

Progress Report

December 1, 1973 - November 30, 1974

Contract No. AT(11-1)2172

GRAIN BOUNDARY SLIDING AND STRUCTURE

by

Che-Yu Li
Professor

Department of Materials Science and Engineering
Cornell University
Ithaca, New York 14850

September 1, 1974

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

INTRODUCTION

The work undertaken in this project is a part of the total effort of this laboratory to develop concepts and methods for the characterization of mechanical properties of polycrystalline solids at elevated temperatures. Grain boundary sliding is emphasized because it is an important deformation process in the temperature range of interest. The approach based on the concept of plastic equation of state is adopted in this work. This approach has been proven to be successful even though considerable effort is made for its development. The experimental results obtained so far have demonstrated the potentials in the application of the approach of plastic equation of state in mechanical testing and in data correlation and extrapolation. The plastic equation of state provides also a rigorous and precise description of the deformation behavior of the grain matrix. Deviations from the grain matrix behavior can therefore be measured easily and can be used as a quantitative basis for the investigation of the contribution of grain boundary sliding and the mechanical properties of the grain boundary.

During the past fiscal year the principal investigator and his co-workers have co-authored a review paper with Dr. E. W. Hart of G. E. Research and Development Center. The title of this paper is "Phenomenological Theory: A Guide to Constitutive Relations and Fundamental Deformation Properties", which will be a chapter in the forthcoming book "Constitutive Equations in Plasticity" (Ed. A. Argon, MIT Press). Much of the theoretical and experimental results on plastic equation of state

obtained at G. E. Research and Development Center and in this laboratory are included in this paper. The constitutive relations for plastic deformation developed are of the analytical form such that they can be readily adopted for mechanical analysis. Mechanics groups at Cornell University, Ohio State University and at G. E. Research and Development Center have in fact shown interest in their applications.

In-reactor creep is a topic which has been given increasing attention recently. Since the behavior of plastic equation of state has been found to be exhibited by a variety of materials covering a wide range of deformation conditions, it is natural to suspect that the same concept may be extended to include in-reactor mechanical behaviors. Some of the current thinking of the principal investigator on this subject will be described in the renewal proposal for the next fiscal year. Arguments will be made to propose that grain boundary sliding and irradiation enhanced grain boundary migration could play an important role in in-reactor creep. At the present Argonne National Laboratory is engaged in post-irradiation and in-reactor load relaxation experiments and is also planning load relaxation experiments under the influence of cyclotron irradiation. In their work the approach of plastic equation of state is also adopted. Some of the results obtained in this laboratory especially those on austenitic stainless steels will be useful in supporting the type of activities mentioned above.

The highlights of the progress made since the last report will be described in the following sections. During the past fiscal year efforts are made in several areas including work

hardening properties, creep analysis and grain boundary sliding in type 316 stainless steel.

WORK HARDENING AND PLASTIC EQUATION OF STATE

It is reported in the last progress report that the work hardening parameter $\gamma \left(\frac{\partial \ln \sigma}{\partial \epsilon_p} \right)_{\dot{\epsilon}_p, T}$ of type 316 stainless steel, niobium and 1100 aluminum alloys is found experimentally to depend only on the current values of stress and plastic strain rate and is independent of deformation history. σ , ϵ_p , $\dot{\epsilon}_p$ and T are stress, plastic strain, plastic strain rate and temperature respectively. For the interest of creep analysis and data correlation and extrapolation a new work hardening parameter Γ which measures the change in the value of hardness parameter with plastic strain increment is defined.

$$\Gamma = \frac{\partial \ln \sigma^*}{\partial \epsilon_p} \Big|_{\dot{\epsilon}_p, T} = \frac{\mu}{\mu - \nu} \gamma$$

where σ^* is the hardness parameter, μ is the slope of the translation path for the constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves and ν is the slope of the constant hardness curves. The constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves are measured in the load relaxation experiments. Since these materials exhibit the behavior of plastic equation of state, based on the results of the load relaxation experiments, the parameter Γ can be specified by any two of the three variables, stress, plastic strain rate and the hardness parameter. For the convenience of creep and mechanical analysis, σ and σ^* are used in correlating the experimental data. All the work hardening data obtained show that the work hardening

parameter Γ is a power function of σ and σ^* . The data of 1100 aluminum alloy are shown in Fig. 1 in the form of constant Γ lines in a plot of $\log \sigma^*$ vs $\log \sigma^*/\sigma$. These data are obtained in a temperature range from room temperature to over one-half of the absolute melting temperature and at a wide range of plastic strain rates. The constant Γ lines in Fig. 1, suggest the following relation,

$$\Gamma = C \sigma^\alpha / \sigma^{*\beta} \quad (1)$$

where C , α , β are constants. The correlation shown above can be used as a strong evidence for the applicability of the concept of plastic equation of state. The existing theories on work hardening which do not include plastic strain rate or the hardness parameter as an important variable, are found to be inadequate for the present results. Physically the correlation for the parameter Γ can be considered as a law for absolute work hardening because Γ measures the changes in hardness with plastic strain. The theoretical significance of this work hardening law remains to be examined but its analytical form is adequate for creep calculations which will be described next.

CREEP ANALYSIS

From the viewpoint of plastic equation of state, an arbitrary deformation path at a given temperature is represented by

$$\gamma d \epsilon_p = d \ln \sigma - \nu d \ln \dot{\epsilon}_p \quad (2)$$

where $\gamma = \frac{\partial \ln \sigma}{\partial \epsilon_p} \big|_{\dot{\epsilon}_p, T}$ and $v = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}_p} \big|_{\epsilon_p, T}$ and are a work hardening parameter and a strain rate sensitivity parameter respectively. As discussed in the previous section these parameters depend only on the current values of stress and the hardness parameter, and their functional dependence has simple analytical form. At high homologous temperatures the constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves obtained from the load relaxation experiments for grain matrix deformation can be represented by

$$\ln \left(\frac{\sigma^*}{\sigma} \right) = \left(\frac{\dot{\epsilon}_p^*}{\dot{\epsilon}_p} \right)^\lambda \quad (3)$$

where λ is a materials constant which describes the shape of the constant hardness curve and the hardness parameter σ^* is related to $\dot{\epsilon}_p^*$ through the translation path of these curves ($\ln \sigma^* = \mu \ln \dot{\epsilon}_p^* + \ln D$ with D a constant). From Equation (3) the strain rate sensitivity parameter v is

$$v = \lambda \ln \left(\frac{\sigma^*}{\sigma} \right) \quad (4)$$

Knowing the parameters γ and v , Equation (2) can be integrated to describe an arbitrary path of interest. The integration has been performed for constant load creep. The results of the creep calculation are compared with experimentally determined creep data in Fig. 2. In this figure the solid lines are calculated curves and the data points are measured values. It is seen that the agreement is within experimental scatter of typical creep experiments.

The exercise described here demonstrated the following points:

1. It shows the capabilities of the approach of plastic equation of state in using history independent parameters to describe plastic deformation which is path dependent.
2. The data and the calculated results of the cold worked material suggest strongly the absence of recovery during creep and load relaxation at temperatures up to one-half of the absolute melting temperature. This is a point of controversy in current thinking on creep theories. Other experimental evidence exists from the viewpoint of plastic equation of state suggesting also the absence of recovery in the same temperature range.
3. Since load relaxation and work hardening measurements are less time consuming than creep experiments, it is felt that the approach of plastic equation of state will be potentially useful in mechanical testing and in data correlation and extrapolation.

GRAIN BOUNDARY SLIDING IN TYPE 316

STAINLESS STEEL

The principal investigator has been interested in experimental and theoretical work for the development of fuel element cladding failure criteria for liquid metal cooled fast breeder reactor applications since 1969. The importance of grain boundary sliding and associated intergranular failure has long been recognized. The lack of quantitative information on these processes has prevented much of the desired progress in the opinion of the principal investigator. This inadequacy has been one of the strong motivations for the principal investigator to undertake the present project.

With the development of high temperature capabilities in this laboratory it is possible to obtain load relaxation data at temperatures up to 650°C. During the past fiscal year these capabilities are utilized to examine the mechanical behavior of type 316 stainless steel. The results obtained are revealing. Together with the development in the approach of plastic equation of state, the principal investigator feels that he is in a better position to accomplish some of the objectives outlined in the original proposal. The data on type 316 stainless steel also provides useful insight for in-reactor creep which will be discussed in the renewal proposal.

Good temperature control is essential in load relaxation experiments especially in the low strain rate range. At the present the capabilities in this laboratory allow measurements of plastic strain rate in the 10^{-9} sec⁻¹ range in a run typically lasting 4-5 days. To obtain similar data in creep experiments will be much more time consuming. It will be seen later that measurements in the low strain rate range are important also in identifying the contribution of grain boundary sliding.

The phenomenological models for the interplay between grain boundary sliding and grain matrix deformation proposed by Hart have been useful in providing useful insight for the interpretation of creep-rupture and low cycle fatigue data. Hart's idea can be summarized as follows. At low homologous temperatures and/or high strain rates the deformation of a polycrystalline solid is controlled by grain matrix processes. As the temperature increases and/or the strain rate decreases the contribution of grain boundary sliding will become important. In this region

the $\log \sigma - \log \dot{\epsilon}_p$ curves measured in load relaxation experiments will deviate from the corresponding grain matrix curve in the direction such that the slope of the measured curve will be higher. If the temperature increases further and/or the strain rate decreases further, the grain boundary will offer little resistance to deformation, the deformation of the specimen will be controlled again by grain matrix processes, in some cases resulting in a S-shaped $\log \sigma - \log \dot{\epsilon}_p$ curve. From the viewpoint of plastic equation of state based on Hart's model, the introduction of grain boundary sliding will occur at higher strain rates with increasing values of the hardness parameter. Since intergranular failure in creep rupture is associated with grain boundary sliding, the ideas described above are consistent qualitatively with the results of creep-rupture and low cycle fatigue tests on stainless steels and other alloys. For example as temperature increases the failure mode in creep rupture and low cycle fatigue tests changes from transgranular to intergranular. With increasing amount of cold work, the failure mode will also change from transgranular to intergranular at a given temperature. It is encouraging that the present results obtained from load relaxation experiments on type 316 stainless steel show the entire range of behavior described above and can be used as a quantitative basis for the evaluation of the contribution of grain boundary sliding.

Figure 3 shows experimental data on type 316 austenitic stainless steel as a function of temperature. For the 550°C curve the high strain rate region where the slope is very small is identified as controlled by grain matrix deformation. As the

strain rate is lowered the slope of the curve increases indicating that the contribution of grain boundary sliding becomes increasingly important. As the temperature increases the data in Fig. 3 show the introduction of grain boundary sliding at higher strain rates. The 650°C curve is of particular interest because it is S-shaped. This shape results from the fact that the grain matrix curve has a rather small slope when the strain rate is decreased into the region where grain matrix deformation is controlling again, the measured curve will show a decrease in slope. This observation would not be possible without the present capabilities in this laboratory to measure strain rates in the 10^{-9} sec⁻¹ range. The temperature dependence of the interplay between grain matrix deformation and grain boundary sliding shown in Fig. 3 is consistent with that suggested previously. Figure 4 shows experimental data obtained at 650°C on type 316 austenitic stainless steel as a function of grain matrix hardness. The $\log \sigma - \log \dot{\epsilon}_p$ curves in this figure demonstrate the introduction of grain boundary sliding at higher strain rates as the grain matrix hardness is increased.

At the present the experimental data on type 316 stainless steel are being analyzed in an attempt to obtain quantitative information on grain boundary sliding. These data clearly demonstrate that part of the creep data of type 316 stainless steel for LMFBR applications are in stress-strain rate regions where grain boundary sliding is important. The principal investigator is not aware of the consideration of grain boundary sliding contribution in creep data analysis by investigators in LMFBR program. The present data suggest also that the creep analysis

given in the previous section requires further modification to include grain boundary sliding.

SUMMARY

During the past fiscal year the following accomplishments have been made in the present program

1. A generalized work hardening relation has been established based on the approach of plastic equation of state.
2. Using the same approach capabilities are developed to predict creep behavior of polycrystalline solids.
3. Experimental data on grain boundary sliding in type 316 stainless steel are obtained.

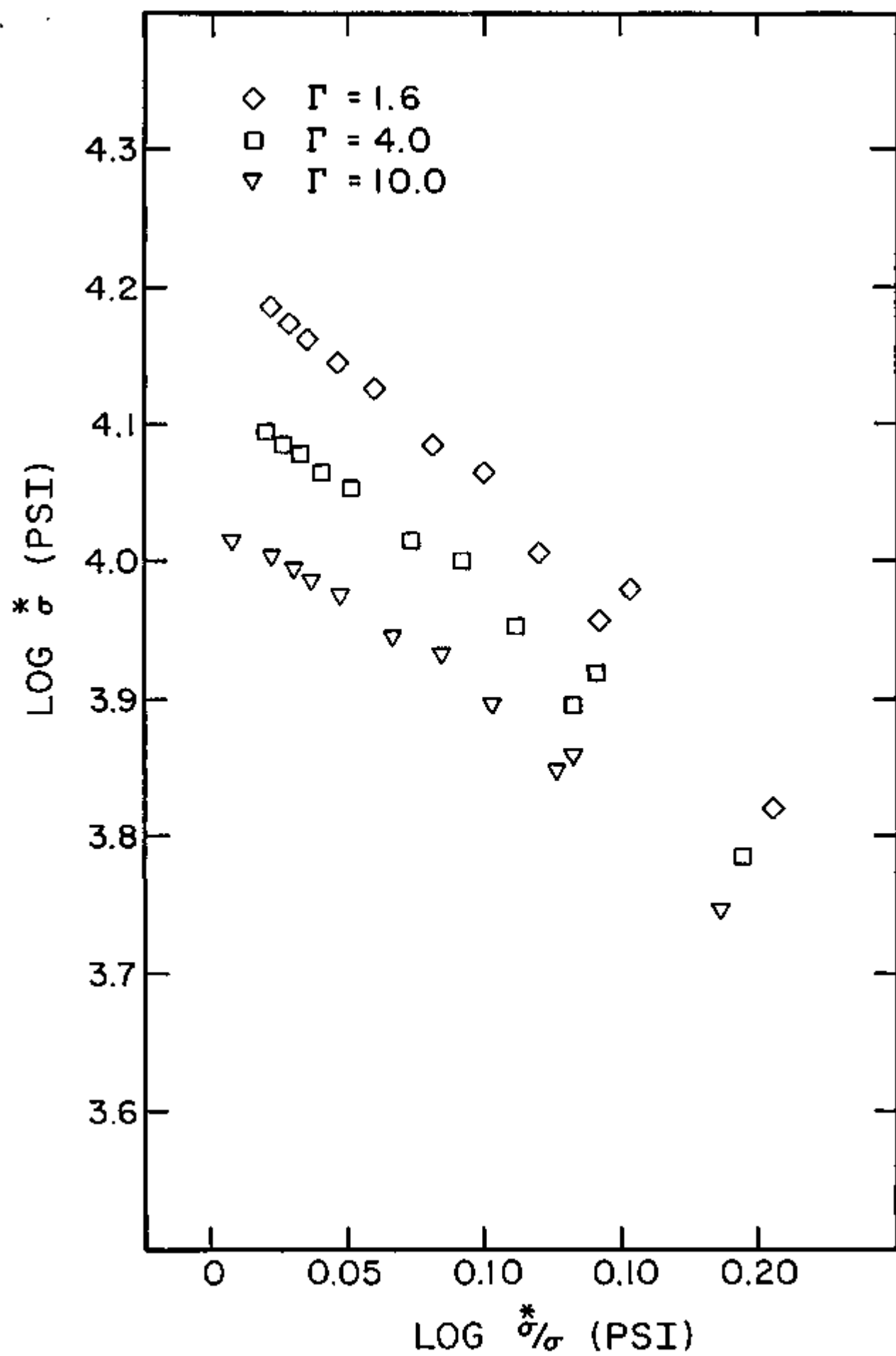


Figure 1

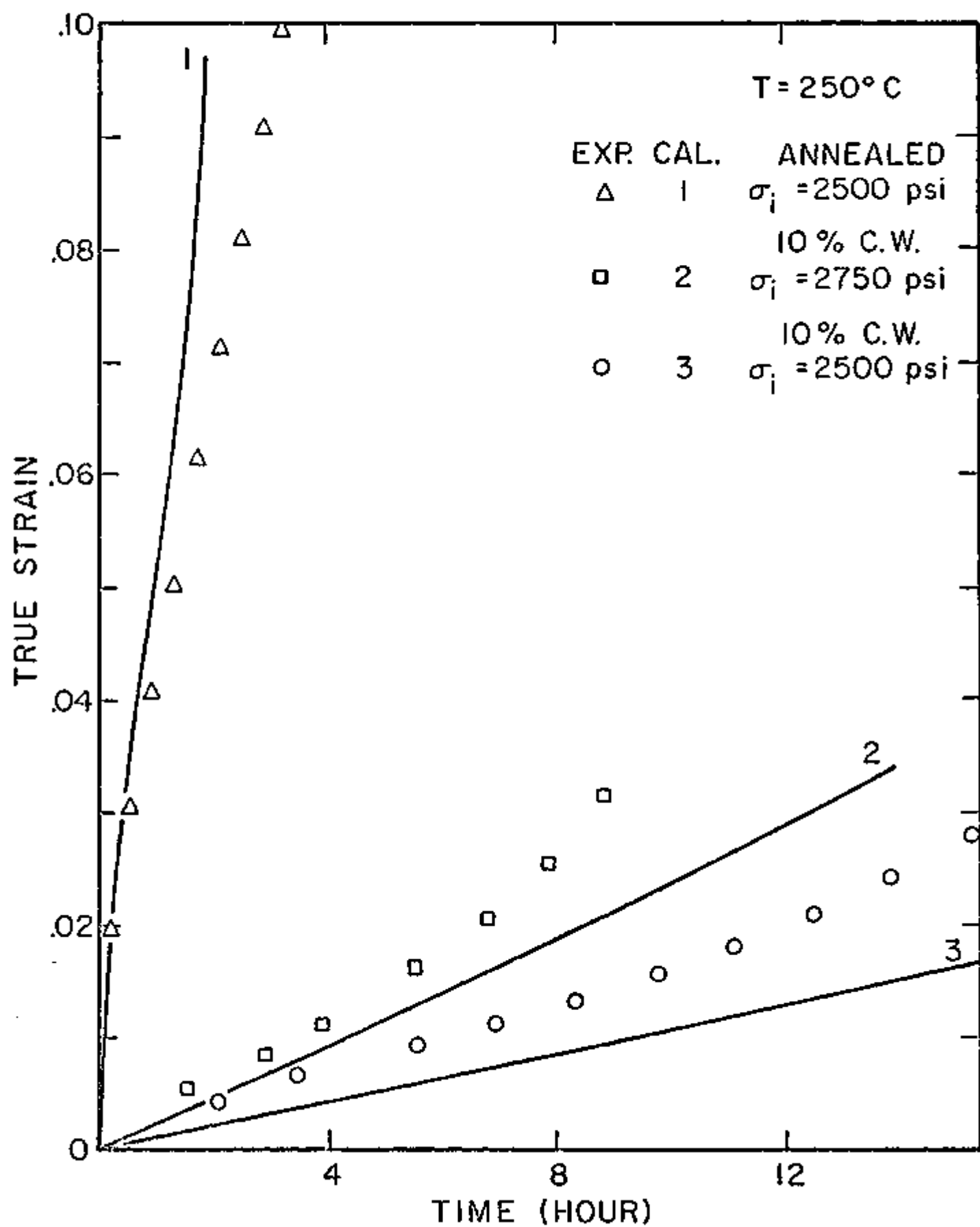


Figure 2

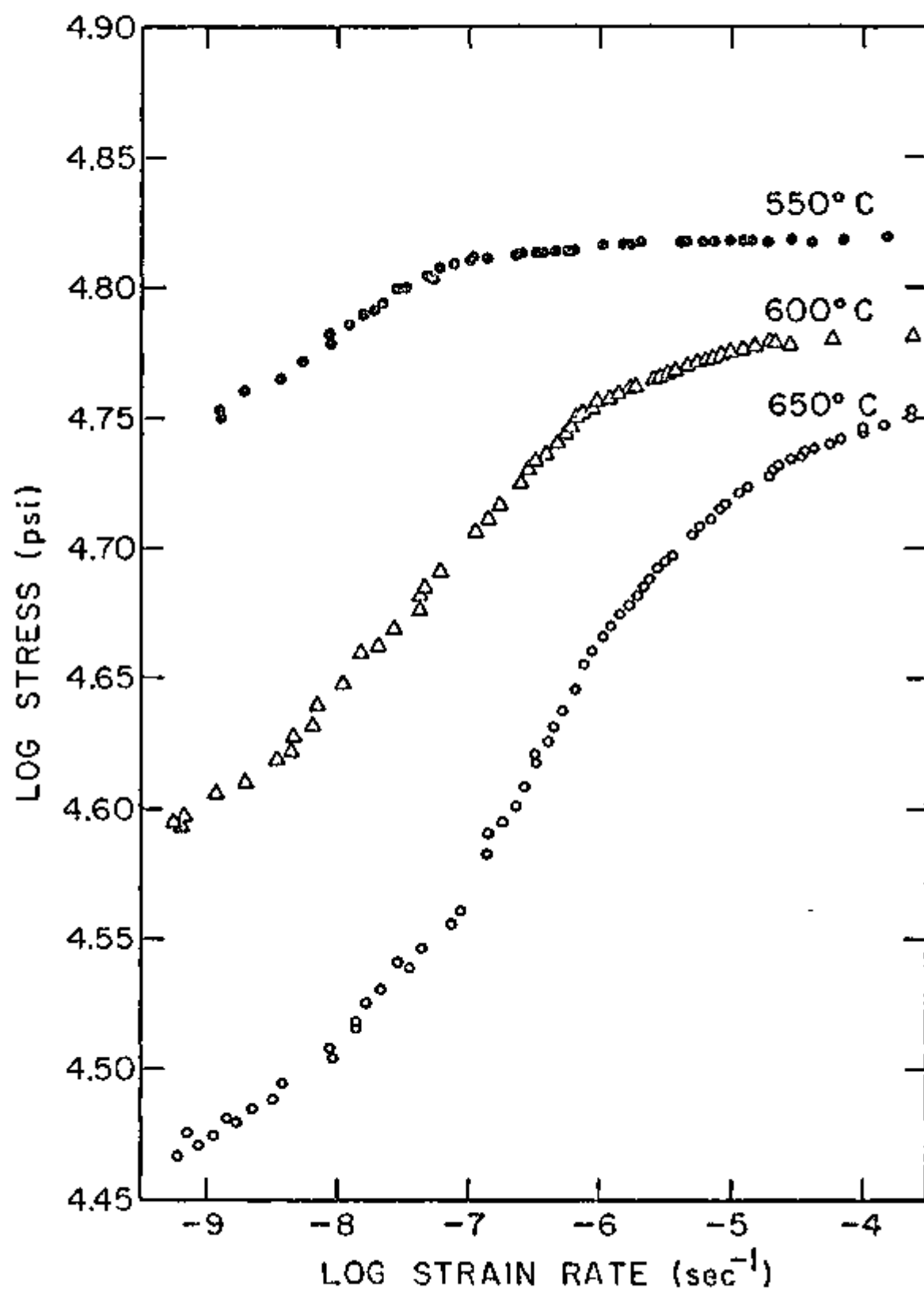


Figure 3

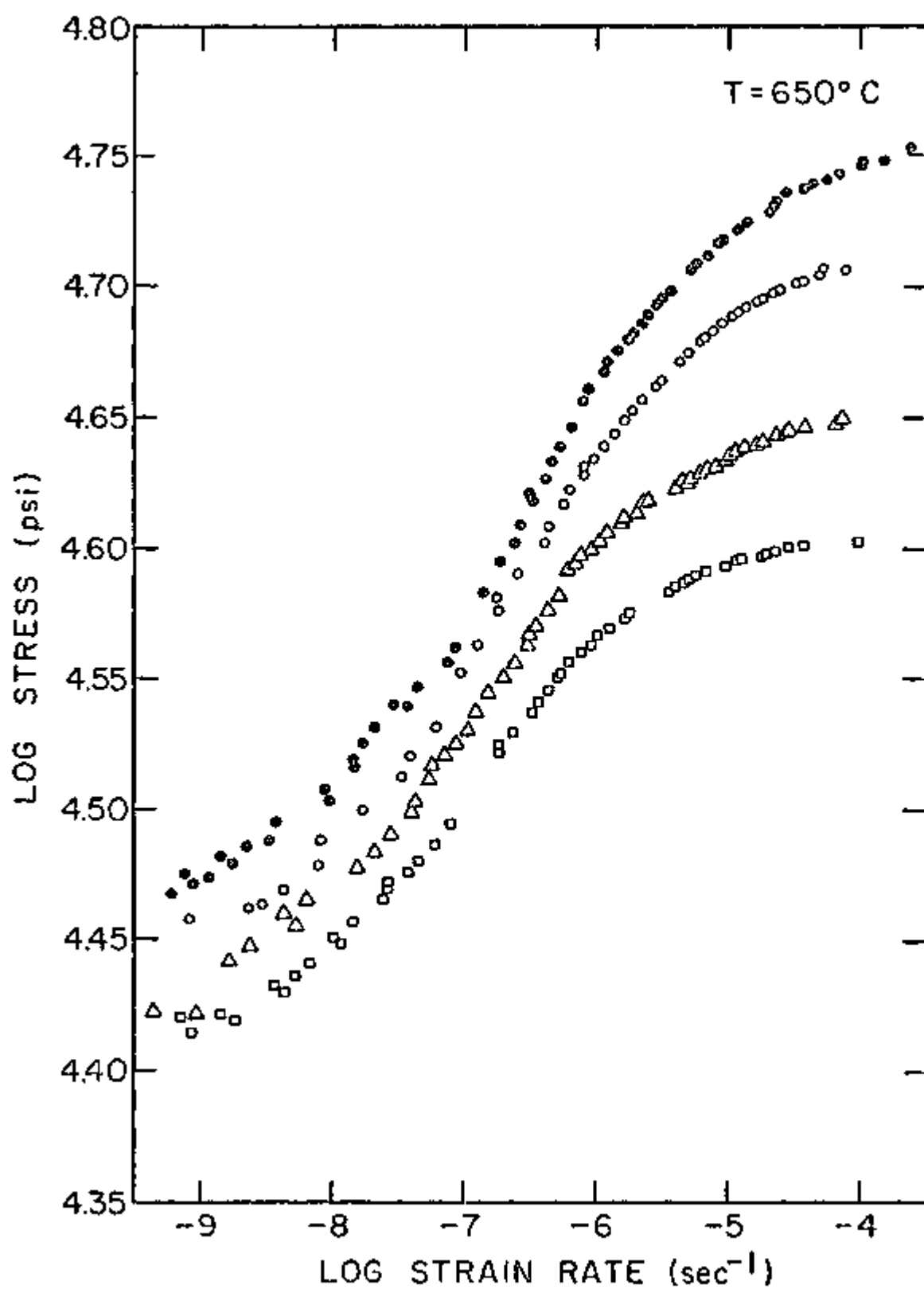


Figure 4

UNIVERSITY-TYPE CONTRACTOR'S RECOMMENDATION FOR
DISPOSITION OF SCIENTIFIC AND TECHNICAL DOCUMENT

(See Instructions on Reverse Side)

1 AEC REPORT NO COO-2172-7	2 TITLE Progress Report Dec. 1, 1973 - Nov. 30, 1974 Grain Boundary Sliding and Structure
-------------------------------	---

3 TYPE OF DOCUMENT (Check one)

☒ a Scientific and technical report

☐ b Conference paper

Title of conference _____

Date of conference _____

Exact location of conference _____

Sponsoring organization _____

☐ c JOURNAL ARTICLE

Submitted to _____

☐ d Other (Specify) _____

4 RECOMMENDED ANNOUNCEMENT AND DISTRIBUTION (Check one)

☒ a AEC's normal announcement and distribution procedures may be followed

☐ b Make available only within AEC and to AEC contractors and other U.S. Government agencies and their contractors

5 REASON FOR RECOMMENDED RESTRICTIONS

6 SUBMITTED BY NAME AND POSITION (Please print or type)

Che-Yu Li, Professor

Organization

Department of Materials Science and Engineering
Cornell University

Signature



Date

August 30, 1974

FOR AEC USE ONLY

7 AEC CONTRACT ADMINISTRATOR'S COMMENTS, IF ANY, ON ABOVE ANNOUNCEMENT AND DISTRIBUTION RECOMMENDATION

8 PATENT CLEARANCE

☐ a AEC patent clearance has been granted by responsible AEC patent group

☐ b Report has been sent to responsible AEC patent group for clearance

☐ c Patent clearance not required