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STRESS RELAXATION AND HILLOCK GROWTH IN THIN FILMS

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Abstract

The relaxation of thermal stress in a thin film adhering to a substrate of differing expansion coefficient is discussed. Good agreement is found between literature data on relaxation during isothermal anneals of Pb films at up to 350 K and model calculations based on a state variable description of plastic flow.

The stress system during relaxation is explored, and the absence of diffusional creep is explained. The plasticity-dominated relaxation process suggested by this analysis is shown to be in good qualitative agreement with data on rapid relaxation over the course of a cycle between room and cryogenic temperatures.

The implications of this picture for long-range material transport in the film are discussed. In particular, it is shown that hillock volume should increase over the course of a temperature cycle. Finally, a mechanism for hillock nucleation based on grain boundary sliding is suggested.

1. Introduction

The use of thin films of low melting point materials in modern electronic devices has recently focused attention on damage processes in these structures. Some of these damage processes are mechanical in origin. For example, a thin film bonded to a substrate may experience large stresses due to differential thermal expansion under temperature changes. For low melting point materials, both diffusive and plastic deformation processes may operate to relax this stress at room temperature and below. Prediction of device failure due, for example, to hillock or void growth requires knowledge of these stress relaxation mechanisms and the rates at which they operate. This paper attempts to define a framework within which the required quantitative calculations can be made.

Figure 1 depicts a common if simplified configuration. A film of thickness of the order of microns or less has been depostied on a relatively massive substrate. The film is polycrystalline with a grain size of the order of its thickness. Under a temperature change, a biaxial strain will be introduced in the plane of the film; in the absence of relaxation this strain will be elastic and will be given by

$$\varepsilon_e = \int_{T_0}^{T_1} (\alpha_s - \alpha_f) dT \qquad (1)$$

where α_s, α_f are the coefficients of thermal expansion for the substrate and the film, respectively. In films of moderate thickness, relaxation processes occur which reduce this strain value substantially.

These effects have been studied experimentally by Caswell et al,[1] Gangulee,[2] and Murakami,[3] among others. Various mechanisms have been proposed to account for the observed relaxation as a function of temperature, and deformation mechanism maps have been prepared.[4] Generally, the models adopted to describe plastic flow were derived based on creep data.

In this work, we analyze recent data reported by Murakami on isothermal stress relaxation as a function of time. The constitutive equations adopted to describe plastic deformation were derived primarily on the basis of load relaxation tests on bulk specimens. The effect of the substrate constraint on possible stress relaxation mechanisms is considered in detail. On the basis of these results, it is concluded that the overall stress relaxation is dominated by plastic flow even in the presence of rapid grain boundary diffusivity. This picture is in good qualitative agreement with observed behavior during cyclic temperature changes.

The implications of the interaction between plasticity and grain boundary diffusion for hillock growth are discussed. Finally, a mechanism for nucleation of hillocks based on grain boundary sliding is suggested.

2. Constitutive Relations for Plasticity

In order to determine the possible role of plastic deformation during stress relaxation in these thin films it is necessary to adopt a model for plasticity. We choose the state variable approach of Hart,[5] which has enjoyed considerable success in describing the nonelastic behavior of a number of bulk materials.[6] In particular, the basic experimental confirmation of this model has been the load relaxation test.

In such a test, elastic strain of the testing system is converted into non-elastic strain of the specimen; the change in the applied load is related to the sample strain through a machine modulus. In the present problem, elastic strain in the film plane is converted to plastic strain; the rate of change of the film stress is then given by

$$\dot{\sigma} = M\dot{\epsilon}_e = -M\dot{\epsilon} \tag{2}$$

where $\dot{\epsilon}_e$ is the elastic strain rate, $\dot{\epsilon}$ the nonelastic strain rate (constant temperature), and M an appropriate modulus.

The relationship between stress and plastic strain rate is given by[5,6]

$$\log(\frac{\sigma^*}{\sigma}) = (\frac{\dot{\epsilon}^*}{\dot{\epsilon}})^\lambda \qquad (3)$$

where σ^* is a hardness parameter (playing the role of a yield stress); $\dot{\epsilon}^*$ a rate parameter; and λ a constant, found experimentally to be equal to 0.15 in bulk materials.

Although plastic deformation is the result of dislocation glide on slip planes, the kinetics are determined by climb processes at the leading edge of dislocation pileups. The parameters σ^* and $\dot{\epsilon}^*$ are not independent; they are found to be related by

$$\dot{\epsilon}^* = (\frac{\sigma^*}{G})^m f\ e^{-Q/kT} \qquad (4)$$

where m is a materials constant (m = 4.0 for Pb[7]), G a shear modulus, f a frequency factor, and Q the activation energy for self-diffusion. The significance of eq. (4) is that once the relationship between σ^* and $\dot{\epsilon}^*$ is established at a given temperature, it is known at all temperatures. Thus, stress relaxation data at various temperatures can be modelled using only one adjustable parameter, σ^*. Further, σ^* can be expected to be nearly constant for similarly-prepared specimens. Thus, eq. (4) provides a powerful constraint on the application of the constitutive equations based on state variables to the present problem.

It is known that dislocation motion occurs in these films, although yield stresses are considerably higher than in bulk specimens.[1] Figure 2 shows typical load-relaxation results for bulk lead samples at room temperature; σ^* is on the order of 6.9 MPa (1000 psi). The single crystal data are well described by eq. (3); increasingly polycrystalline specimens show the effect of grain boundary sliding, significant even at room temperature in Pb. As will be demonstrated, grain boundary sliding should not affect the overall stress relaxation in these films, although it may be important in other ways.

3. Stress-Relaxation Data

In a series of studies,[3,4,8-10] Murakami and co-workers at the IBM Watson Research Center have examined the stress relaxation behavior of thin lead films subjected to various thermal histories. Strain perpendicular to the film plane was measured using an X-ray diffraction technique, and the stress in the film plane calculated using a biaxial strain model.[8] It was found that, for films of moderate thickness subjected to a change in temperature, a substantial fraction of the elastic strain expected on the basis of differential thermal expansion had relaxed either during or shortly (2-3 minutes) after the temperature ramp (primary relaxation). Continued observation at the final temperature showed further strain relaxation at a much slower rate (secondary relaxation).[8]

Although the primary relaxation was too rapid to follow in detail using the X-ray diffraction technique, the secondary process could be studied. This group has kindly provided to us experimental data, in the form of values for the change in lattice parameter as a function of time, for comparison with model calculations. Film preparation and experimental procedures have been described elsewhere.[3] The films in question were deposited onto oxidized (111)-oriented silicon substrates, were of 1μ thickness and 4.5μ average grain size, and were prepared to be strain free at room temperature. Stresses were therefore tensile for $T < T_{ambient}$ and compressive for the single set at 350 K. All the films exhibited a typical (111) fiber structure.

4. Comparison with Model Calculations

Stress values were calculated for the IBM data using the biaxial strain model and compared to a model calculation for stress relaxation by plastic flow. The procedure was to calculate $\dot{\sigma}$ from the current value of the stress by use of eqs. (3) and (2) and integrate the result stepwise in time. The modulus M in eq. 2 was taken as equal to Young's modulus. Ignoring the elastic anisotropy of Pb and the biaxial nature of the deformation introduces an error factor of order unity in σ[4]

but does not affect the kinetics or comparisons between results at different temperatures.

Best-fit values of σ^* and $\dot{\varepsilon}^*$ were determined at 240 K; at other temperatures, $\dot{\varepsilon}^*$ was calculated from the best-fit values of σ^* using eq. (4). Because of the uncertainty in the stress at times earlier than the first available data, a convenient value of the stress near σ^* was assumed for t=0. Since plasticity is highly nonlinear the effect of this procedure was a slight uncertainty in the time axis of the simulation, which was not significant given the nature of the data. Thus σ^* was the only adjustable parameter.

Experimental data and model calculations at these temperatures are shown in Figs. 3-5. Agreement for other temperatures at which data were available (260 and 220 K) was also excellent. The hardness parameter σ^* was found to be 83 MPa (12000 psi) within about 10% for different specimens; $\dot{\varepsilon}^*$ was $10^{-11} sec^{-1}$ at 240 K.

The agreement between theory and experiment at 350 K is particularly striking. For this temperature, stresses were compressive rather than tensile and the degree of stress relaxation was substantially higher. These data are presented in semi-logarithmic form in Fig. 6, along with the present model calculation. Estimates of Coble creep (due to grain boundary diffusion) and Nabarro-Herring creep (due to matrix diffusion) based on the thin-film analysis of Gibbs[11] are also shown. Not only is the relaxation rate incompatible with creep, but the kinetics are not exponential as would be expected.

We conclude that plastic flow is the dominant mechanism of stress relaxation in these films, at least for the secondary process studied here. In the remainder of this paper, we will discuss the structure of the stress system in thin films and its implications for stress relaxation and long-range materials transport. As will be seen, it is possible to develop a coherent picture of the primary stage of stress relaxation as well.

5. Thermal Stress System in Thin Films

Given the stress relaxation rates evident from Fig. 6, the apparent absence of grain-boundary diffusional creep in these films is somewhat surprising. On the other hand, Gibbs' expressions take account of neither the presence of a constraining substrate nor the biaxial nature of the stress system in these specimens. As we now show, consideration of these aspects changes the picture considerably.

Consider a single grain far from the film edge. Deformation of such a grain during a temperature change is controlled by two constraints: that the strain at the substrate interface be given by eq. 1 (condition of good adherence), and that the deformation be compatible with that of neighboring grains.

For a grain of finite thickness, the equilibrium deformation due to the interface constraint alone clearly involves decreasing biaxial strain with distance from the substrate. For compatibility with neighboring grains, additional strains must be introduced by stresses acting across grain boundaries. The instantaneous grain boundary stresses, then, will be determined by (but not directly proportional to) the difference between the actual elastic strain state of the grain and that which would result from the interface constraint acting alone.

For an isotropic material, grain boundaries are irrelevant to the elastic response and uniform biaxial strain is expected. Ideally, deformation would be supported entirely by film stresses and interfacial forces would be unimportant except near the film edges. Essentially, for a thin isotropic film the deformation in the interior due to the strain at the edges is compatible with the strain which would be imposed at the interface directly by adhesion. For anisotropic materials, elastic constants are not continuous across grain boundaries and the strain state of each grain will be more complex. We believe that this effect can be neglected, and in particular that a biaxial strain model based on uniform strain can give reasonable average values of plane stress based on average values of the normal strain from X-ray diffraction. In the following discussion, therefore, we ignore

anisotropy. For convenience, we take grain boundaries as being normal to the film plane, and illustrate the argument using a state of compressive strain.

Figure 7 depicts an idealized single grain, with reference atomic planes indicated to illustrate the state of elastic strain, under various conditions of deformation. In (a) the grain is unstrained (at the reference temperature), and in (b) a temperature change has imposed a uniform strain with grain boundary normal stresses as shown.

Given a suitable sink, grain boundary diffusion will occur. Since the strain is biaxial, all grain boundaries are more or less equally stressed and transport must be to the film surface or to strain-free extrusions (hillocks) on the surface. For many film materials of engineering interest, including Pb, a native oxide layer inhibits surface diffusion. We assume, therefore, that any transport is primarily in the film (presumably to distant hillocks); we neglect for the moment relaxation processes in the matrix.

Transport of material from the grain boundary allows the grain to relax outward, relieving the grain boundary and matrix stresses. Since the displacements in the film plane representing the difference between the initial strain state and that dictated by the interface constraint are larger near the surface, the transport is nonuniform. Schematically, the grain will relax through configurations similar to Fig. 7(c) to the limiting case of Fig. 7(d), where all grain boundary stresses have been relaxed, there is no further driving force for grain boundary diffusion, and the remaining strain is due solely to the interface constraint.

To illustrate the nature of the material transport, the final state is shown unstrained in Fig. 7(e). Note that returning the film to the reference temperature would require the existence of tensile grain boundary and matrix stresses even though no matrix processes have taken place.

· The reason for the absence of grain boundary diffusional creep is now clear. For polycrystalline creep to operate in the manner analyzed by Gibbs to operate, the grains must be free to move relative to each other. This is necessary to maintain grain boundary stresses and provide material for continuing diffusion, but it is not possible if the film adheres to the substrate on a microscopic scale. In these films, once diffusion has proceeded to the state of Fig. 7(d) (relaxing only a small portion of the average plane stress) further relaxation requires the operation of matrix processes (bulk diffusion or plastic flow).

Before turning to the role of matrix processes, it is necessary to observe that the sequence described above depends critically on the absence of rapid relaxation at the interface (condition of good adherence). In fact, it is the adherence between film and substrate on this microscopic scale which leads to the gradient normal to the film plane in the biaxial strain shown in Figs. 7(c) and 7(d).

Using an X-ray line broadening technique, Murakami has observed this gradient in lead films.[3] It is found that the gradient is established during the initial stress relaxation and persists during longer anneals. This is the result expected if grain boundary diffusion initially operates more rapidly than bulk relaxation processes.

This result indicates that no interfacial process, involving sliding or other rearrangement between film and substrate, plays a significant role compared to bulk processes. Since it is difficult to imagine that substantial diffusion in the interface can occur if this is so, we conclude that the film-substrate interface is not a good diffusion path.

It has been argued that, grain boundary transport being too slow, interfacial diffusion is necessary to explain hillock growth.[12] This conclusion, however, depends on the assumption that diffusional creep processes compete with hillock growth to relax stress. As we have seen, the usual polycrystalline diffusional creep processes involving grain rearrangement cannot operate in the presence of good sub-

6. Primary Deformation During a Cyclic Temperature Change

The complex nature of the stress system, and of the interaction between plastic deformation and grain boundary diffusion, in thin films make development of a quantitative model of stress relaxation difficult. It is possible that a finite-element analysis of the stress system within a typical grain, carried out for a suitable thermal history and using the plasticity model employed here, can be integrated into the overall picture of mass transport in thin films. This problem is presently being explored.

By taking account of the foregoing analysis and of experimental data from the literature, however, it is possible to describe the qualitative nature of stress relaxation. It is instructive to discuss the behavior for a temperature cycle between room temperature and 4.2 K -- a thermal history of obvious interest for application of thin film devices. We now discuss the primary relaxation process which operates during the temperature change.

Consider a thin film adhering to a substrate of lower coefficient of thermal expansion, such as the Pb films on silicon discussed earlier. We assume the presence of sources and sinks of material for grain boundary diffusion, such as hillocks resulting from previous temperature cycles.

As the temperature is lowered, tensile strain is continuously introduced into the film. Grain boundary stresses which result will be relaxed by diffusion; the equilibrium stress will be determined by the cooling rate, the length of the diffusion path, and the grain boundary diffusion coefficient at the instantaneous temperature. A gradient in the plane stress normal to the film will be quickly established.

The matrix stresses will increase until they approach the yield stress, at which point plastic flow will begin. Thereafter, the temperature dependence of the plastic strain rate, reflected in eq. (4), will be overwhelmed by the highly nonlinear nature of the stress dependence of plastic flow (eq. (3)). We expect the temperature-normalized matrix stress σ/G to remain more or less constant during further cooling.

Since grain boundary diffusivity decreases with temperature, however, there will come a point at which it is negligible compared to plastic flow. We then have the case illustrated in Fig. 8(a); plastic deformation continues, but intergranular compatibility localizes the material transport. This condition continues until cooling ceases at cryogenic temperatures.

Upon reheating of the film, the strains introduced are compressive. Initially, this serves to remove the residual tensile stresses. (Because of the normal gradient in the plane stress, during heating the top of the film will go into compression before the region near the interface. This is not important to the present discussion.) As heating continues, the matrix stresses once again approach the yield stress and plastic flow occurs in compression.

At sufficiently high temperatures, grain boundary diffusion again becomes a factor. Transport of material to hillocks will relax the grain boundary stresses and further plastic deformation will occur under the conditions shown in Fig. 8(b). When the temperature is then stabilized at the original point, further isothermal relaxation will reduce the final elastic strains to quite small values.

The behavior described above is shown schematically in Fig. 9. In that figure, the matrix stress during reheating is shown as having a smaller magnitude than that during cooling. This is a manifestation of the well-known Bauschinger effect; a specimen deformed plastically in tension exhibits extra strain as a function of stress when subsequently compressed. In the present circumstance, this implies a reduction in the equilibrium stress during reheating. The essential features of Fig. 9 are evident in the exeprimental work of Murakami (compare Fig. 6 of his paper[4]).

The foregoing description exhibits an asymmetry between heating and cooling which explains net long-range material transport over a temperature cycle. As noted previously, plastic deformation under conditions of rapid grain boundary diffusivity will contribute to diffusive transport, while in the absence of such diffusion only local thickness changes result. During the thermal history of Fig. 9, more plastic

strain above the minimum temperature for grain boundary diffusion occurs in compression than in tension.

The following argument should make this clear. The elastic strain introduced into the film, in the absence of relaxation, upon a change of temperature is given by eq. (1). The plastic strain at anytime is given by the difference between the relaxation-free value and the actual value, which is known from the stress. On cooling from room temperature (T_0) to the minimum temperature for diffusion (T_1), the accumulated plastic strain is

$$\Delta\varepsilon_C = \int_{T_0}^{T_1} (\alpha_s - \alpha_f)dT - \frac{\sigma_{1C}}{M} \tag{5}$$

where σ_{1C} is the stress at T_1 during cooling and M is an appropriate modulus.

Upon reheating, the accumulated plastic strain above T_1 is of opposite sign and is given by

$$\Delta\varepsilon_H = \int_{T_1}^{T_0} (\alpha_s - \alpha_f)dT - \frac{\sigma_{1H}}{M} \; ; \tag{6}$$

we neglect the residual elastic strain at room temperature as it is small.[4] The net plastic strain is then

$$\Delta\varepsilon_{net} = \Delta\varepsilon_C + \Delta\varepsilon_H = -(\frac{\sigma_{1C} + \sigma_{1H}}{M}) \tag{7}$$

Since $|\sigma_{1C}| > |\sigma_{1H}|$, $\Delta\varepsilon_{net}$ has the sign of a compressive strain. Thus hillock volume is expected to increase over the course of a temperature cycle.

This is in good accord with experiment. Hillocks are observed to grow and shrink with changes in temperature, depending on the tensile or compressive nature of the plane stress.[12,13] Repeated cycling between room and cryogenic temperatures, however, promotes continued hillock growth.[1,10]

7. Hillock Nucleation and Grain Boundary Sliding

Because the stress levels achieved in these films are generally too small to allow homogeneous nucleation of hillocks,[13] it is generally assumed that some stress concentration process must take place. Grain boundary sliding has been invoked as

a likely mechanism, and as previously mentioned, such sliding strongly affects the deformation rate of bulk lead specimens at room temperature.[7] On the other hand, the model calculations based on matrix plasticity at various temperatures suggest that this is not true in these films.

It seems clear that the role of grain boundary sliding in stress relaxation is severely limited by the interface constraint. Substantial deformation by sliding in the film plane would require relative motion of the film grains, and as we have seen good adherence to the substrate will prevent this. In any event, the shear stresses necessary to drive this type of deformation are nearly zero in the biaxial strain field imposed by the temperature change.[14]

On the other hand, for grain boundaries not normal to the film plane there will be a shear component promoting sliding out of the plane. The displacements possible by this mechanism are limited by elastic accommodation between the fixed interface and the film surface, and should be small. Thus sliding is unlikely to affect the overall strain relaxation.

It is clear from the geometry of the problem, however, that plastic flow can provide additional accommodation for this sliding motion. The basic grain boundary sliding rate, then, will be a function of boundary orientation, matrix accommodation processes, and the intrinsic viscosity of the boundary. Additional retardation, and stress concentration, will result from impurity particles and grain boundary ledges.

It is the stress concentration at ledges which may lead to hillock nucleation. Figure 10 depicts an oblique grain boundary with a ledge making a shallow angle with the film surface. Sliding in the sense shown will increase stress at the ledge; transport out of the ledge by grain boundary diffusion will relax it. The peak value of the resulting stress will occur only after some incubation period and will depend on the relative rates involved; if the stress concentration is sufficient, the tip of the ledge can extrude to form a hillock nucleus on the surface.

Once formed, the hillock can quickly relax residual plane stresses by sliding at the hillock-film boundary. This boundary is free of normal stresses and will be a sink for grain boundary diffusion; the hillock can increase in height by accretion at the bottom surface, a process which is suggested by the fact that the top surface structure of hillocks is generally stable during growth or shrinkage.[12,13] Lateral growth of the hillock will be resisted by the surface oxide layer, although local weakness of the layer, and slip processes which disturb the film surface, should facilitate such growth. In fact, lateral growth of hillocks is found to be erratic.[13]

If the sliding rate is sufficiently slow, grain boundary diffusion will relax the stress concentration and nucleation will not occur. It has been observed that a $300\overset{o}{A}$ layer of Ni at the film-substrate interface prevents the formation of hillocks in 2μ Sn films.[12] This was assumed to result from a reduction in interfacial diffusion, although retardation of grain boundary sliding was mentioned as an alternative explanation. Since diffusion at the film-substrate interface does not appear to play a role in hillock growth, we prefer the second explanation, which effect is probably due to formation of Ni or Ni-Sn compound particles in the grain boundary.

Since the critical balance between sliding rate and diffusive relaxation will depend on the specific geometry of the boundary, some sites will be more favorable for hillock nucleation than others. It would be interesting to explore the effect of intergranular impurity particles, heating rate, and temperature on hillock density in a controlled manner.

8. Conclusions

Model calculations have been carried out for the contribution of matrix plasticity to the slow secondary relaxation of thermally-stressed thin films, based on a state variable approach. The results suggest that plastic flow is the dominant mechanism and is well represented by the constitutive equations based on state variables even at the high yield stresses involved.

Consideration of the nature of the substrate constraint (condition of good adherence) suggests an explanation for the evident absence of diffusional creep. The interaction between plastic flow and grain boundary diffusion seems capable of explaining the observed nature of the rapid primary relaxation stage as well.

The picture that has been developed predicts continued accumulation of material in surface hillocks during cycles between room and cryogenic temperatures. Diffusion in the film-substrate interface does not appear to play a role in hillock growth. Hillock nucleation appears to be the result of concentration of stress at grain boundary ledges near the surface by sliding.

The complex nature of the stress system in these films makes quantitative predictions difficult, except in the relatively simple case of the secondary relaxation. It is hoped that further work based on the framework presented here will allow the calculation of the increase in hillock volume, and the extent of other damage processes leading to device failure, during various thermal histories.

Acknowledgements

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Figure Captions

1. Simplified picture of a portion of a thin polycrystalline film adhering to a substrate. Under a temperature change, a biaxial strain is imposed in the plane of the film. When the resulting stress is compressive as shown, one result of relaxation is the formation of hillocks on the film surface.

2. Results of load relaxation tests on bulk Pb specimens at room temperature (from Ref. 7).

3. Measured and calculated values of the tensile plane stress during an isothermal anneal at 220 K.

4. Measured and calculated values of the tensile plane stress during an isothermal anneal at 240 K.

5. Measured and calculated values of the compressive plane stress during an isothermal anneal at 350 K.

6. Measured values of the compressive plane stress during an isothermal anneal at 350 K, compared with the plasticity model, grain-boundary diffusional (Coble) creep, and bulk diffusional (Nabarro-Herring) creep.

7. Single film grain on a substrate, with reference atomic planes indicated to illustrate the state of elastic strain, under various conditions of deformation. Arrows illustrate grain boundary normal stresses.

    (a) Undeformed.

    (b) After an instantaneous temperature change.

    (c) During relaxation due to grain boundary diffusion.

    (d) Limiting case of (c).

    (e) Unstrained grain after diffusion, to illustrate nature of material transport.

8. Nature of material transport due to plastic flow.

    (a) Local transport due to multiple slip in the presence of grain boundary stresses.

    (b) Transport of material into the grain boundaries, by slip and by relaxation

of equilibrium elastic strain, in the absence of grain boundary stresses.

9. Plane stress as a function of temperature during cooling, reheating and subsequent room temperature anneal.

10. Shear stress and resultant sliding at an oblique grain boundary, showing a grain boundary ledge configuration at which stress concentration and hillock nucleation may occur.
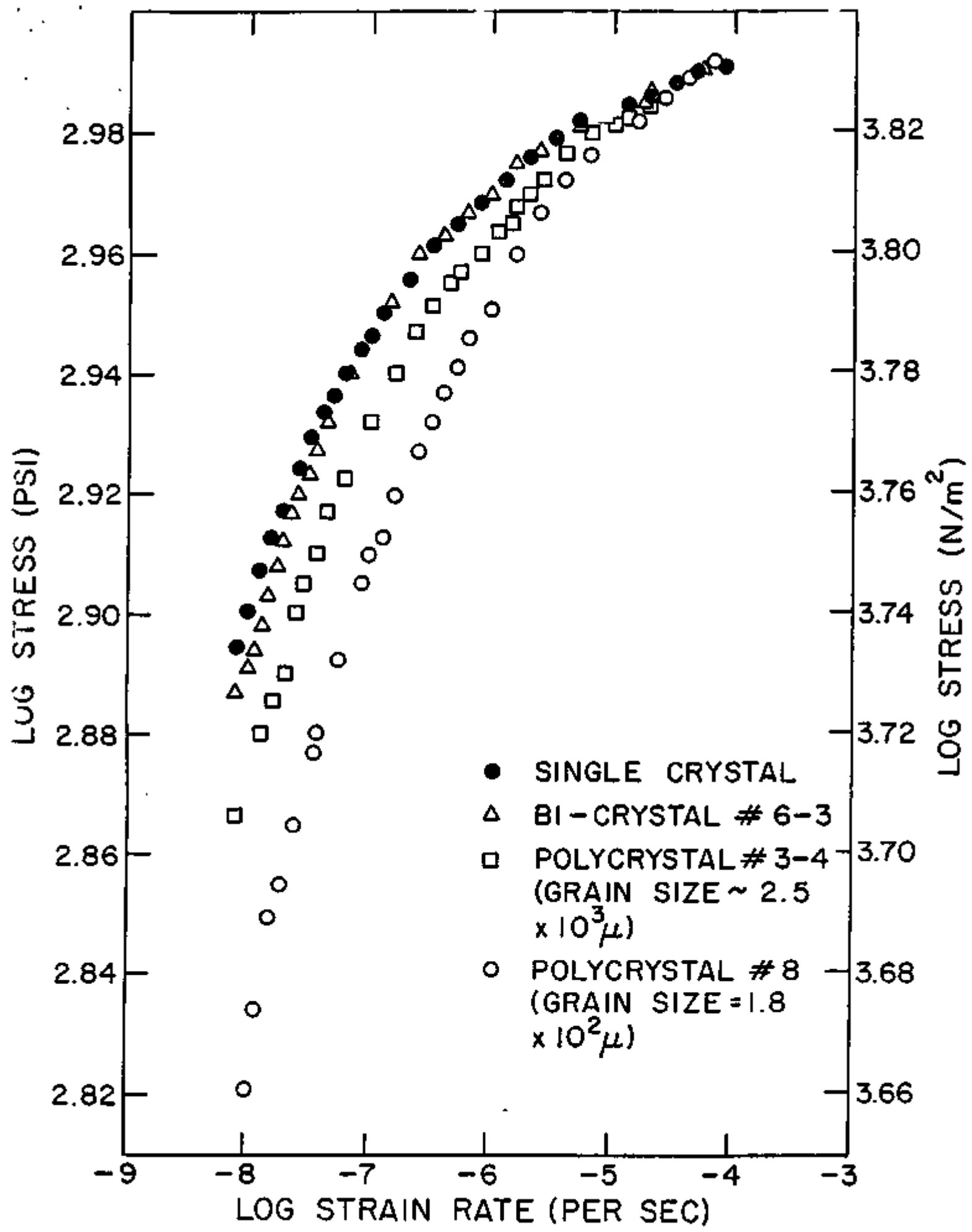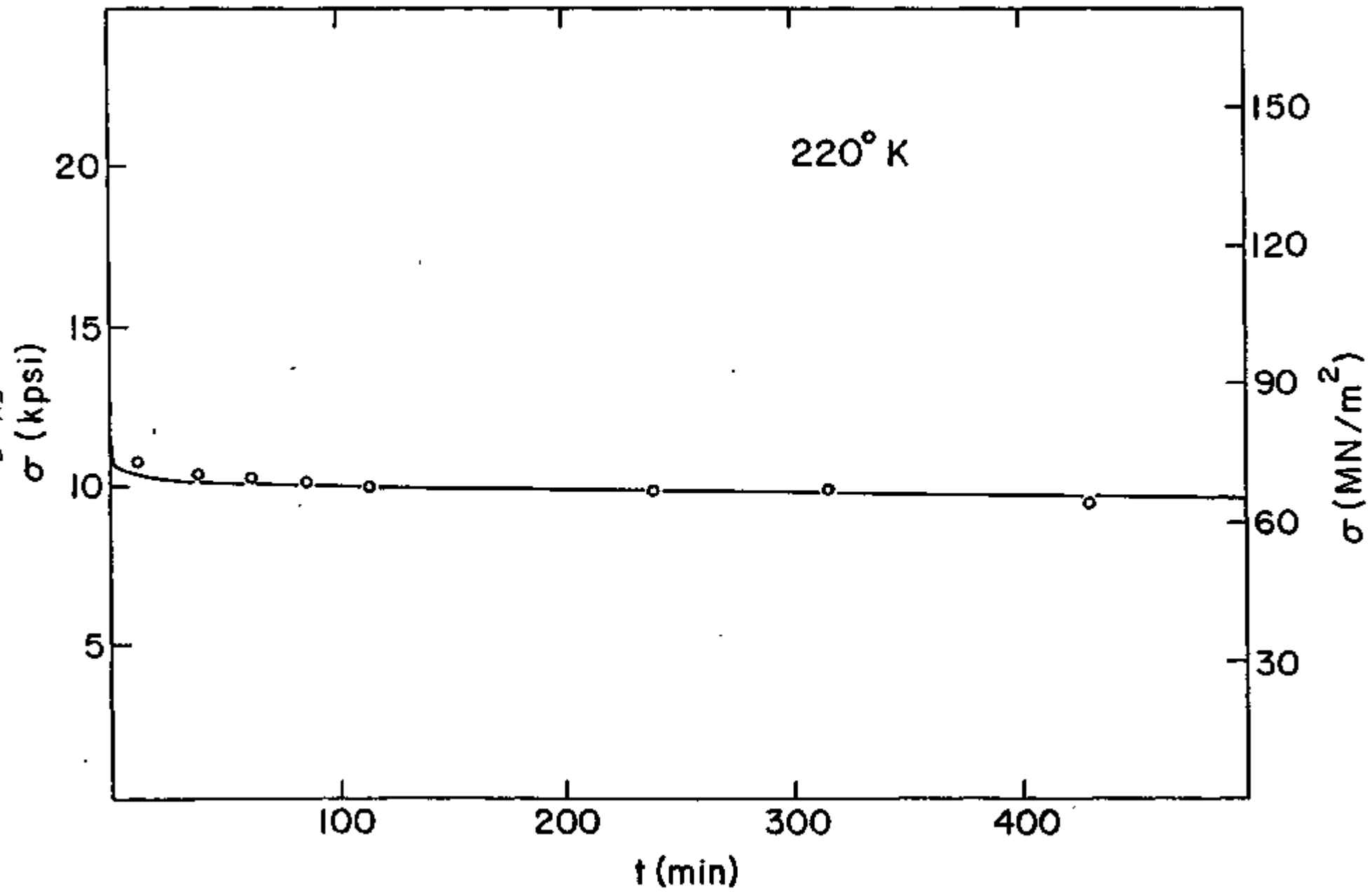
σ

σ

hillock

substrate

FIG. 1

FIG. 2

FIG. 3

220° K

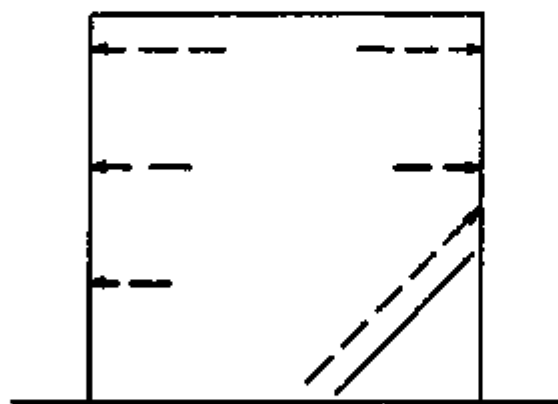FIG. 4

240°K

350°K

FIG. 6

(a)

(b)

(c)

(d)

(e)

FIG 7

(a)

(b)

FIG. 8

FIG 9

$$\sigma_s = \sigma \; Sin \; \theta \; Cos \; \theta$$

FIG 10