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PUBLICAÇÃO IEA 417
CCTM 30

MAIO/1976

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CCTM

INSTITUTO DE ENERGIA ATÔMICA
SÃO PAULO – BRASIL

APROVADO PARA PUBLICAÇÃO EM JANEIRO/1976

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ABSTRACT

An analysis of the deformation behavior of high purity LiF single crystals is given using yielding and work hardening data and thermally activated deformation parameters obtained in the temperature range 77 – 423°K It is found that while the Fleischer mechanism is apparently valid experimentally over the thermally activated temperature range vacancies produced in large numbers at 77°K could also play a role in determining the critical resolved shear stress at that temperature The temperature dependence of the work hardening rate is seen to depend on the increase in dislocation debris production at lower temperatures Work hardening is controlled by long range dislocation dislocation dipole interactions over the entire temperature range studied

INTRODUCTION

The mechanical behavior of ionic crystals having the rock salt structure has been studied extensively as a means of acquiring a better understanding of dislocation behavior in solids In LiF the early work of Gilman and Johnston(1) included the investigation of many aspects of dislocation behavior and the effect of impurities on yielding and flow In particular Gilman(2) showed that the critical resolved shear stress in LiF single crystals is determined by the stress required to move fresh dislocations while Johnston(3) demonstrated that the resistence to motion for these fresh dislocations is provided by impurities The influence of impurities on yield stress was attributed to edge dislocation interactions with tetragonal lattice distortions by Fleischer(4) and more recently the theory of such hardening has been further refined(5 6) Experimental data demonstrating Fleischer hardening in LiF has been presented by Reppich(7) and by Guiu and Langdon(8)

Most of the previous deformation studies in LiF single crystals have concentrated on yielding effects and many have utilized impure crystals Impure crystals have a high yield stress and show only one stage of work hardening High purity LiF on the other hand shows consistent low yield values(9) and three stage work hardening behavior above room temperature(10) Below room temperature the ductility and work hardening behavior have reported anomalies(11) which are not well understood

The purpose of this paper is to present an overall analysis of yielding and work hardening in high purity LiF single crystals over a wide range in temperatures Yielding and work hardening data are reported in terms of thermally activated stresses and thermally activated deformation parameters are analyzed as a function of temperature stress and strain in order to identify the various rate controlling mechanisms contributing to work hardening in the temperature range 77 – 423°K

EXPERIMENTAL PROCEDURE AND ANALYSIS

High purity LiF single crystals of size $10 \times 4 \times 4\ mm^3$ purchased from the Harshaw Chemical Company in 1971 were annealed at 700°C in air for 48 hours and furnace cooled These crystals contain less than 3 ppm of divalent cation impurities, chiefly Mg^{2+} The samples were deformed in compression along a (001) direction in an Instron universal testing machine using a specially designed low temperature jig[(12)] The stress strain curves of several samples were determined at each of the temperatures 77 186 or 198 296 373 473 and 633°K at a strain rate of 0 002/min , the results typically being reproducible to within ±10% The critical resolved shear stress (CRSS) was taken as the 0 1% offset proof stress The CRSS may by seperated into its components τ^* the short range or effective stress that can be thermally overcome and τ_μ, the long range or athermal stress this was done by obtaining τ_μ from the temperature-independent portion of the CRSS vs temperature curve

Thermal activation parameters were obtained from strain rate cycling tests in the temperature range 77 – 473°K Strain rate cycling utilized strain rate change rations (ϵ_2/ϵ_1) of 20 and 40 for a base strain rate of 5 0 ± 0 2 x 10^{-4}/min τ_μ was calculated as a function of strain as the difference between the applied shear stress τ_a at that strain and the value for the effective stress τ^*, determined from strain rate cycling[(13)]

Alternately double strain rate change tests were conducted at room temperature to evaluate τ_μ as a function of strain utilizing one specimen each at each 1% strain increment In this test three strain rates are used in such a way that $\epsilon_1/\epsilon_2 = \epsilon_2/\epsilon_3$ enabling the use of the relation[(13)]

$$\tau_\mu = \frac{\tau_2 - \tau_1 \tau_3}{2\tau_2{}^2 - (\tau_1 + \tau_3)} \tag{1}$$

A plot of τ_a τ_μ and τ^* as a function of strain then indicates the relative contributions of the long range and short range stresses to deformation processes and work hardening

The activation volume V^* can be calculated from the relation

$$V^* = kT\,\left(\frac{\Delta \ell n \epsilon}{\Delta \tau}\right)_T \tag{2}$$

where K is the Boltzmann constant T is the absolute temperature and $\Delta\tau$ is the change in flow stress when changing from high to low strain rate A plot of activation volume V^* versus strain at different temperatures or a plot of V^* versus τ^* at a particular strain can then be obtained In this paper V^* values are taken at 2% strain so as to evaluate the work hardening mechanisms during Stage I deformation in LiF single crystals

Activation enthalpies ΔH were determined using the relation[(14)]

$$\Delta H = -TV^*\,\left(\frac{d\tau_c}{dT}\right)_\epsilon \tag{3}$$

where τ_c is the CRSS A plot of ΔH versus τ^* was obtained using values of $(d\tau_c/dT)$ obtained as slopes from the CRSS temperature plot at various temperatures The total enthalpy of activation for dislocation motion ΔH_o was determined using the relation[(14)]

$$\Delta H_o = \Delta H + \int_o^{\tau^*} V^* d\tau^* \tag{4}$$

Values for the integral in eq (4) were obtained by graphical integration of the V^* versus τ^* plots Such ΔH_o determinations are helpful in establishing whether low temperature work hardening in LiF single crystals is a singly or multiply activated process over the effective stress range studied

RESULTS

A. TEMPERATURE DEPENDENCE OF YIELD STRESS

Figure 1 shows the effect of temperature on the critical resolved shear stress of these LiF single crystals. The temperature independent portion of the CRSS temperature curve gives τ_μ = 92 gm/mm^2. The critical temperature T_o below which the CRSS becomes temperature dependent is 375°K to an accuracy of about ±25°K. A computed polynomial least squares fit to the low temperature data yields an extrapolated value for the CRSS at 0°K of 642 gm/mm^2. Using τ_μ above, the short range effective stress at 0°K is calculated to be τ_o^* = 550 gm/mm^2.

The Fleischer relationship[4] written to involve the efective stress τ^* and T_o, the critical temperature where τ^* = 0, is given by

$$\frac{\tau^*}{\tau_o^*} = [1 - (\frac{T}{T_o})^{1/2}]^2$$

This indicates that a plot of $\sqrt{\tau^*}$ vs. $\sqrt{T}$ should be linear with τ_o^* and T_o being determined from the intercepts with the coordinate axis. Figure 2 shows such a plot for the data in Figure 1. This yields a linear relationship and gives values of τ_o^* = 1050 gm/mm^2 and T_o = 425°K.

B. STRESS STRAIN CHARACTERISTICS

Figure 3 shows characteristic curves of true resolved shear stress vs. true strain for pure LiF samples at 77°K, 186° and 296°K. At these temperatures the samples show only one stage of work hardening before fracture. At 77°K LiF seems relatively ductile and invariably fractures at about 0.13 true strain, while at 186°K samples fracture at about 0.10 true strain. At 296°K fracture usually occurs at about 0.08 true strain; at this temperature samples generally show one stage of work hardening up to 0.06 true strain, after which a transition to a higher work hardening rate is observed. At higher temperatures the ductility again increases and multi stage work hardening behavior is observed, depending on purity, temperature, strain rate, and specimen geometry, as has been reported earlier[10].

Typical thermal and athermal contributions to the flow stress are shown as a function of strain for two temperatures in Figure 4. Here the applied shear stress τ_a is resolved into its components τ^* and τ_μ utilizing strain rate cycling at 77°K in Figure 4(a) and using double strain rate change tests at 296°K in Figure 4(b). It is seen that the thermal component of the applied stress remains constant with strain. This is typical of other temperatures as well, and indicates that work hardening is predominantly athermal in nature in this system.

The work hardening rate observed at 0.02 true strain is shown as a function of temperature in Figure 5. Utilizing our preciously published work[10] data covering the range 77 to 473°K is shown. The work hardening rate θ calculated as $\frac{d\tau}{d\gamma}$ is normalized in Figure 5 in terms of the shear modulus μ. Here it is seen that the work hardening rate decreases as the temperature is raised above 77°K, then increases again above 296°K, as reported earlier. Above 473°K dynamic strain aging has been observed, further increasing work hardening rates, as reported elsewhere[15].

C. THERMALLY ACTIVATED DEFORMATION PARAMETERS

Activation volume measurements were undertaken over a wide temperature range to aid in the establishment of deformation mechanisms in these pure LiF single crystals. Figure 6 shows a composite plot of activation volume vs. strain for temperatures from 77°K to 423°K. It is seen that at a given strain the activation volume values increase with increasing temperature With increasing strain the activation volume decreases uniformly and rapidly above 296°K Below 230°K the decrease is less uniform and smaller in magnitude at 77°K the activation volume varies with strain in a complicated manner (not obvious on the scale of Figure 6)

The variation of activation volume with the thermal component of the flow stress τ^* taken at 0 02 strain is shown in Figure 7 for a strain rate ratio of ϵ_1/ϵ_2 = 40 (base strain rate 4 8 x 10^{-4}/min) It is seen that the activation volume decreases with increasing τ^*

The total activation enthalpy ΔH_o can be determined from a graphical integration of Figure 7, utilizing eq (4) and ΔH values calculated from eq (3) Figure 8 shows the variation of the resultant total activation enthalpy with effective flow stress τ^* Three regions can be distinguished with (a) ΔH_o increasing with increasing τ^* in the range of τ^* between 0 and about 180 gm/mm^2 then (b) ΔH_o remaining nearly constant between the τ^* values of 180 gm/mm^2 and 300 gm/mm^2 corresponding to ΔH_o = 1 5 eV ΔH_o apparently decreases above τ^* = 300 gm/mm^2 (c)

DISCUSSION

A CRITICAL RESOLVED SHEAR STRESSES

In thermal equilibrium at temperatures below 500°K LiF crystals contain impurity vacancy dipoles even if the content of divalent cations is less than 5 ppm[16] The resistance offered by impurity vacancy dipoles to the motion of dislocations leads to a strong temperature dependence of the CRSS due to the short range nature of the dipole stress field This is the justification for the application of Fleischer s model in this system which is shown to be experimentally justified in Figure 2 The value for the critical temperature from Figure 2 T_o = 425°K agrees rather well with the value of T_o = 375 ±25°K obtained from the CRSS-temperature data in Figure 1 On the other hand the τ_o^* value obtained from Figure 1 is much lower than that obtained using the Fleischer analysis Similar discrepancies between the experimental value and that obtained using the Fleischer analysis have been observed in other systems[17] but the significance of such discrepancies is not known That Fleischer s model accounts relatively well for the observed temperature dependence of the CRSS in high purity LiF single crystals is in agreement with the work of Reppich[7] who has shown that in samples containing one to 50 ppm of divalent cation impurities the temperature dependence of the CRSS for the region below 473°K can be explained by Fleischer hardening similar results are shown by Guiu and Langdon[8] for crystals containing 7 ppm Mg

According to Pratt Harrison and Newey[18] the assumption in Fleischer s theory that the impurity vacancy dipole is a rigid defect is not valid at high temperatures since the relaxation time for reorientation of dipoles becomes relatively small in a number of ionic systems Pratt Chang and Newey[19] have considered the interaction between dislocations and dipoles that can reorient themselves in the strain field of the dislocations so as to lower the elastic energy of the system The long range of this interaction leads to a retarding force essentially independent of temperature This theory successfully explains the temperature independent flow stress of Mg^{2+}-doped LiF between 300 and 500°K[19] It is possible that a similar mechanism is operative in high purity LiF crystals in the athermal portion of the CRSS-temperature curve between 375°K and 630°K

B WORK HARDENING AT LOW TEMPERATURES

The work hardening characteristics of LiF single crystals depende on temperature impurity content and specimen geometry[9 10] High purity crystals above 296°K exhibit multi stage work hardening behvior[10] but for lower temperatures only one stage of work hardening is observed as shown in Figure 3 Deformation at low temperatures also shows an anomalous ductility such that in our results below room temperature the ductility increases from 0 08 true strain at 296°K to 0 13 true strain at 77°K This is in qualitative agreement with the observations of Okada and Ozawa[11]

The basic deformation mechanism in Stage I in LiF involves the initial operation of two orthogonal slip systems one of which becomes the primary glide plane while the other becomes inactive Dislocations on the inactive slip system act as barriers to slip on the main slip system leading to the formation of deformation bands due to multiple cross glide and Stage I work hardening At low temperatures slip bands in LiF develop from surface dislocation sources and have microscopically straight edges with a very sharp boundary between the slipped and unslipped regions[1 20] Within the slip band the residual dislocation density is very high, whereas outside the slip band the dislocation density is only that of the grown in configuration In NaCl[21] there is progressive penetration of slip lines into the crystal volume as stresses increase at temperatures as low as 4°K

The kinetics of deformation in LiF are controlled by the slower moving screw dislocations[1] As the temperature decreases the dislocation velocity stress exponent m^* increases[22] so that the velocity difference between edge and screw dislocations should increase Edge dislocations will move out of the crystal much faster than screw dislocations leaving a higher density of screw dislocations behind These will multiply by the double cross glide mechanism producing a high concentration of dislocations debris such as vacancies and dislocation dipoles

The mechanism for the observed high rate of work hardening observed at low temperatures involves these dislocation debris which act as obstacles to the passage of dislocations Increased debris concentrations as the temperature is lowered give increased rates of work hardening as the temperature decreases Since work hardening in LiF is predominantly due to long range athermal stresses (Figure 5) work hardening must be due to the production of high jogs on dislocations and to dislocation dipoles having a long range stress field[23] The vacancies produced at low temperature can also be important here since they can form monojogs on dislocations leading to jog draggings[23] Similar interaction mechanisms have been used to account for the low temperature work hardening behavior of MgO[17]

The slight increase in the work hardening rate observed above 296°K in Stage I is probably caused by the temperature dependence of multiple cross glide which gives to a greater concentration of dislocation dipoles at the higher temperatures and thus a higher work hardening rate[10] A similar temperature dependence has been observed in NaCl[24]

C RATE CONTROLLING DEFORMATION MECHANISMS

The overall rate controlling mechanisms for deformation may be confirmed using the measurements of activation volume V^* and of total activation enthalpy ΔH_o At 77°K where $\tau^* = 560$ gm/mm^2 the activation volume is of the order of 500 b^3 as shown in Figures 7 and 8 This indicates that a dislocation point defect interaction is operative and rules out any Peierls Nabarro type of activation mechanism (the latter would require V^* in the range 5 to 50 b^3)^{25} Further V^* shows a slight strain dependence at this temperature Since impurity effects should not be strain dependent this indicates that vacancies produced during deformation play a role in the thermally activated deformation process and that dislocation vacancy interactions are an important rate-controlling mechanism at 77°K

This observation is somewhat surprising since the discussions above indicate that dislocation interactions with impurity vacancy dipoles (the Fleischer mechanism) control yielding and that long range interactions control work hardening in this temperature range However it is expected that a very high concentration of vacancies will be produced at 77°K as a result of the high probability of stress-assisted cross slip which increases continuously with decreasing temperature[26] However since the activation volume is only slightly strain dependent it is probable that dislocation interactions both with vacancies and with imputity vacancy dipoles contribute to the deformation behavior at 77°K This conclusion is aided by the ΔH_o data given in Figure 8 Here region (c) includes 77°K and the apparent τ^* dependence of ΔH_o in this region indicates the operation of more than one rate controlling deformation mechanism

At temperatures above 77°K dislocation vacancy interactions are not important in governing the deformation behavior as discussed below Hence the deformation mechanism at 77°K differs from that at higher temperatures due to the importance of these deformation produced vacancies. This could be a factor in explaining the discrepancy in τ_o^* values between the Fleischer model and experiment since the one lowest temperature point would not follow the theoretical model due to a change in mechanism.

At temperatures of 198°K and above the activation volume V^* is greater than 1000 b^3 indicating that dislocation impurity and/or dislocation-dislocation debris interactions are operating. The strain dependence of V^* shown in Figure 7 confirms that dislocation dislocation debris interactions control work hardening at these temperatures. Of course there is still a large contribution to the flow stress due to impurities in the form of impurity vacancy dipoles. Region (b) in Figure 8 corresponds to the temperature region between 198°K and 250°K $(180 < \tau^* < 300 \text{ gm/mm}^2)$ where the constant value of ΔH_o indicates that a singly activated deformation is rate controlling. With this in mind it is probable that at 0.02 strain dislocation interaction with impurity vacancy dipoles is the rate controlling deformation mechanism at these temperatures.

For temperatures greater than 250°K Figure 7 shows that V^* is greater than 2 500 b^3 and decreases uniformly with strain. Therefore dislocation-dislocation dipole interactions giving rise to predominantly long range stresses which control the work hardening above 250°K, also provide a large contribution to the overall mode of deformation at 0.02 strain. This mechanism has been proposed previously by the authors(10) in explaining the Stage I work hardening behavior of high purity LiF for temperatures above 296°K. However since a unique value is not obtained for ΔH_o at the higher temperatures region (a) in Figure 8 other factors including the temperature dependence of entropy(17) interactions with reorientable impurity vacancy dipoles(18 19) or dynamic strain aging effects(15) may also be active.

CONCLUSIONS

In summary this work has shown that in high purity LiF single crystals work hardening is athermal in nature and may be ascribed to dislocation-dislocation dipole interactions over the entire temperature range studied. The temperature dependence of the work hardening rate is caused by the variation in dislocation debris production between 77°K and 296°K. Vacancies produced in abundance at lower temperatures play an important role in the deformation mechanism at 77°K possibly to such an extent as to affect the use of the Fleischer model in the thermally activated temperature range. At the higher temperatures it is indicated that several rate controlling deformation mechanisms are present but specific identification is not possible at present.

ACKNOWLEDGMENTS

The authors wish to acknowledge valuable discussions with Dr. M. Srinivasan and the experimental assistance of Mr. David Cremers. This research work was supported by the NASA Ceramic Materials Research Program at the University of Washington.

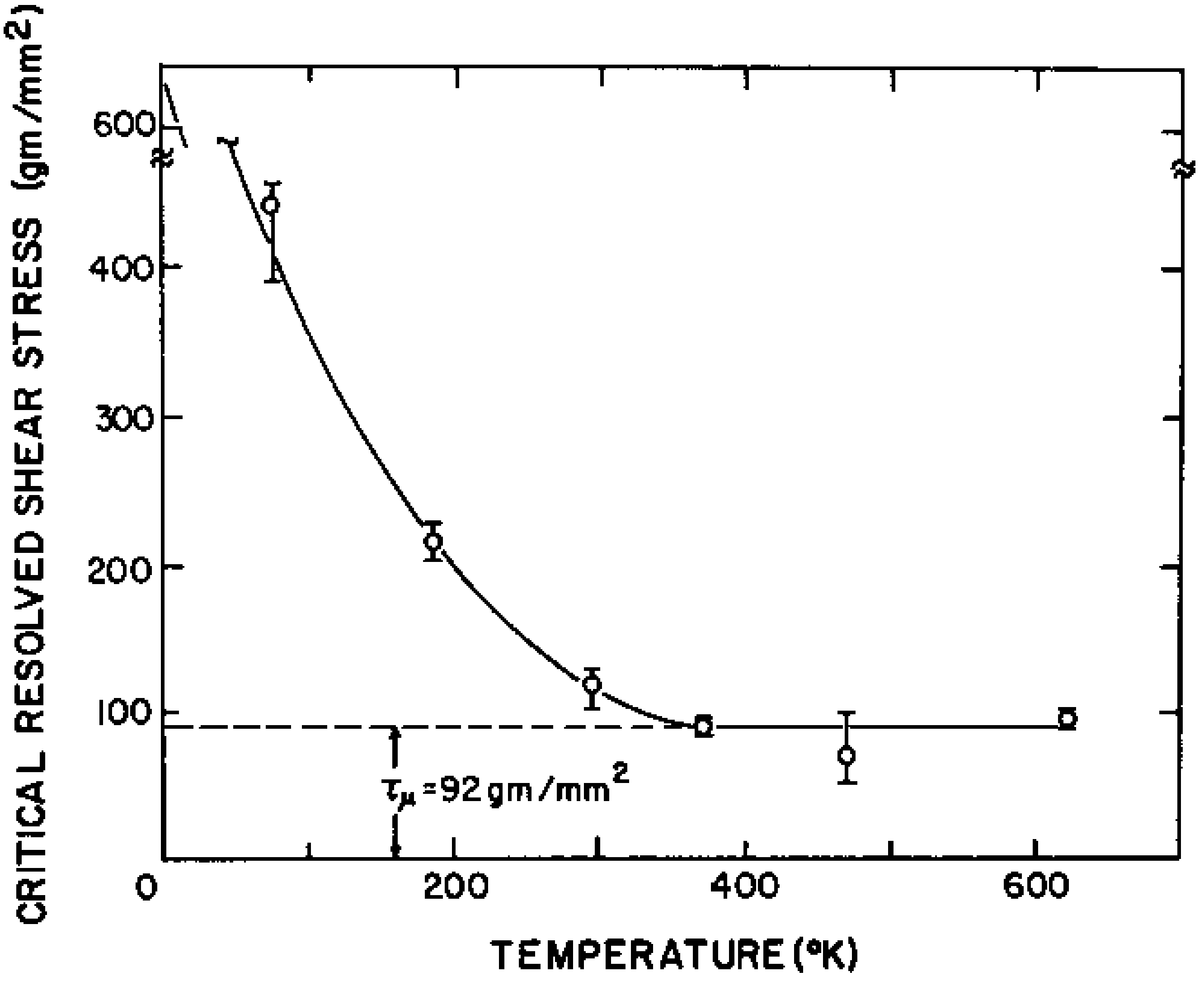

Figure 1 – Effect of temperature on the critical resolved shear stress of high purity LiF single crystals Low temperature line fit by polynomial

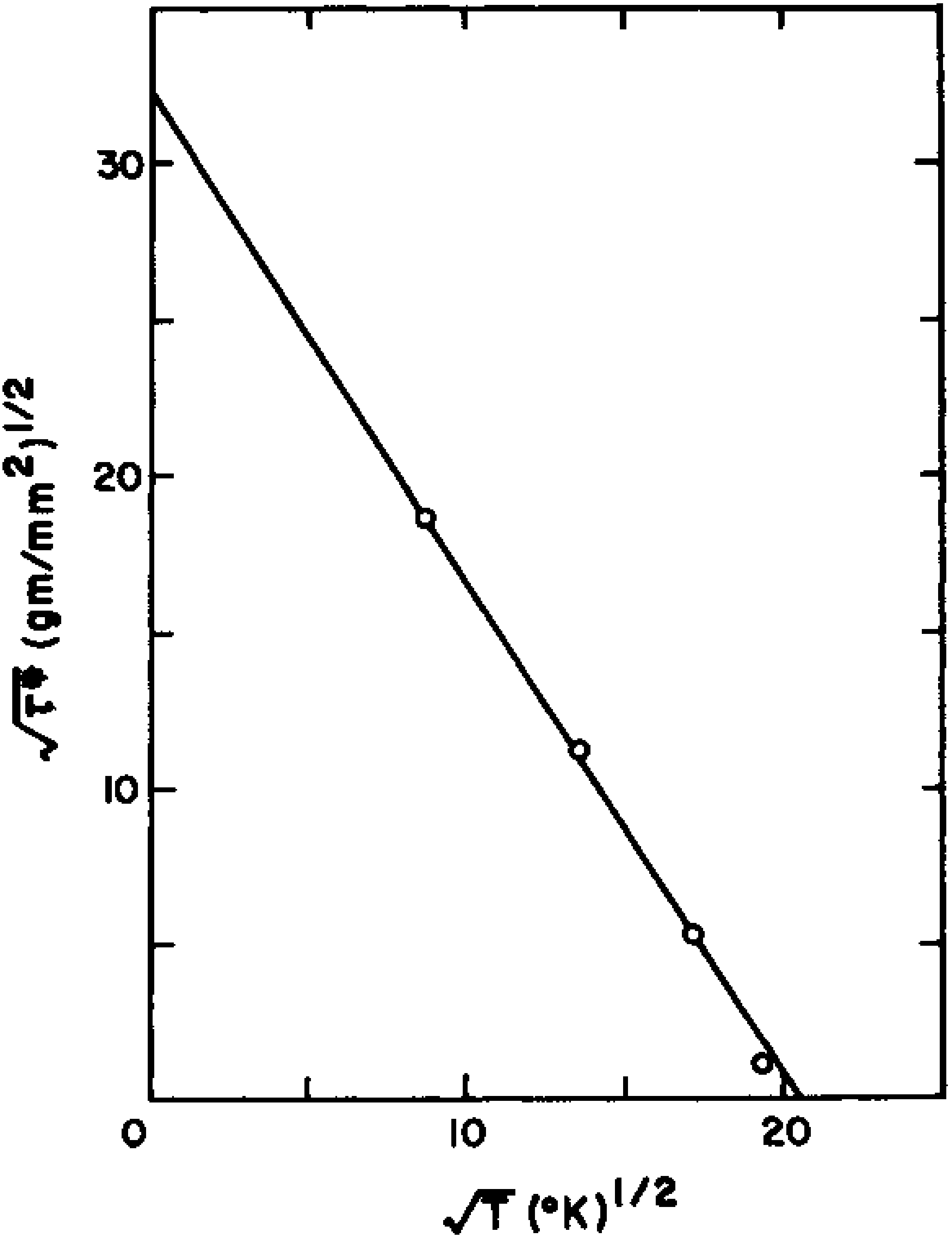

Figure 2 – Effect of temperature on the effective CRSS of LiF single crystals, plotted according to Fleischer's theory

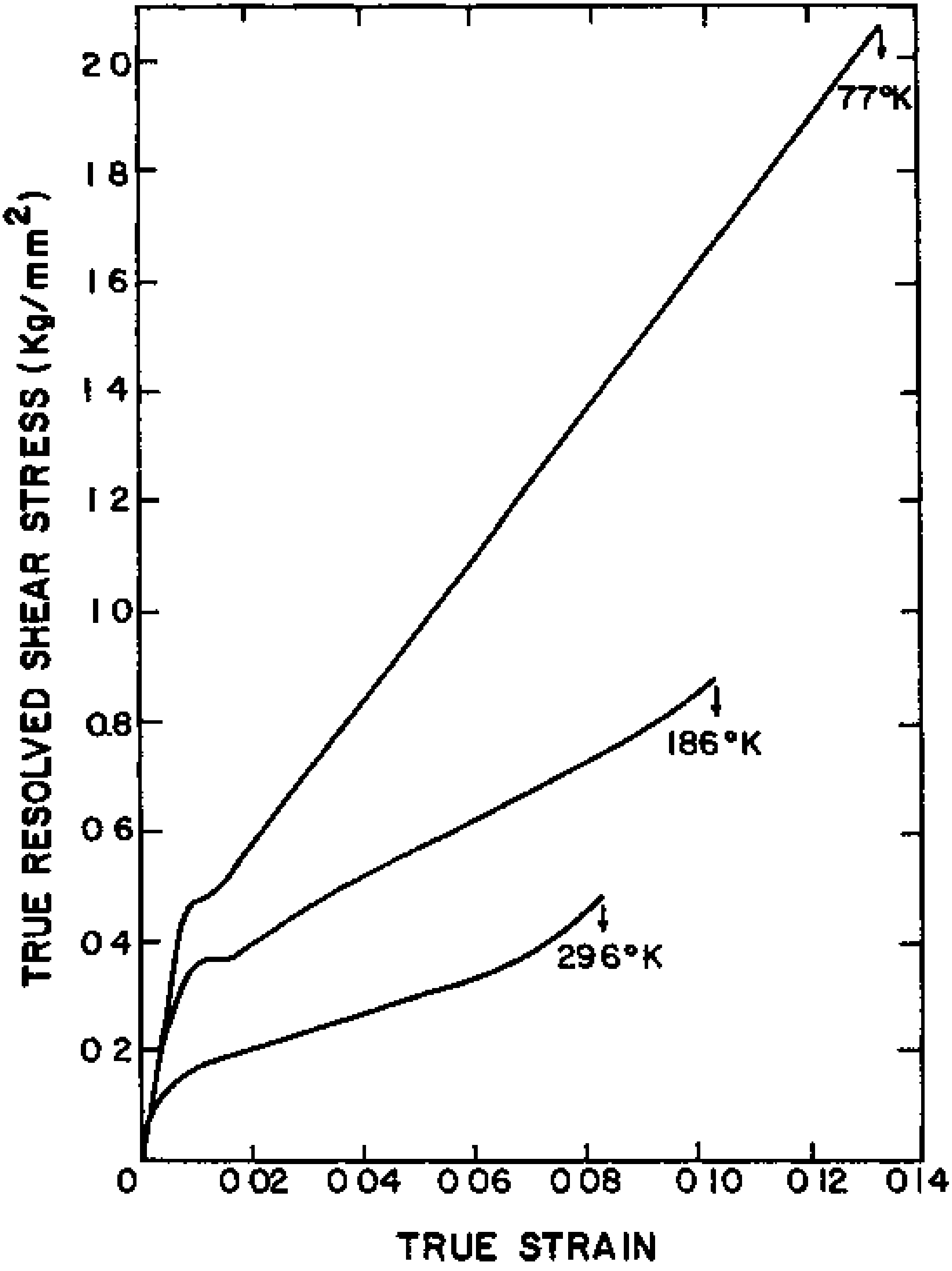

Figure 3 — Stress strain curves for typical high purity LiF samples at 77°K and 186°K at a strain rate of 0 002/min

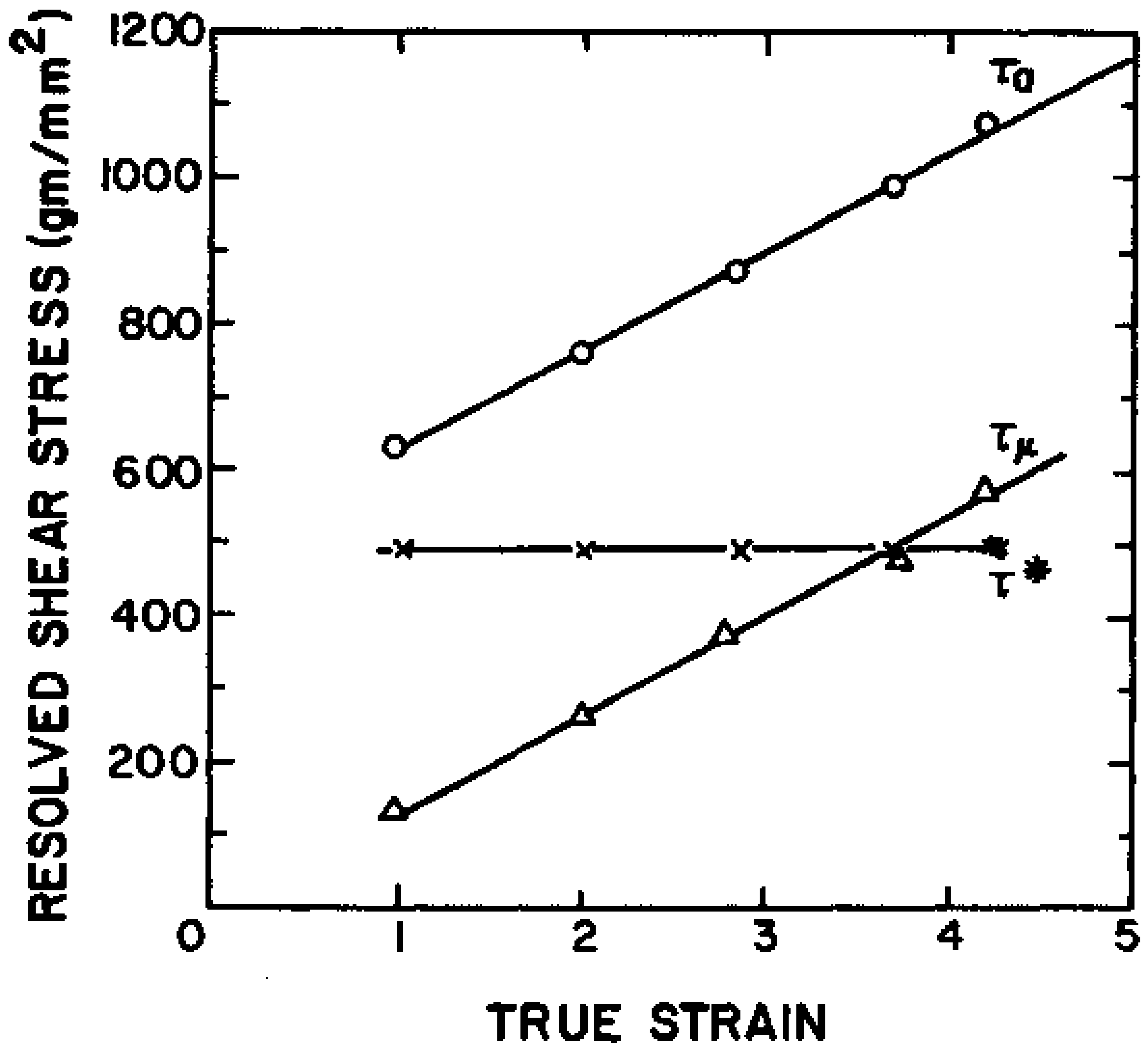

Figure 4a – Stress strain behavior of high purity LiF at 77°K The applied stress τ_a is separated into its athermal and thermal components τ_μ and τ^* utilizing the strain rate change test.

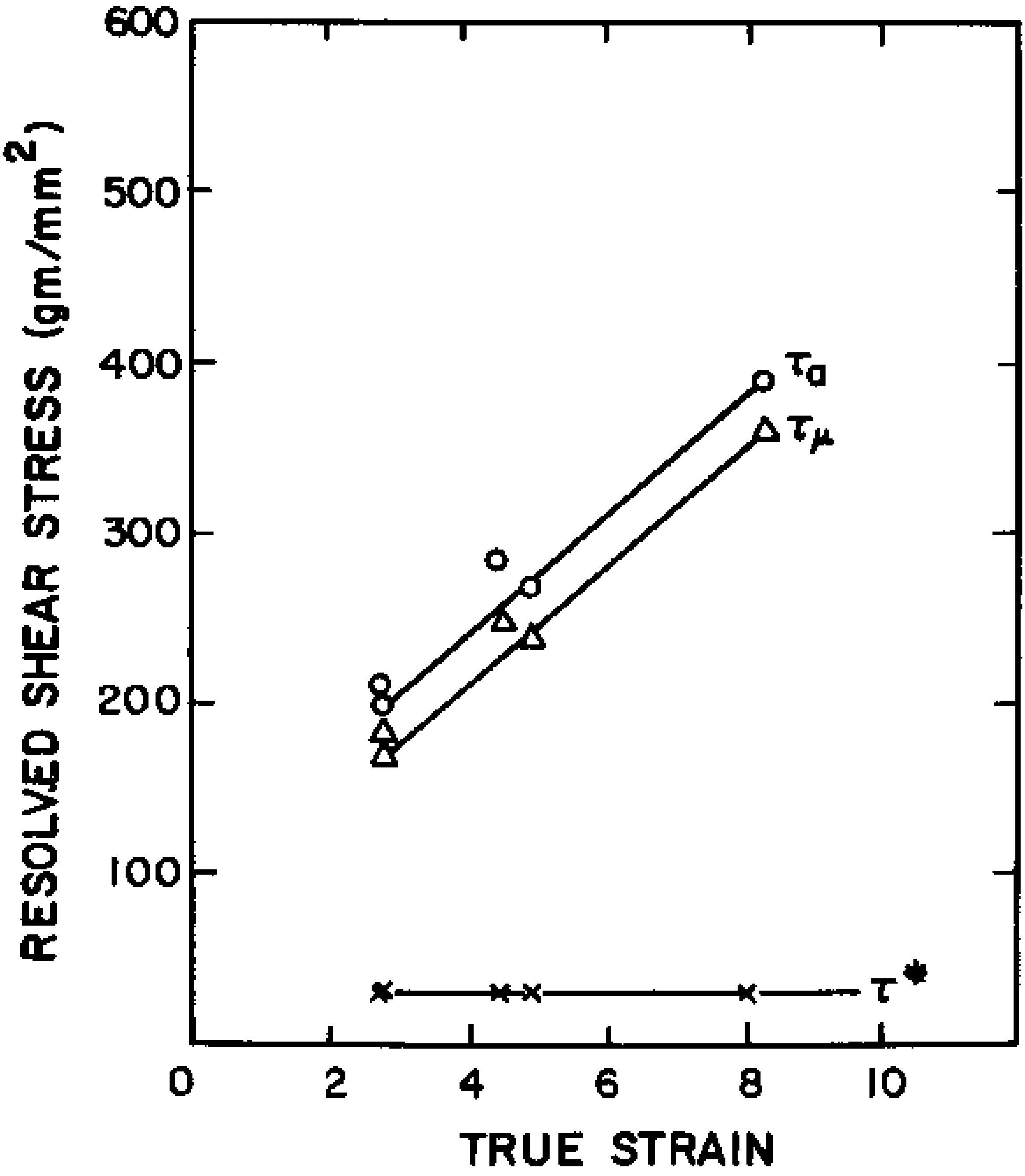

Figure 4b — Stress strain behavior of high purity LiF at 296°K. The applied stress τ_a is separated into its athermal and thermal components τ_μ and τ^* utilizing the double strain rate change test

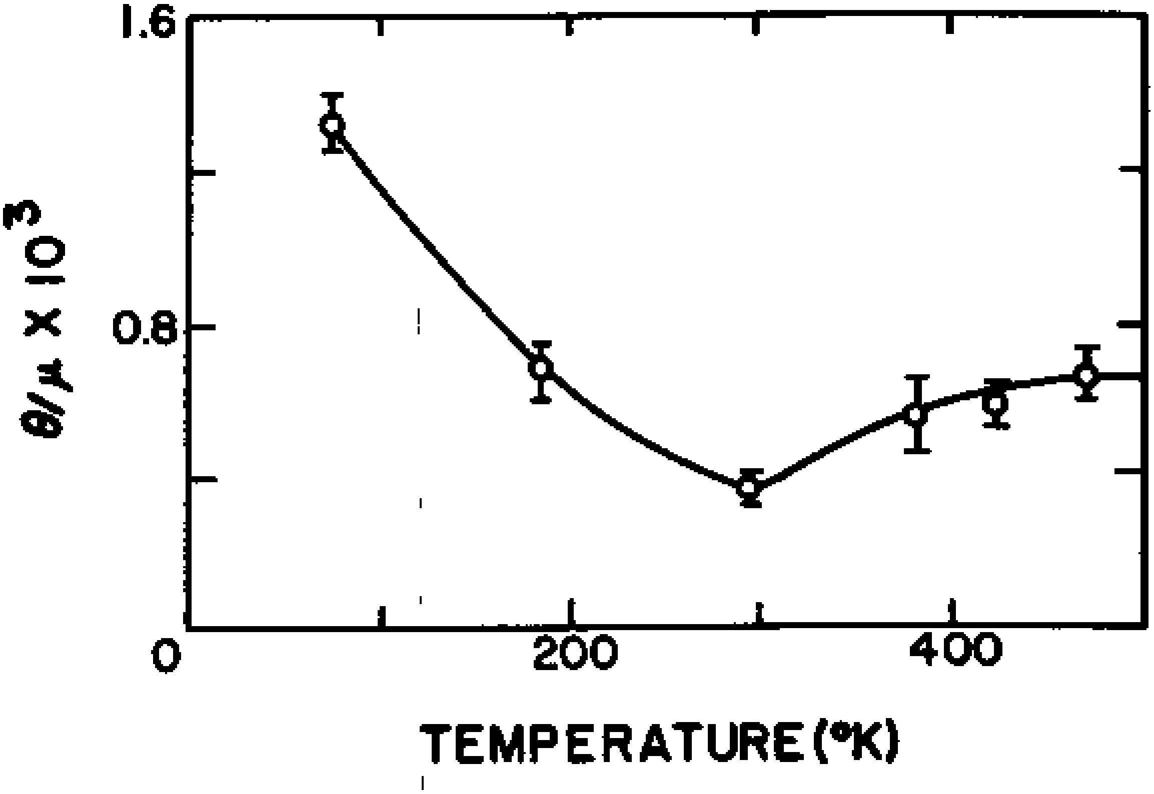

Figure 5 — Temperature dependence of the work hardening rate $\theta = d\tau/d\gamma$ expressed in terms of shear modulus μ for the first stage of work hardening in high purity LiF single crystals

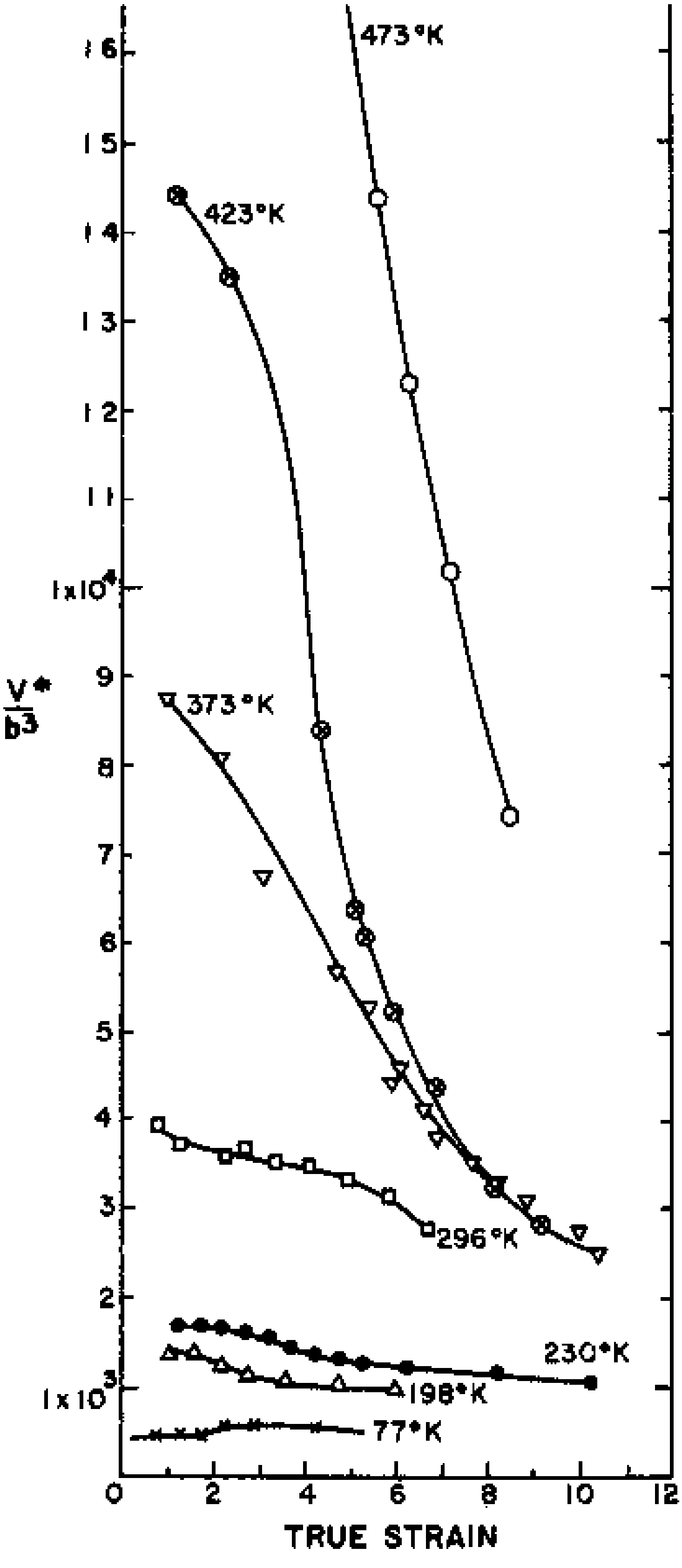

Figure 6 — Strain dependence of activation volume as a function of temperature

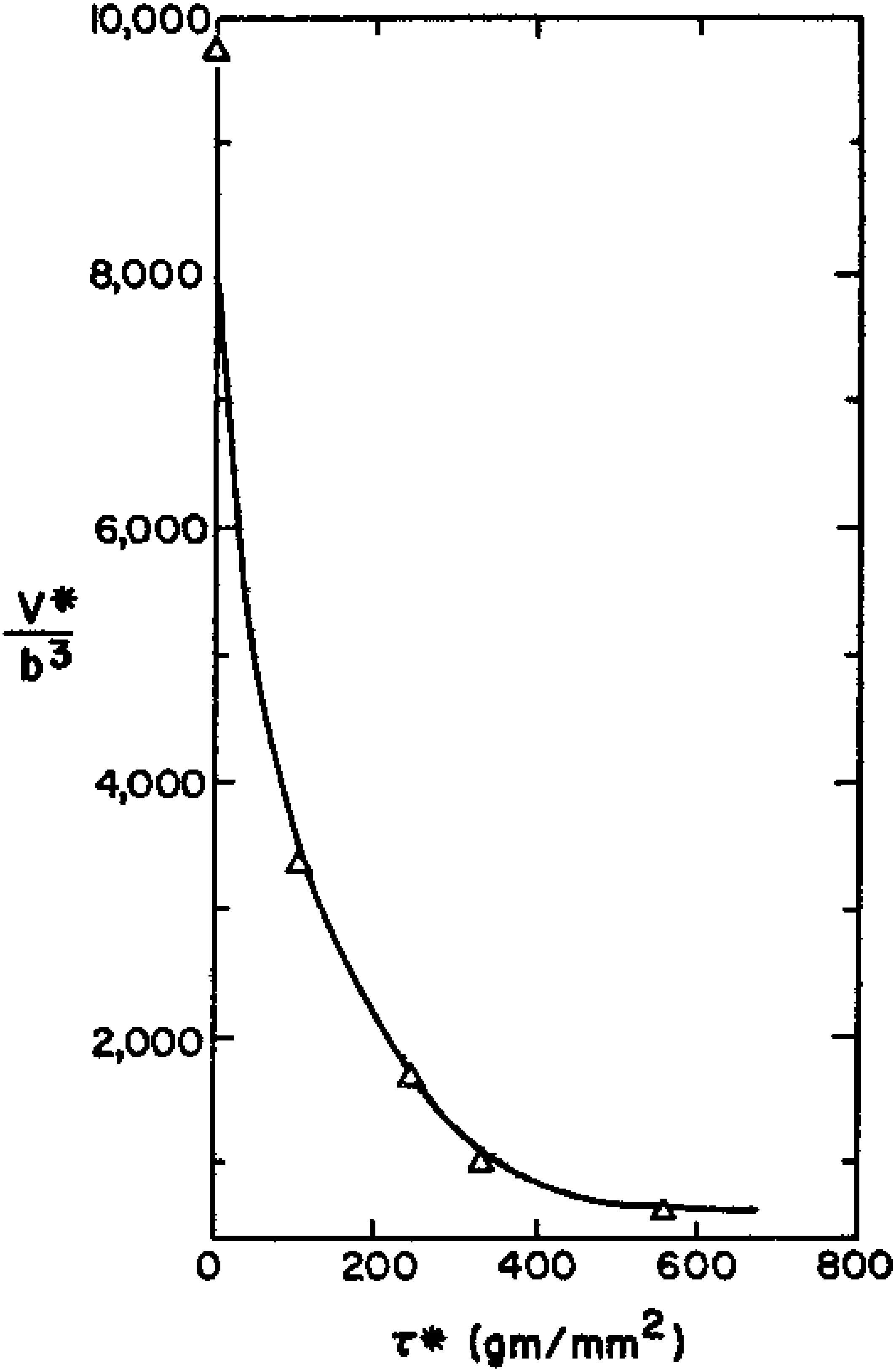

Figure 7 — Activation volume as a function of effective flow stress at 2% strain

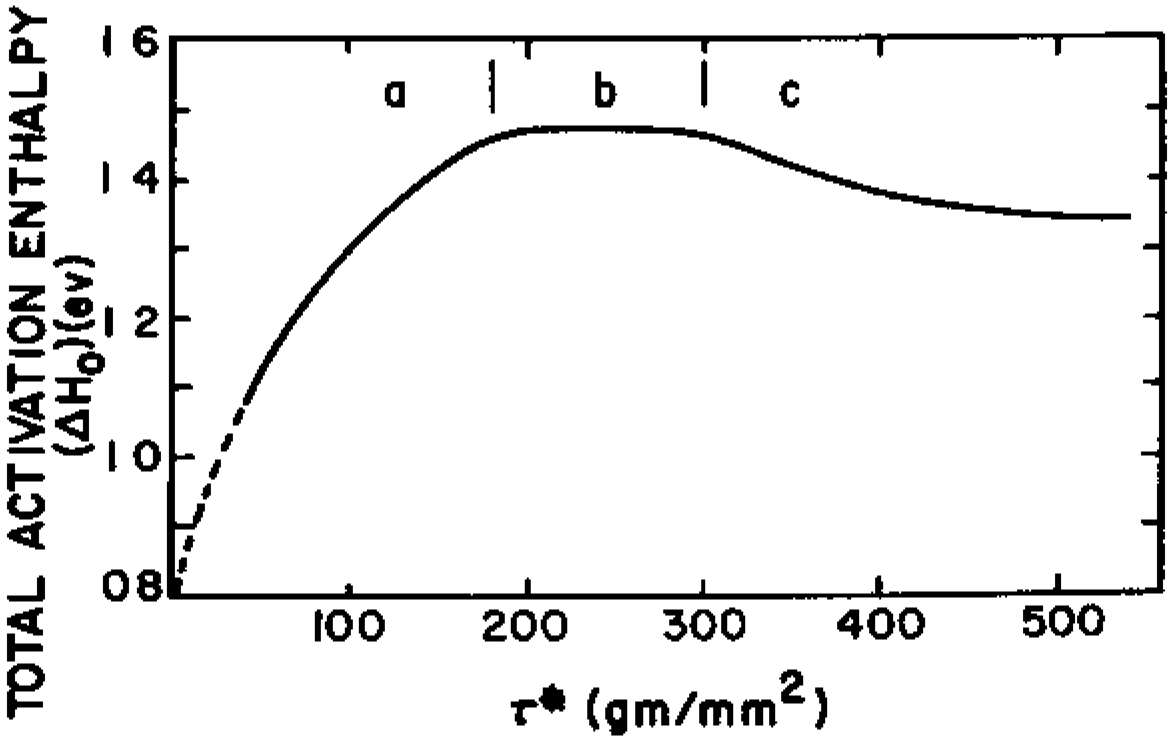

Figure 8 — Effective stress dependence of total activation enthapy ΔH_o

RESUMO

Foi feita uma análise do comportamento na deformação de monocristais de LiF de alta pureza, usando dados de escorregamento e encruamento e parâmetros de deformação ativada termicamente, obtidos no intervalo de temperaturas de 77 a 423°K. Encontrou-se que, enquanto o mecanismo de Fleischer parece ser experimentalmente válido na região de temperatura termicamente ativada, as vacancias produzidas em grande quantidade em 77°K poderiam também desempenhar um papel na determinação da tensão crítica resolvida de cisalhamento naquela temperatura. A dependência com a temperatura da taxa de encruamento parece depender do aumento na produção de "debris" de discordâncias em temperaturas mais baixas. O encruamento é controlado por interações dipolares discordância-discordância de longo alcance dentro do intervalo interno de temperatura considerado.

RÉSUMÉ

Le comportement de la déformation en LiF monocristalin très pur est analysé à travers les resultats obtenus par "yielding" et "work hardening". On a analysé aussi des paramètres de la déformation thermiquement activé dans l'intervalle 77 – 423°K. On a trouvé que tandis que la mécanisme de Fleischer est valable apparentement du point de vie expérimental dans l'intervalle de température thermiquement activé, les lacunes produits en grande quantité a 77°K pourraint jouer un rôle dans la determination de la contrainte effective e cisaillement critique résolue a cette température. La dépendence de la température dans le taux de "work hardening" semble être dependent de l'augmentation dans la production des débris des dislocations aux plus basses temperatures. "Work hardening" est controlé par interaction dipolaire dislocation-dislocation de grand rayon d'action dans tout l'intervalle de la température etudié.

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