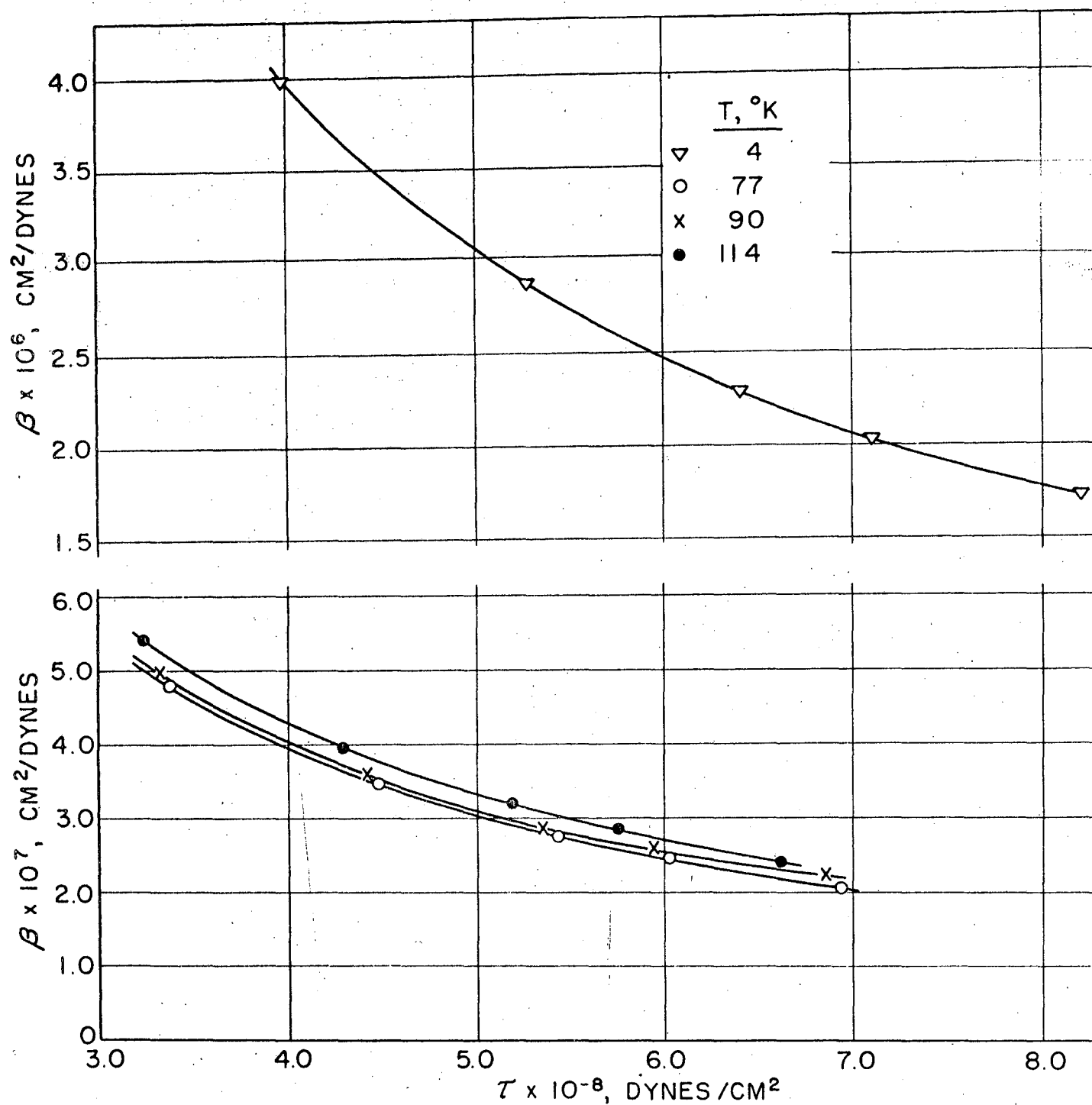


FIGURE 2. VALUES OF β FOR ALLOY 5-F FOR FIVE STRAIN-HARDENED STATES OBTAINED BY PRETRAINING AT 77 $^{\circ}\text{K}$.

and 0.034, respectively. At each of these states, the values of β at 4, 77, 90 and 114°K were obtained using

$$\beta = \left(\frac{\partial \ln \dot{\gamma}}{\partial \tau} \right)_T \approx \left(\frac{\ln \dot{\gamma}_2 / \dot{\gamma}_1}{\Delta \tau} \right) \approx \left(\frac{\ln \dot{\epsilon}_2 / \dot{\epsilon}_1}{\Delta \sigma / 2} \right) \quad (8)$$

Figure 2 shows the β values, appropriate to the above strain hardened states, plotted against the flow stress at different temperatures. The modulus adjusted* flow stress $\tau G_0 / G$ were then plotted as a function of βkT as shown by the solid curves of Fig. 3. Similar data obtained for other dispersion alloys at different states are also shown in the same figure. The broken curves in the above figure represent the variation of $\tau G_0 / G$ against βkT for the three alloys at a fixed temperature of 77°K and strain rate 1×10^{-4} /sec.

As shown by Eq. 3, the activation energy U for constant strain rate tests increases linearly with the absolute temperature. Consequently, this energy is very small at 4°K and, therefore, as shown in Fig. 1, constriction having been completed, the value of x for such low temperatures is associated with the displacement necessary to produce a jog, namely, about one Burger's vector. Applying this approximation, we obtain from the data of Fig. 3 that $Lb^2 = \beta kT$ at 4°K. Consequently, L can be identified for each strain hardened state represented in Fig. 3.

As shown by Eq. 5

$$\tau L b G_0 / G = F G_0 / G + \tau^* L b G_0 / G = F_0 + \tau_0^* L b \quad (9)$$

where τ_0^* gives the back stresses referred to the absolute zero of temperature. And since L is now known, both $\tau L b G_0 / G$ and $x = \frac{\beta kT}{Lb}$ can be deduced from the data of Fig. 3 as shown recorded in Fig. 4. As

* Previously reported moduli for pure Al were used.¹³

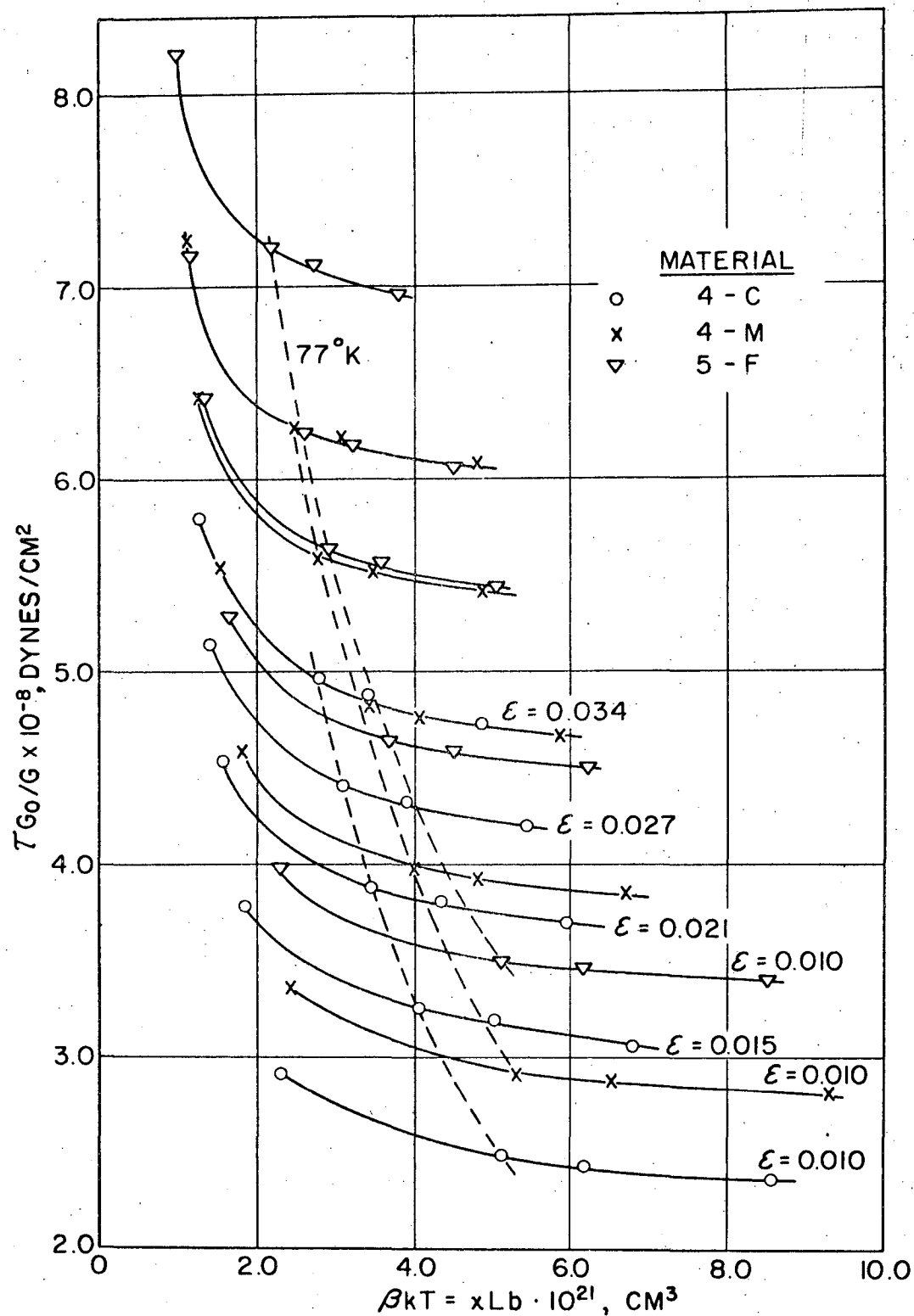


FIGURE 3. PLOT OF STRESS AGAINST ACTIVATION VOLUME AT 4, 77, 90 AND 114°K WITH $\dot{\epsilon} = 1 \times 10^{-4}$ /SEC. FOR DIFFERENT STRAIN-HARDENED STATES.

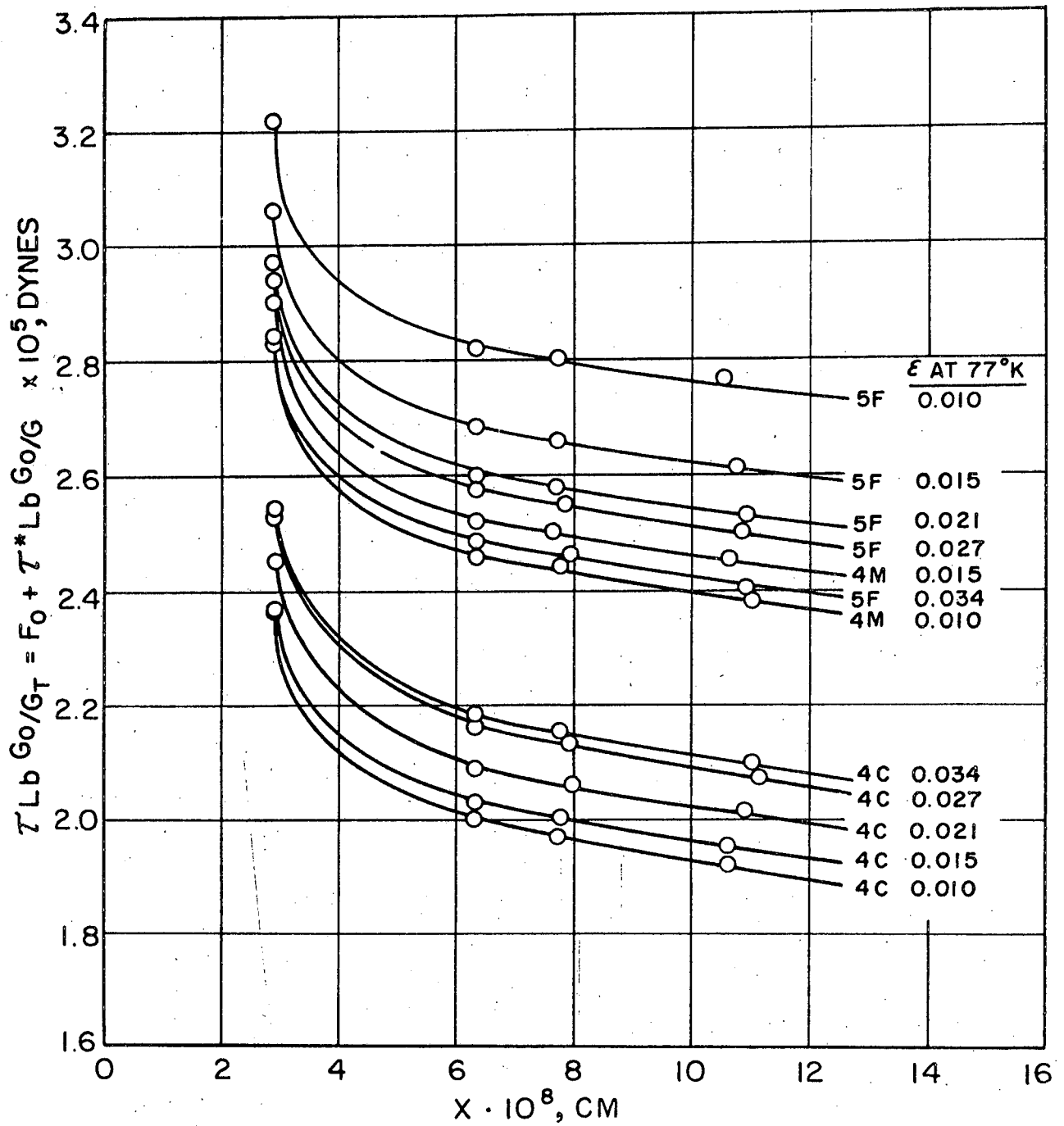


FIGURE 4. PLOT OF $\tau Lb G_0/G_T$ vs X FOR THE 3 DISPERSION ALLOYS.

shown by Eq. 9, each curve represents the $F_0 - x$ curve except for a vertical displacement arising from the back stresses equal to $\tau_0^* L b$. In Fig. 5 a comparison of the previously obtained $F_0 - x$ curve for pure Al with that for the Cu dispersion alloys of Al is shown, the curves being brought into coincidence at $x = 12$. The minor difference between these two curves is suggestive of a slightly higher constriction energy for the Cu-Al alloys which would arise from a slightly lower stacking fault energy. Therefore, the thermally activated mechanism of low temperature deformation in dispersion hardened alloys is attributable to the same intersection mechanism that applies to pure metals and alpha solid solutions.

Whereas a preliminary concept of the variation of the back stresses τ_0^* with strain ϵ at 77°K could be deduced from the vertical displacement of the various curves for the same alloy given in Fig. 4, a more detailed investigation was conducted using the technique developed by Mitra and Dorn.⁸ The total stress required to cause plastic deformation arises from the sum of four terms

$$\tau = \tau_T + \tau_i^* + \tau_\ell^* + \tau_s^* \quad (10)$$

where τ_T is the stress required to aid the thermal fluctuations in promoting intersection, τ_i^* arises from the local interactions between the intersecting dislocations, τ_ℓ^* is due to long range back stress fields and τ_s^* is the stress necessary to overcome short range order hardening of the matrix and Suzuki locking. The previously defined τ^* is equal to $\tau_i^* + \tau_\ell^* + \tau_s^*$, each of which represent stress fields that dislocations cannot readily surmount by thermal fluctuations at low temperatures. Because of the low alloy content of the matrix, τ_s^* must be quite small in the present case and, therefore, this factor will henceforth be included in τ_ℓ^* . Both τ_T and τ_i^*

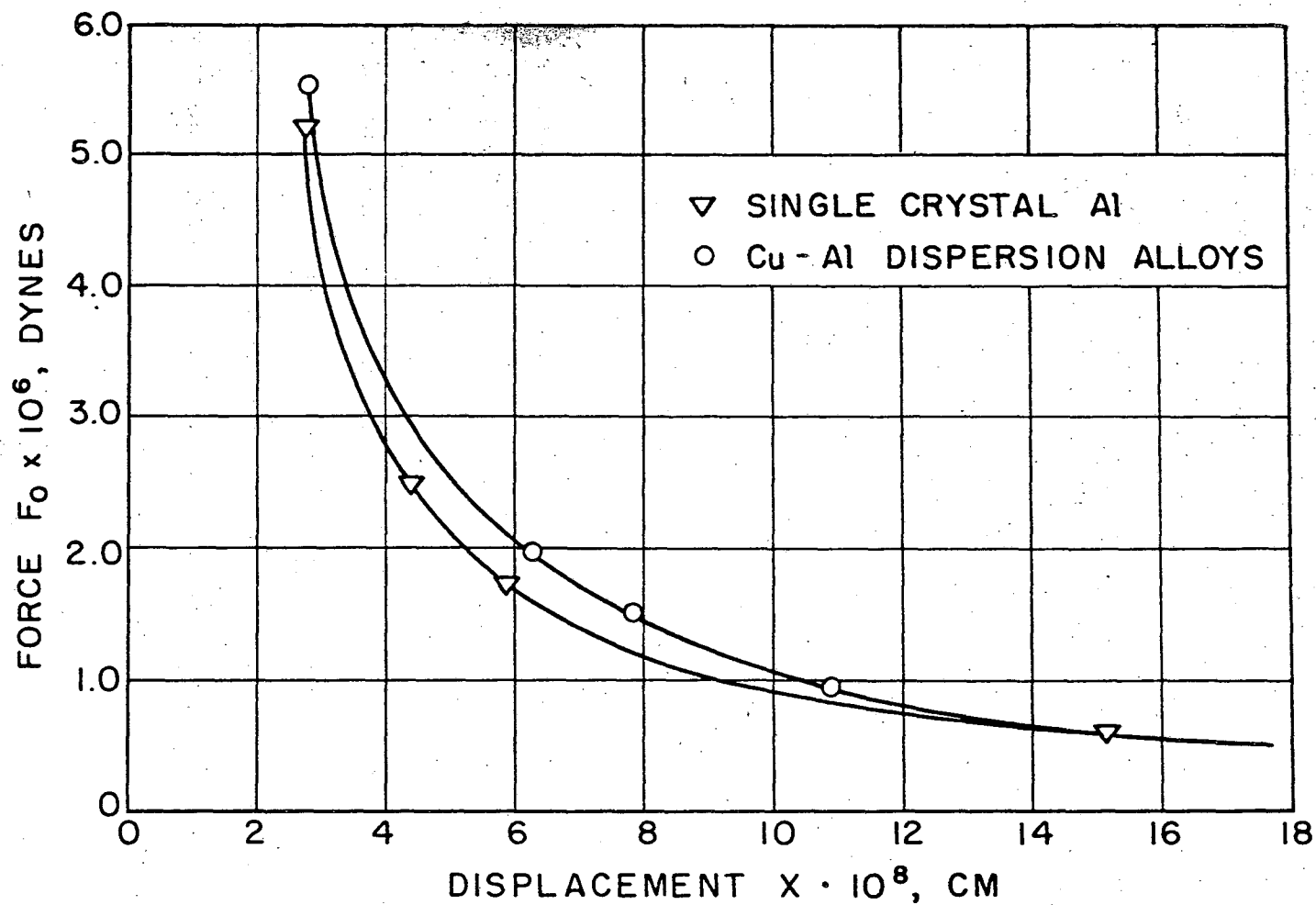


FIGURE 5. FORCE DISPLACEMENT CURVES FOR SINGLE CRYSTAL Al AND Cu - Al DISPERSION ALLOYS.

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are easily estimated and, therefore, τ_l^* can be deduced from the known value of the flow stress, τ .

Previous investigations have shown that $NAb\gamma$ is substantially constant regardless of the strain. Consequently, as shown by Eq. 3 and Fig. 1, when tests are conducted at a constant temperature and a fixed strain rate, U is a constant and, therefore, F and x are also constant throughout the test. The value of F and x appropriate to a test conducted at 77°K and a strain rate of 1×10^{-4} /sec is directly obtainable from Fig. 5. Therefore,

$$\tau_T = F/Lb \quad (11)$$

is known when L is established. Furthermore, the local interaction stress has been reported¹⁴ for pure Al to be

$$\tau_l^* = 0.035 \frac{Gb}{L} \quad (12)$$

which is also known when L has been determined.

Tests for the determination of β as a function of τ were conducted on each dispersion hardened alloy at 77°K and a strain rate of $\dot{\epsilon} = 1 \times 10^{-4}$ /sec. These data are recorded in the three uppermost curves of Fig. 6. Since $x = 6.2$ for these conditions of test as deduced from Fig. 5, the value of L is given by $\beta kT/xb$. The decrease of βkT with increasing flow stress during deformation is directly proportional to the decrease in L . The two lower curves for τ_T and τ_l^* were deduced from Eqs. 11 and 12. Therefore, τ_l^* could be determined as shown in Fig. 6.

Since

$$\tau = \frac{F}{Lb} + 0.035 \frac{Gb}{L} + \tau_l^* \quad (13)$$

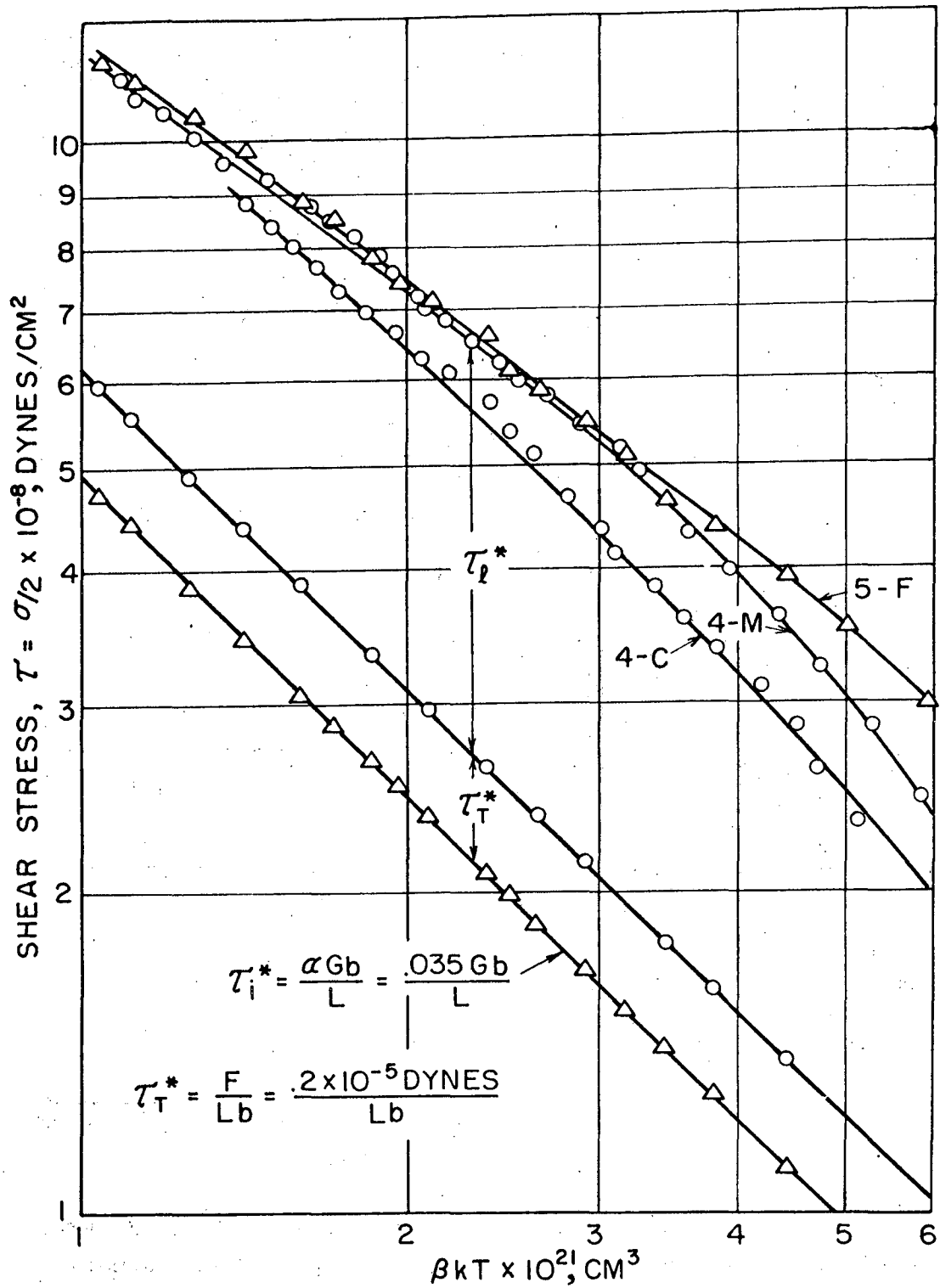


FIGURE 6. PLOT OF LOG τ vs LOG βkT FOR THE 3 DISPERSION ALLOYS STRAINED AT 77°K. $\dot{\epsilon} = 10^{-4}$ SEC.

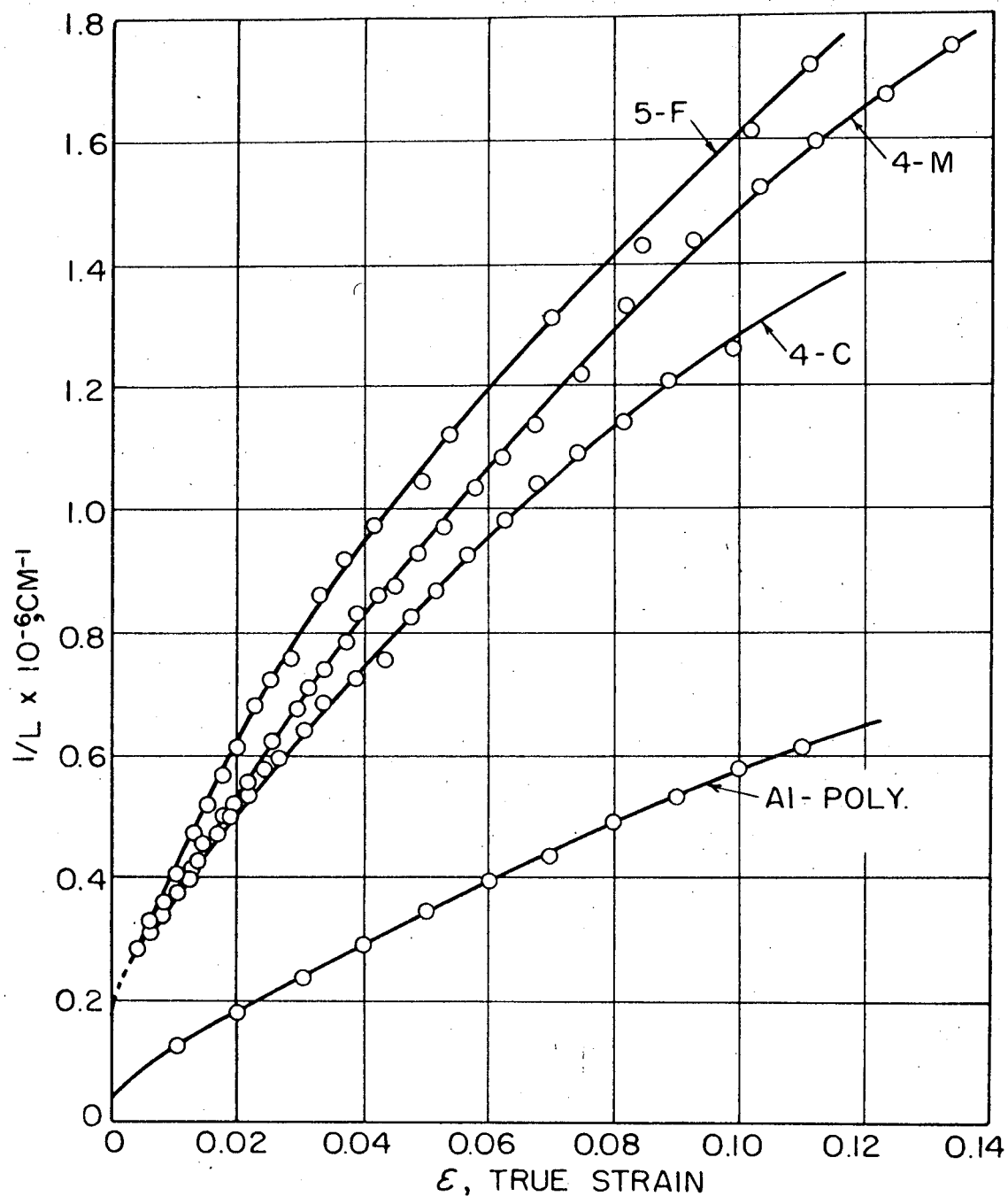


FIGURE 7. VARIATION OF $1/L$ WITH STRAIN FOR THE 3 DISPERSION ALLOYS AND FOR PURE Al POLYCRYSTALLINE SPECIMENS STRAINED AT 77°K . $\dot{\epsilon} = 10^{-4}/\text{SEC}$.

the log-log plot of τ versus $\beta kT = xLb$ would have been linear at 45° if τ_2^* were also proportional to $1/L$. Therefore the deviation of the three uppermost curves of Fig. 6 from a 45° line attests to the fact that does not vary exactly linearly with $1/L$. As shown more forcefully in previous investigations, the Cottrell-Stokes ratio is rather insensitive to this deviation and, therefore, is not a wholly satisfactory criterion for suggesting that the long range and local interaction stresses might arise from the same mechanism.

The effect of straining at 77°K under a strain rate of $\dot{\epsilon} = 1 \times 10^{-4}/\text{sec}$ on $1/L$ is shown in Fig. 7 and the effect of strain on the flow stress, the long range back stress and the local interaction stress is shown in Figs. 8A to 8C.

Discussion

The close identity of the $F_0 - x$ curve for the dispersion strengthened Cu alloys of Al with that for single crystals of Al, polycrystalline Al and Mg alpha solid solutions of Al reveals that in all these cases deformation takes place by substantially the same thermally activated mechanism. The uniquely different $F_0 - x$ curve obtained previously⁹ for Cu single crystals is consistent only with the intersection model since it is in agreement with the lower stacking fault energy of Cu and its consequent higher constriction energy. In addition, the minor differences between the $F_0 - x$ curves of the dispersion alloys and the Mg alpha solid solutions of Al can be attributed to the effect of a slight decrease in the stacking fault energy of Al upon alloying with either Mg or Cu. We, therefore, conclude that the thermally activated rate controlling mechanism for the plastic deformation of the dispersion strengthened Cu alloys of Al is determined by the intersection mechanism.

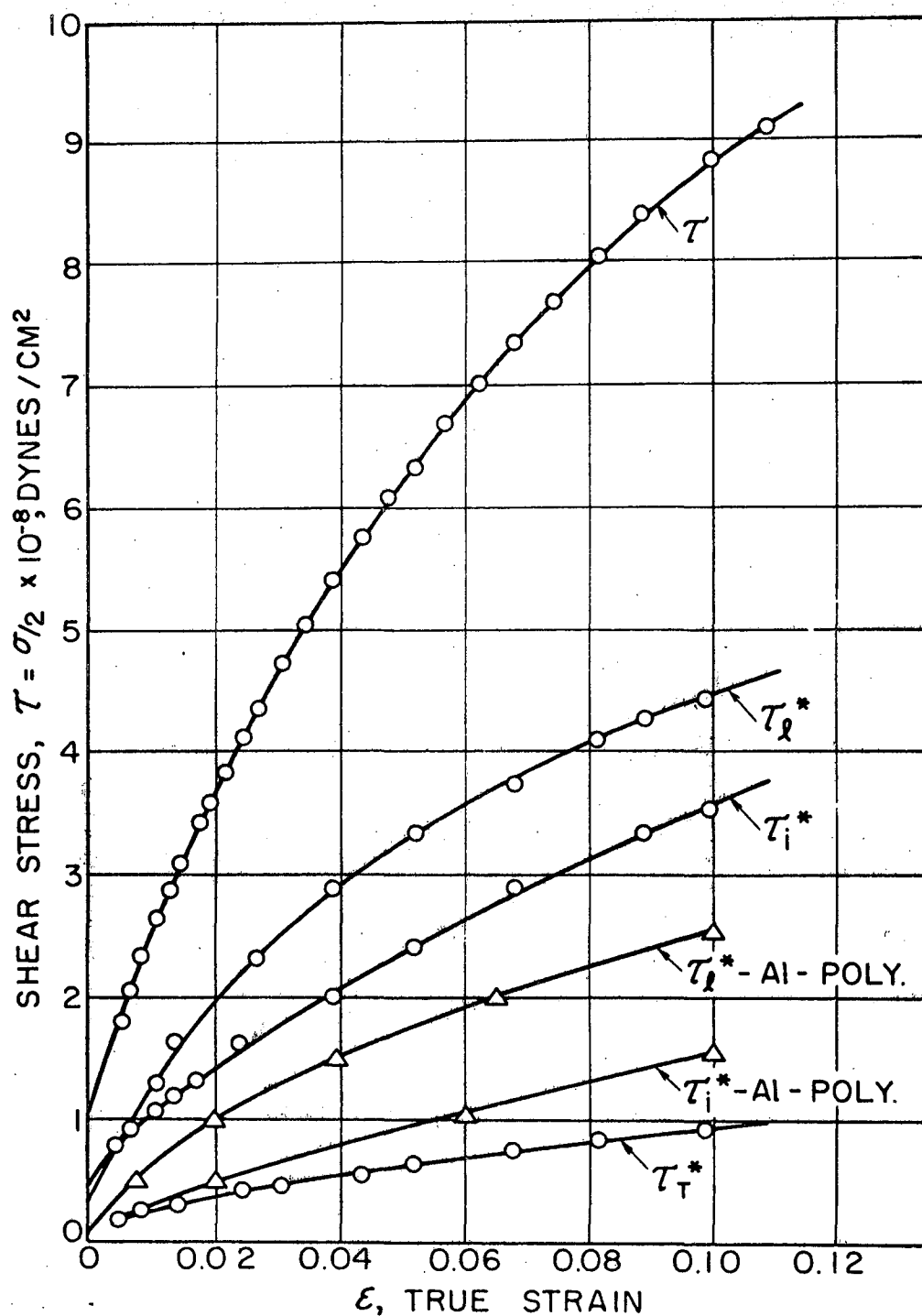


FIGURE 8A. VARIATION OF CONTRIBUTIONS TO THE FLOW STRESS WITH STRAIN FOR THE DISPERSION ALLOY 4-C AND PURE Al POLY-CRYSTALLINE STRAINED AT 77°K, $\dot{\epsilon} = 10^{-4}/\text{SEC}$.

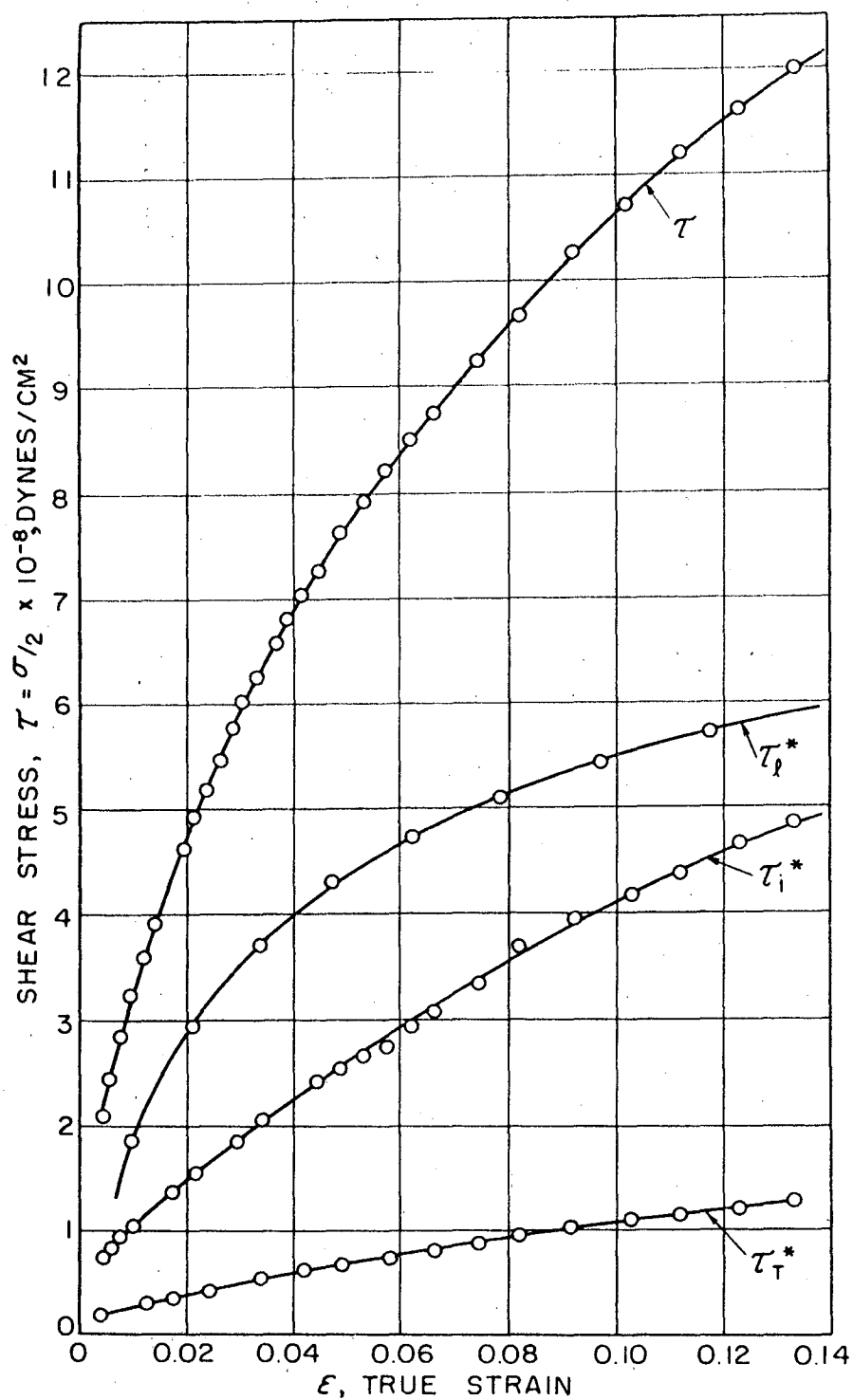


FIGURE 8B. VARIATION OF CONTRIBUTIONS TO THE FLOW STRESS WITH STRAIN FOR THE DISPERSION ALLOY 4-M STRAINED AT 77°K.
 $\dot{\epsilon} = 10^{-4}$ /SEC.

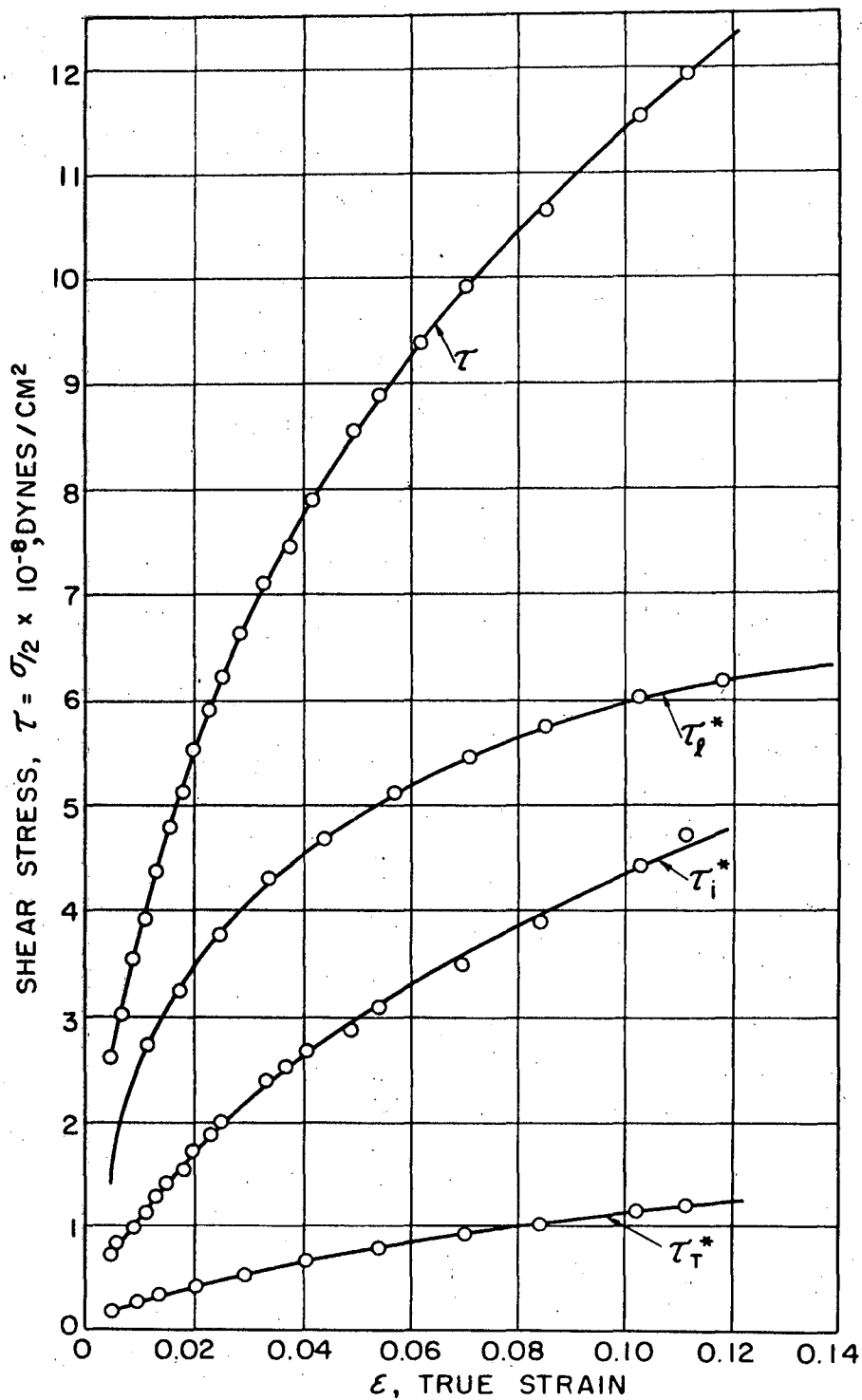


FIGURE 8C. VARIATION OF CONTRIBUTIONS TO THE FLOW STRESS WITH STRAIN FOR THE DISPERSION ALLOY 5-F STRAINED AT 77 °K. $\dot{\epsilon} = 10^{-4}/\text{SEC.}$

A distinctive parameter of the intersection mechanisms is the distance L between the forest dislocations. Since single and polycrystalline Al form entanglements, the distance L refers to some mean value in the entanglements. As shown in Fig. 7, the significant values of $1/L$ for the dispersion strengthened alloys is somewhat greater than that in high purity polycrystalline Al. This, plus the much more rapid increase in $1/L$ with strain for the dispersion strengthened alloys is a major factor responsible for the strain hardening of dispersion strengthened alloys.

Transmission electron microscopic observations of the deformed structure of these dispersion strengthened alloys revealed the presence of complex tangles of dislocations of relatively high density which tended to be localized at individual particles and at groups of particles. The simple patterns of dislocation loops about the particles postulated in the Fisher-Hart-Pry theory for dispersion hardening were not present. These observations, which are in agreement with the results of several previous electron-microscopic investigations on dispersion strengthened Cu alloys of Al, reveal that the true role of dispersion strengthening is not contained in the Fisher-Hart-Pry model. Rather, the dispersed phase, perhaps as a result of induced cross slip and other cooperative phenomena in the vicinity of the particles, causes the formation of dense entanglements. The mechanically determined values of $1/L$ were in good qualitative agreement with the average spacings that were observed in the dislocation tangles by means of thin film electron microscopy.

The finer dispersions not only induced more rapid increases in $1/L$ with strain but also, as shown in Figs. 8A to 8C, caused much more rapid increases in the local interaction stresses τ_i^* and the long range back stresses τ_l^* . As shown most pronouncedly in Fig. 8C, however,

τ_l^* begins to level off at the higher strains. Such leveling off occurred before extensive fracturing took place in the dispersed CuAl_2 phase, and might, therefore, be attributed to relief of the long range back stresses due to cross slip.

Conclusions

1. The rate controlling mechanism for deformation of dispersion strengthened alloys of CuAl_2 in Al is the thermally activated intersection of dislocations.
2. The initial density of dislocations is higher in the alloy than in high purity Al.
3. Strain hardening results primarily from the increase in the density of dislocations in the entanglements that are produced in the vicinity of the dispersed particles and from the increase in the long range back stresses.

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