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DISLOCATION MECHANISMS IN AN IRON-MANGANESE ALLOY AT LOW TEMPERATURES

by

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ABSTRACT

The effect of strain rate, temperature and interstitial impurity concentration on the flow stress was investigated in a polycrystalline iron-2% manganese alloy. The temperature dependence of the flow stress was found to be independent of the interstitial impurity concentration, removal of interstitials merely decreasing the athermal stress level. Below 160°K, the large temperature dependence of the flow stress was interpreted in terms of the Dorn-Rajnak theory of the Peierls mechanism of plastic deformation. Above 160°K, some other thermally activated intrinsic mechanism seems to be operating.

I. INTRODUCTION

The strong temperature dependence of the flow stress of b.c.c. iron at low temperatures has been studied by numerous investigators and interpreted in terms of several different thermally activated dislocation mechanisms. The following mechanisms have been proposed:

(a) Interaction of dislocations with interstitial impurity atoms[1] or with solute atoms in general,[2]

(b) Intersection of dislocations with clusters of impurity atoms,[3]

(c) Resistance to the motion of dislocations due to jogs on screw dislocations,[4]

(d) Resistance to the motion of dislocations due to the Snoeck effect[5] and,

(e) Interaction of dislocations with the intrinsic resistance of the b.c.c. lattice or Peierls "hills".[6-8]

In previous work,[9] it was found that the plastic behavior of polycrystalline iron containing 2 wt.% Mn (and 100 p.p.m. carbon + nitrogen) is in good agreement with predictions based on the Peierls mechanism[10] from 77°K to 160°K whereas from 160°K to about 370°K another as yet unidentified thermally activated mechanism is operative.

The purpose of the present investigation was to determine the effect of interstitial impurities on the temperature dependence of the flow stress, by purifying the previously investigated alloy with respect to interstitial impurities. The investigation revealed that removal of the interstitial impurities to a level that eliminated Cottrell locking and the Portevin-LeChatelier effect affects neither the strong temperature dependence of the flow stress at low temperatures nor the

general characteristics of the higher temperature behavior. Such purification, however, lowered the athermal yield stress.

II. EXPERIMENTAL PROCEDURE AND RESULTS

The material used in this investigation consisted of an iron 2 wt.% manganese alloy having the following additional elements present: 0.004% C, 0.006% N, 0.05% O, 0.004% S, 0.003% P and 0.001% Si. This was the same material as that studied in the previous investigation and will henceforth be referred to as the "impure material". The as received 3/8" x 3/4" hot rolled bars were cold rolled to 0.08" thickness, recrystallized under argon for 30 minutes at 800°C and further cold rolled to 0.053" thickness. Flat tensile specimens 1/4" wide having a 1.625" long gage section were machined from the sheet and then purified.

The first stage of purification consisted of holding the specimens at 850°C for 24 hours in a stream of hydrogen saturated with water vapor at room temperature. Material prepared in this fashion will subsequently be referred to as "wet hydrogen purified material". It has been estimated that this type of treatment reduces the carbon content of iron to less than 5 p.p.m.[1]

The second stage of purification consisted of holding the specimens in a closed system through which hydrogen was circulated past the heated specimens as well as a zirconium hydride getter. The specimens were held at 850°C and the getter at 800°C. The process was continued for 212 hours. The starting material for this process was wet hydrogen

purified material. Material prepared in this manner will be referred to as "ZrH_2 purified material". The technique used was substantially the same as that described by Stein et al.[1] who have shown that the carbon level at the end of the process is reduced to about 5 parts per billion.

All tensile testing was carried out on an Instron Testing Machine either in controlled temperature baths or at fixed point baths such as boiling liquid nitrogen, etc... When fixed points were used the temperature was kept constant to better than $\pm 1^{\circ}C$ whereas in all other tests the temperature variation was less than $\pm 2^{\circ}C$.

Test of Purity

It was not possible to determine the purity achieved at each stage of the purification because the resulting level of interstitial impurities was well below the limit of sensitivity of standard analytical techniques. It was possible, however, to obtain a qualitative measure of the extent of purification from the appearance of the stress strain curves for the three materials. Figure 1 shows the stress strain curves for the three different materials tested at 300°K. It can be seen that the effect of wet hydrogen purification on the impure material was threefold. The upper yield point was entirely eliminated, the lower yield stress was decreased and the extent of Lüders band strain was reduced by about a factor of ten. The three effects provide strong evidence that a significant reduction of the interstitial content was achieved. The effect of ZrH_2 purification was to eliminate all yield and Lüders band phenomena. These trends are in substantial

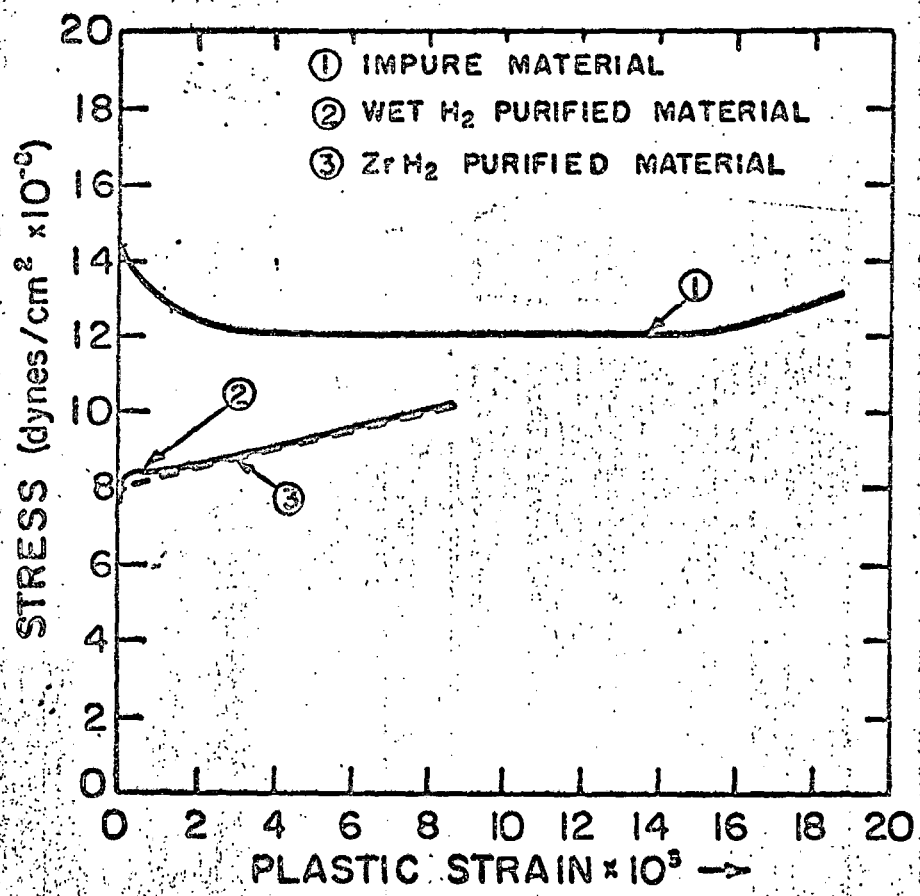

Figure 1. Stress-strain curves at 300°K on original specimens.

agreement with those obtained by Stein et al.[1]

Figure 2 shows the stress-strain curves for the three materials tested at 523°K. It can be seen that the impure and wet hydrogen purified materials exhibited a marked Portevin-LeChatelier effect indicating that interstitial impurities were still present in the wet hydrogen purified material. In contrast to these results however, the ZrH_2 purified material exhibited no sign of the Portevin-LeChatelier effect thus indicating that the concentration of interstitial impurities was so low as to make dislocation-impurity interactions undetectable.

Stress-Temperature Relationships

For convenience in the theoretical analysis, the data are presented in terms of the shear stress for flow, τ, the shear strain γ and the shear strain rate $\dot{\gamma}$. Tensile specimens of the wet hydrogen and ZrH_2 purified materials were prestrained at 423°K at a shear strain rate of 1.54×10^{-5}/sec to stress levels of 3.64×10^8 and 3.21×10^8 dynes/cm^2 respectively. Immediately following this prestrain the temperature bath was changed and each specimen tested in tension at either $\dot{\gamma} = 1.54 \times 10^{-5}$/sec or 1.54×10^{-3}/sec. The values obtained for the stress were within ±2% of the stated shear stress and ±5% of the reported values of the strain rate. The tensile stresses were converted to shear stresses using a factor of 1/2 and the shear strains obtained from engineering strains using a factor of 3/2. The shear stress for the initiation of flow at the test temperature was determined by taking a $\gamma = 9.24 \times 10^{-3}$ offset from the modulus line at which good accuracy was achieved in determining $\dot{\gamma}$ and the associated flow stress τ.

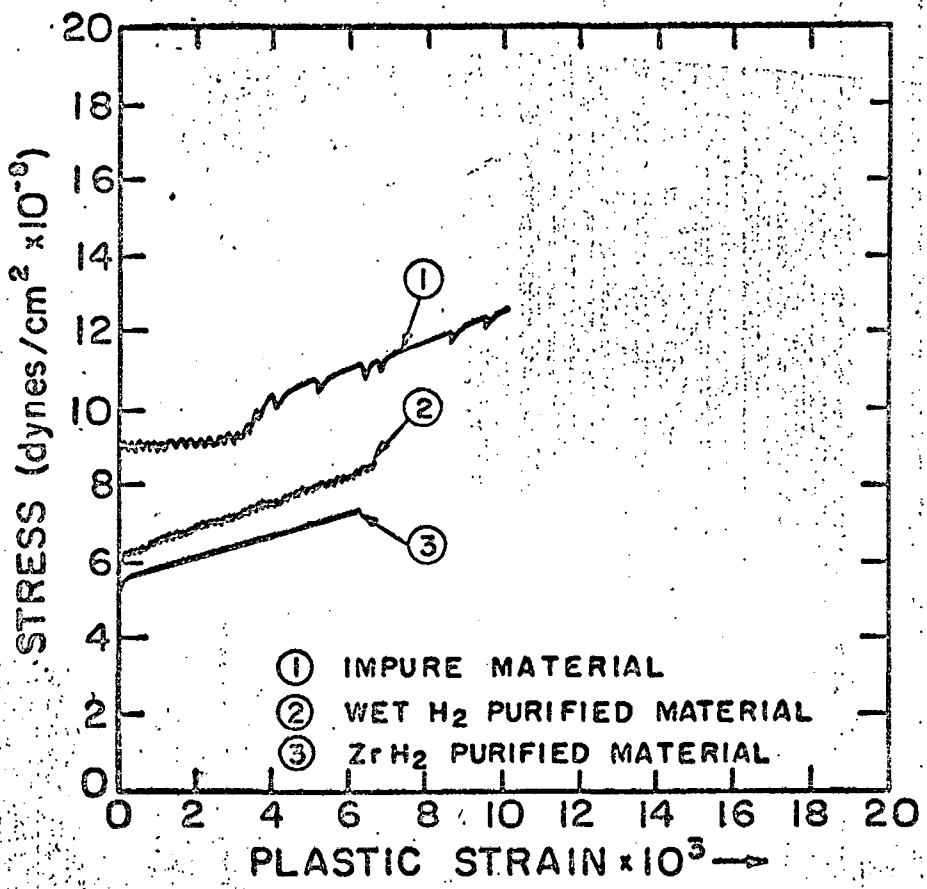

Figure 2. Stress-strain curves at 523°K on original specimens.

The experimental data for the wet hydrogen and ZrH_2 purified materials are given in Fig. 3. Similar data for the impure material, as determined earlier,[9] are given in Fig. 4 for comparison. The ZrH_2 purified material behaved in a brittle manner at temperatures below about 100°K. Metallographic examination showed that twinning occurred in the immediate vicinity of the fracture.

III. DISCUSSION

Examination of Figs. 3 and 4 shows that the flow stress dependence on the temperature is independent of the concentration of interstitial impurities. In earlier work,[9] the impure material was tested in three different strain hardened states and it was established that the temperature dependence of the flow stress did not depend on the strain hardened state. The data from the previous investigation were analyzed in terms of Dorn and Rajnak's[10] model of dislocation motion controlled by nucleation of a pair of kinks over Peierls barriers and were found to fit the model extremely well in the range 77° to 160°K. In view of the earlier success of that approach and of the observation that the temperature dependence of the flow stress is independent of the concentration of interstitial impurities or of the strain hardened state (i.e. dislocation density), it is reasonable to apply the same analysis to the present data.

The stress which must be applied to cause plastic flow is given by

$$\tau = \tau^* + \tau_A \qquad (1)$$

where τ^* is the stress required to aid the thermal activation of the

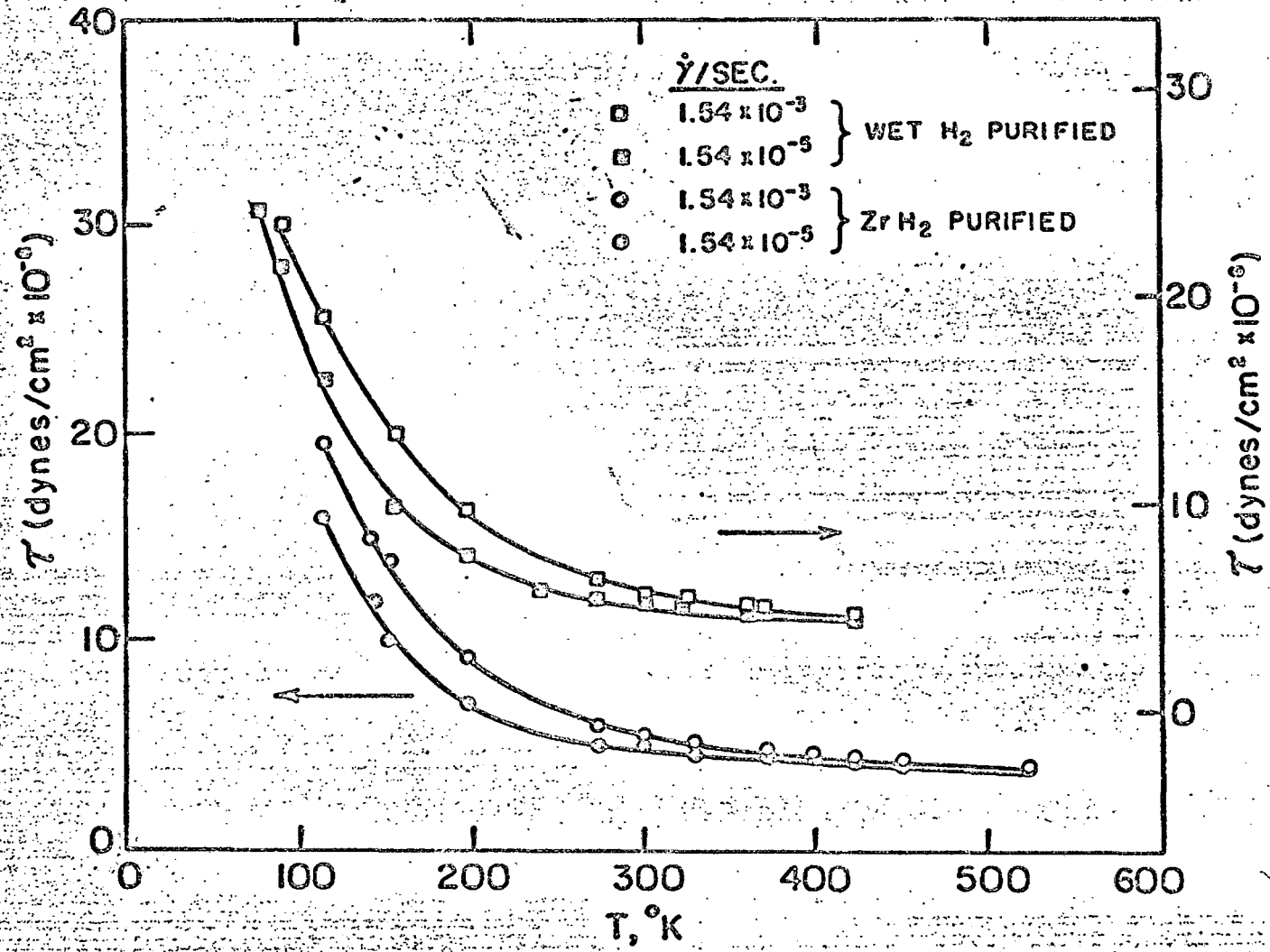

Figure 3. Flow stress vs. temperature curves for the wet hydrogen and ZrH_2 purified materials prestrained to 3.64×10^8 and 3.21×10^8 dynes/cm^2 respectively at 423°K.

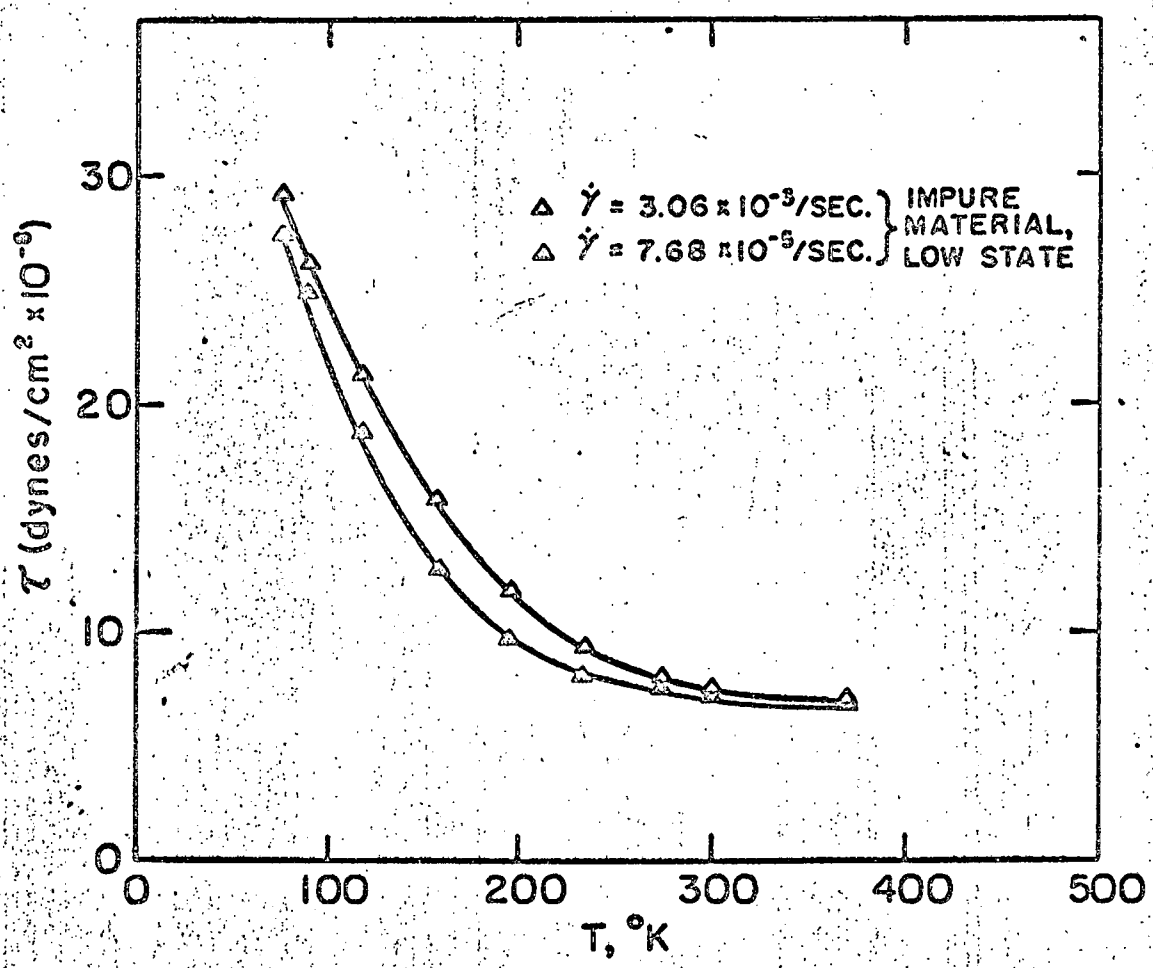

Figure 4. Flow stress vs. temperature curves for the impure material prestrained to 6.89×10^8 dynes/cm^2 at 300°K.

rate controlling mechanism and will therefore increase rapidly as the temperature decreases, and τ_A is the stress required to overcome the athermal dislocation interactions and will consequently decrease with increase in temperature in the same way as the shear modulus of elasticity.

Most low-temperature thermally activated mechanisms are characterized by a relationship having the form

$$\dot{\gamma} = \dot{\gamma}_o \exp(-U/kT) \qquad (2)$$

where $\dot{\gamma}$ is the shear strain rate, $\dot{\gamma}_o$ is a frequency factor, U is the energy which must be supplied by a thermal fluctuation, T is the absolute temperature and k is the Boltzmann constant. A critical distinction between mechanisms can often be obtained from the activation volume, v, which is given by

$$v = -\left(\frac{\partial U}{\partial \tau^*}\right)_T = kT\left(\frac{\partial \ln \dot{\gamma}}{\partial \tau^*}\right)_T \simeq kT\left(\frac{\Delta \ln \dot{\gamma}}{\Delta \tau^*}\right)_T \qquad (3)$$

The activation volumes deduced from the data of Fig. 3 by means of Eq. (3) are plotted in Fig. 5 in terms of the stress τ. Activation volumes for the impure material in three different strain hardened states are also included in the figure. The stress axes were displaced with respect to each other in order to bring all points to a single curve and to correct for the various values of τ_A. The extent of the relative displacement necessary for the points to coincide was found to be closely related to the differences in the flow stresses of the five states at 300°K.

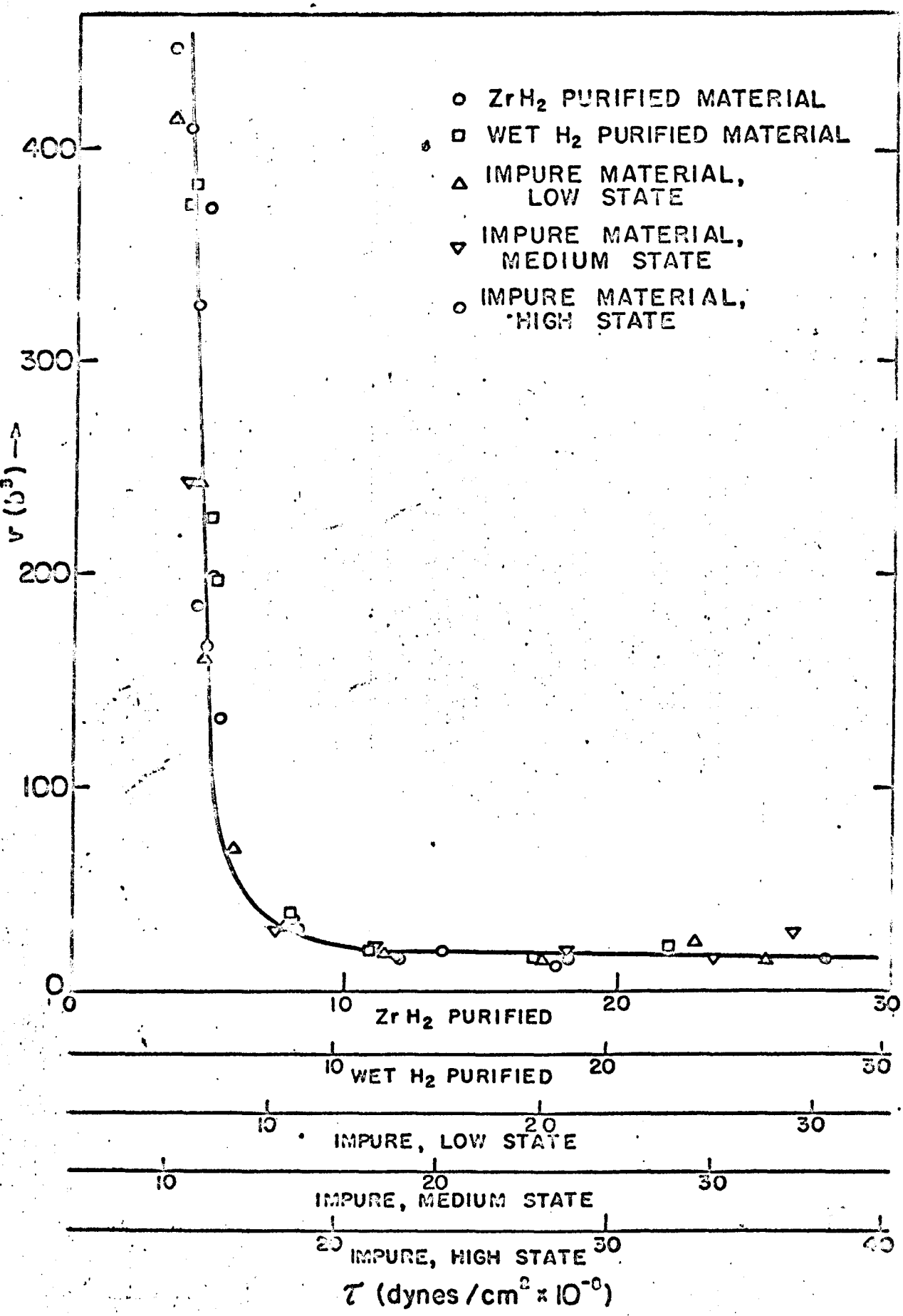

Figure 5. Activation volume vs. stress plot.

The apparent activation energy U, can be written

$$U = \left[\frac{\partial \ln \dot{\gamma}}{\partial(-\frac{1}{kT})}\right]_{\tau} \simeq \left[\frac{\Delta \ln \dot{\gamma}}{\Delta(-\frac{1}{kT})}\right]_{\tau} \qquad (4)$$

U was calculated for all five states and plotted against the stress in Fig. 6, with the stress axes displaced as in Fig. 5. Although scatter is apparent, the tendencies shown by the three materials are reasonably similar.

The analyses of Figs. 5 and 6 suggest that the Peierls mechanism is superseded at low stresses (high temperatures) by some other mechanism. For the Peierls mechanism,[10]

$$\dot{\gamma}_o = \rho a b \nu \frac{L}{w} \qquad (5)$$

where ρ is the dislocation density, a is the distance between adjacent Peierls valleys, ν is the Debye frequency, L is the length of dislocation advanced a distance a by the nucleation of a single pair of kinks and w is about the critical separation of the pair of embryonic kinks at the saddle point as the pair is nucleated. The temperature dependence of the thermal stress for flow, τ^*, is given by the theory as

$$\tau^*/\tau_p = f_1\left(\frac{U_n}{2U_k}\right) \simeq f_1\left(\frac{T}{T_c}\right) \qquad (6)$$

where τ_p is the Peierls stress, $U = U_n$ is the energy which must be supplied by a thermal fluctuation in order to nucleate a pair of kinks, U_k is the energy of an isolated kink, T is the test temperature and T_c is the critical temperature at which $\tau^* = 0$ and is defined by

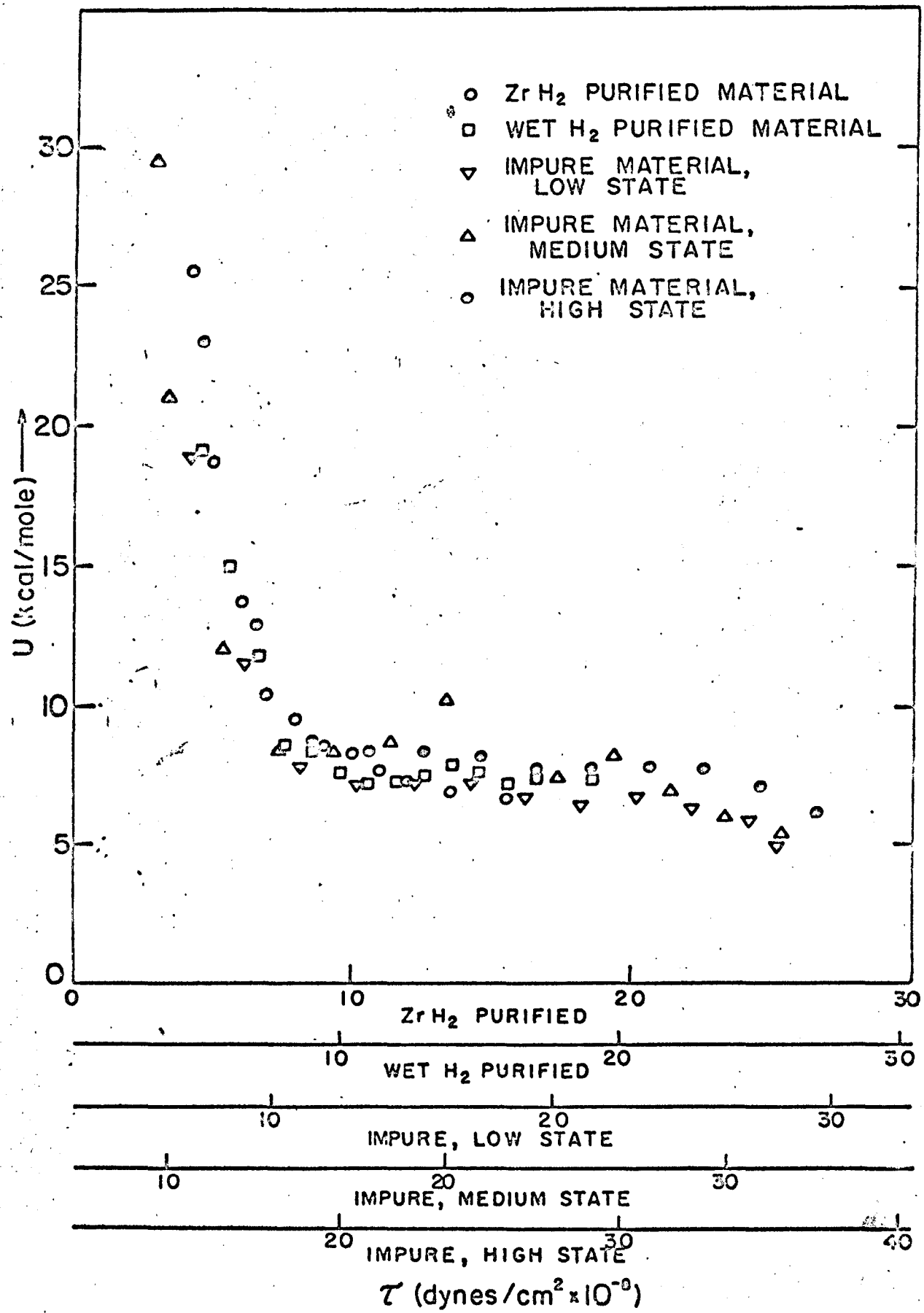

Figure 6. Activation energy vs. stress plot.

$$\dot{\gamma} = \dot{\gamma}_o \exp(-2U_k/kT_c) \tag{7}$$

and consequently depends on $\dot{\gamma}$. Both U_k and τ_p change with temperature in the same way as the shear modulus but this effect is small and has been neglected here. The theoretical τ^*/τ_p versus T/T_c curve of Eq. (6) is given by the solid line of Fig. 7. Since τ_p and T_c are not directly available from the data, the data were compared to master plots of the theoretical relationship for different values of τ_p and T_c. All of the data was examined in this way, this included a re-analysis of the data for the impure material which yielded essentially the same parameters as had been determined earlier (c.f. reference 9). The values of τ_p, T_c and τ_A obtained in this way are listed in Table 1. The data points suitably reduced by means of the parameters of Table 1 are given in Fig. 7, where it can be seen that the upper temperature limit for agreement with the Peierls mechanism has not been significantly affected by the reduction of the interstitial impurity concentration. The values of $2U_k$ in Table 1 were obtained from

$$\dot{\gamma}_1/\dot{\gamma}_2 = \frac{\exp(-2U_k/kT_{c1})}{\exp(-2U_k(kT_{c2})} \tag{8}$$

The kink energies obtained in this way from the different purity materials agree fairly well among themselves. The scatter in the values obtained is due to the high sensitivity of Eq. (8) to errors in the determination of the critical temperatures T_{c1} and T_{c2}. This same effect is reflected by the scatter in the values of U given in Fig. 6. The line energy is

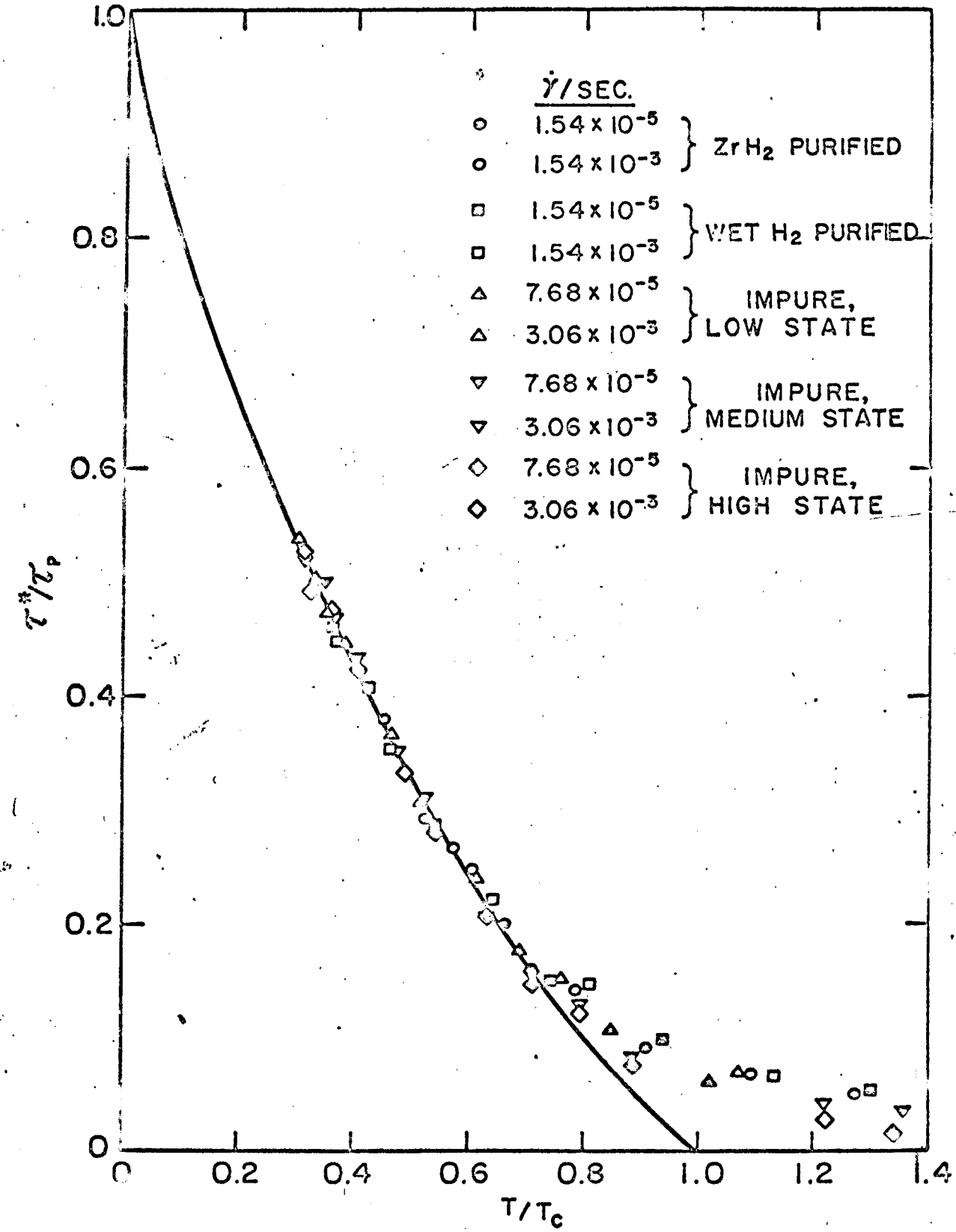

Figure 7. Temperature dependent component of stress vs. temperature, in dimensionless coordinates, comparing data with theoretically predicted curve.

related in terms of the theory to the parameters U_k and τ_p by

$$\frac{2\pi U_k}{a\Gamma_o} = 5.67 \sqrt{\frac{\tau_p ab}{\pi\Gamma_o}} \qquad (9)$$

where Γ_o is the line energy, and it has been assumed that the line energy varies sinusoidally with displacement along the Peierls hills. The values of Γ_o obtained are given in Table 1. These values vary between 1.3 and 1.9 Gb^2 thus showing reasonable values of line energy.

Table 1

	τ_p dynes/ $cm^2 \times 10^{-8}$	T_{c1} °K	T_{c2} °K	τ_A dynes/ $cm^2 \times 10^{-8}$	U_k ergs $\times 10^{+13}$	Γ_o ergs/ cm $\times 10^4$
Impure, high state	47.5	220	245	13.9	5.46	7.53
Impure, medium state	47.5	220	245	9.0	5.46	7.53
Impure, low state	45.0	230	255	4.9	5.98	9.54
Wet H_2 purified	45.0	210	242	3.0	5.07	6.85
ZrH_2 purified	45.0	215	250	2.8	4.93	6.48

In view of the above analysis one can summarize the behavior of iron below about 160°K as follows. At very low temperatures, below 70°K to 100°K depending on purity, twinning appears to be a major mode of deformation as has also been observed by Stein et al.[1] At temperatures above the twinning range and up to about 160°K the temperature dependence

of the flow stress is independent of the strain hardened state and independent of the concentration of interstitial impurities. This suggests an intrinsic lattice resistance to the motion of dislocations. Furthermore, the independence of the activation volume and activation energy on the strain hardened state or impurity content provides additional evidence to the operation of a dislocation mechanism independent of these variables. Finally, the good fit of the data with the theoretical predictions of the Peierls mechanism confirms that this most probably is the rate controlling mechanism of deformation.

The data for iron above about 160°K could not be interpreted. The results are characterized by both a temperature and a strain rate dependence of the flow stress thus indicating a thermally activated dislocation mechanism. The data derived from the impure material and wet H_2 purified material could not be examined in detail at temperatures above ∿400°K because of the superposition of both dynamic and static strain-aging effects. The data for the ZrH_2 purified material, however, seems to show an athermal region beginning at ∿470°K for $\dot{\gamma} = 1.54 \times 10^{-5}$. Throughout the high temperature region, the activation volume increases rapidly with decreasing stress (see Fig. 5) but this characteristic is affected neither by the strain hardened state nor by the purity of the material. The change in activation volume with strain at a particular temperature was determined by a strain rate change experiment at 340°K and is given in Fig. 8. It can be seen that the activation volume remains constant and is unaffected by strain hardening. The evidence is inconsistent with the intersection mechanism and any other known

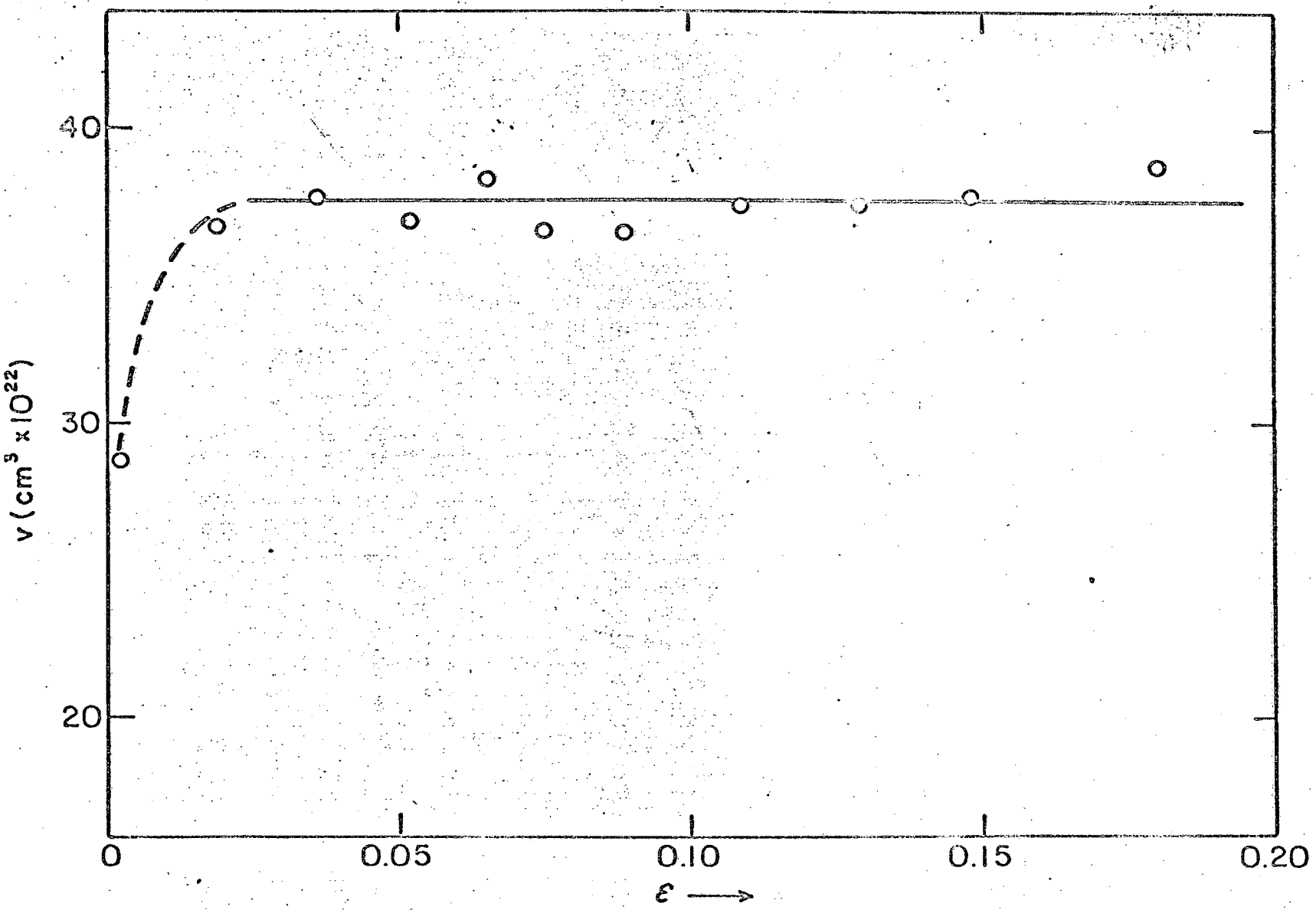

Figure 8. Activation volume vs. engineering strain curve for wet hydrogen purified material at 340°K.

mechanism. The data seem to indicate the operation of dislocation interactions with some intrinsic structural barrier other than the Peierls hills, the barrier being such that the activation volume increases rapidly with a decrease in stress to very high values. It was thought that magnetic domains might provide this kind of intrinsic barrier. In order to test this possibility a simple experiment was carried out. Identical specimens were tested with and without an applied magnetic field large enough to magnetically saturate the specimen (∿30 Kilogauss) and hence to introduce essentially a single magnetic domain in each grain of the specimen. The tests were carried at about 240°K where the presence of a magnetic effect should have been most apparent. The application of a magnetic field was found to have no effect on the flow stress or the activation volume. The nature of the intrinsic barrier is yet open to speculation.

Returning to the Peierls controlled temperature region, the results of this investigation are found to be in disagreement with those obtained by Stein et al.[1] In their work on iron single crystals containing comparable levels of interstitial impurities, they found a significant drop of the temperature dependence of the flow stress with decreasing interstitial concentration. The source of disagreement is difficult to trace; however in more recent work, Keh[11] indicates that the flow stress of iron single crystals does not seem to be much affected by ZrH_2 purification, thus confirming the trends reported here for polycrystalline Fe.

In some work on microyielding of iron at low temperatures, Kossowsky and Brown[12] have shown that the temperature dependence of the stress for dislocation motion is strongly dependent on the strain. This is explained in terms of a number of different Peierls type barriers acting in 3 stages. The initial small reversible strains are interpreted as the straightening out of pre-existing kinks in a randomly oriented dislocation, a second stage is thought to represent the generation of a pair of kinks and a last stage is associated with a Peierls controlled cross slip. It is clear that the strain sensitivity of the deformation process fits in well with the concepts of the Peierls mechanism since pre-existing kinks will undoubtedly be straightened out at low stresses. Moreover, Kossowsky and Brown's suggestion of a Peierls controlled cross slip is reasonable, however this cannot be viewed as a separate mechanism since the Peierls barrier confronting a dislocation that is about to cross slip or about to continue on its original slip plane is identical. In both these cases, a pair of kinks must be nucleated over a Peierls barrier and no additional work is required in the case of cross slip of an undissociated dislocation. Kossowsky and Brown's second stage probably merely represents a transition between straightening out of existing kinks and nucleating new ones.

IV. CONCLUSIONS

The results obtained can be summarized as follows:

(1) The temperature dependence of the flow stress is independent of the strain hardened state or the interstitial impurity

concentration.

(2) Below 160°K, the large temperature dependence of the flow stress can be explained on the basis of the Dorn-Rajnak theory of the Peierls mechanism of plastic deformation.

(3) Above 160°K, some other thermally activated intrinsic mechanism seems to be operating.

(4) Removal of interstitials merely decreases the athermal stress level.

V. ACKNOWLEDGEMENT

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