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GRAIN BOUNDARY SLIDING AND STRUCTURE

by

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INTRODUCTION

The research undertaken in this program is a part of the combined effort of this laboratory to develop concepts and methods for the characterization of mechanical properties of crystalline solids. An important objective of this work is to generate information of interest to mechanical testing and mechanical design at elevated temperatures.

During the past fiscal year extensive load relaxation experiments have been carried out on type 316 stainless steel, a commercial zircaloy and on single crystal and polycrystal nickel. The former two materials are of current technological interest. The work on zircaloy is a joint effort with the Westinghouse R&D Center.

Recently E. W. Hart has extended his theory on plastic equation of state to include mechanistic considerations. It turned out that the range the load relaxation data obtained during this year is such that they are particularly suited to test the current theoretical development. Data analysis based on the new approach is still in progress. It is expected that the results will considerably strengthen and extend the concept and the applicability of plastic equation of state.

The work on grain boundary gas bubble, grain boundary cavity and intergranular fracture with and without the influence of irradiation effects is progressing satisfactorily. Some of the results of theoretical analysis will be included in a paper to be presented in the International Conference on Fundamental Aspects of Radiation Damage in Metals to be held in Gatlinburg, Tennessee in October. It is worth noting that the knowledge developed dur-

ing the past few years on grain boundary sliding has contributed to the solution of some of the problems. Since effects of irradiation on the ductility is being given more attention, it is expected that the research in this area will be emphasized in the future.

The proposed work on in-reactor creep and related phenomena, due to the manpower limitation, has not been progressing at a significant rate and will be given a low priority in the future. The principal investigator is involved, however, in the cyclotron creep project at Argonne National Laboratory where attempts are made to apply the concept of plastic equation of state to in-reactor creep.

Efforts were made during the past year to promote the application of the findings of the present program, especially the approach of plastic equation of state. The principal investigator is participating in the reactor development programs of ERDA on in-reactor creep and on advanced alloy development. The mechanics group at Cornell has initiated a program on the application of plastic equation of state in mechanical design. They will use in their investigation materials data input from this program. The principal investigator has given talks at the following institutes on the findings of this work and on plastic equation of state including NBS, HNL, HEDL, G.E. R&D Center, Westinghouse R&D Center, and Lehigh University.

In the following the results of the load relaxation experiments and the work on grain boundary gas bubble, grain boundary cavity and intergranular fracture will be described separately.

LOAD RELAXATION EXPERIMENTS

In this section some of the results of the load relaxation experiment will be described to illustrate the type of data obtained. The approach of data analysis and its implications will be discussed. At the present, as pointed out previously, the data analysis is still in progress. It should be completed by the end of this calendar year.

In the last renewal proposal the work on the effects of grain boundary sliding and microstructural changes in type 316 stainless steel is emphasized. The scope of the work in this area has been expanded during the last year to include the investigation of a zircaloy and single crystal and polycrystal nickel. The work on nickel is undertaken because single crystal specimens are available from which grain matrix behavior can be determined without the influence of grain boundary sliding and because it is a pure metal in which the effects caused by thermal aging of precipitates are absent. The zircaloy in addition to its commercial interests, represents a material in which the interplay between grain boundary sliding and grain matrix deformation can be different from that in type 316 stainless steel.

Load relaxation experiments have been performed on these materials at temperatures from room temperature to 650°C and as a function of varied hardness produced by deformation. In the following the experimental results will be discussed under four different topics. They are: a) grain matrix deformation; b) anelastic deformation; c) grain boundary sliding; and d) grain matrix deformation in single crystal and in polycrystal.

a. Grain Matrix Deformation

Considerable progress has been made during the past year on the understanding of the basic behavior of plastic deformation in the grain matrix. It can be described best by first discussing the experimental data obtained from load relaxation experiments.

The room temperature constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves (σ , stress and $\dot{\epsilon}_p$, plastic strain rate) of type 316 stainless steel obtained from the load relaxation experiment are shown in Fig. 1. These curves represents data from three different hardness produced by increasing amounts of plastic deformation. The plastic equation of state behavior is exhibited by the ability of these curves to translate along the straight line (slope = 0.13) in Fig. 1 to overlap each other. This is the so-called scaling behavior. Similar data are obtained at higher temperatures and on nickel and on a zircaloy.

As the temperature increases, two important observations can be made based on the data such as those shown in Fig. 1. First, the translation line shown in Fig. 1 no longer applies and some times it becomes a curved line. Second, the shape of the constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves change from those concave upward as shown in Fig. 1 to curves concave downward at higher temperatures. At intermediate temperatures, the measured constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves exhibit an inflection point.

The features of the constant hardness curves described above can be illustrated by the curves shown in Figs. 2 and 3. The curves in Fig. 2 are obtained from a type 316 stainless steel specimen. They are measured at different temperatures but at a constant hardness. It is seen that the curves change from concave

upward to downward as the temperature is increased. The curves in Fig. 3 are obtained from a zircaloy specimen. They are not obtained at a constant hardness but the same features as those in Fig. 2 are seen.

If it is assumed that all the curves in Fig. 2 are independent of the temperature, they can be translated along the $\log \dot{\epsilon}_p$ axis to overlap each other to form a continuous curve. The resulting composite curve is shown in Fig. 4. The required shift in $\log \dot{\epsilon}_p$ gives a measure of the apparent thermal activation energy. The apparent activation energy can be calculated from

$$\Delta \log \dot{\epsilon}_p = \frac{Q}{2.3R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (1)$$

where $\Delta \log \dot{\epsilon}_p$ is the shift required to overlap the constant hardness curves measured at T_1 and T_2 and R is the gas constant. The calculated apparent energy from the data in Figs. 3 and 4 and Equation 1, is found to depend on temperature and are shown in Fig. 5. The activation energy for creep has also been shown to be dependent on the temperature.¹

The work on plastic equation of state so far has considered data only in a limited temperature and plastic strain rate range. It should be noted, however, that the range of plastic strain rate covered by load relaxation data, for example in Fig. 1, is much wider than that usually covered by creep data to allow the observation of the behavior of plastic equation of state (scaling).

The composite constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curve shown in Fig. 4 covers a wider range of plastic strain rate compared to that in Fig. 1, such that it exhibits qualitatively the

entire range of behavior predicted by Hart² in a single curve. His predictions have generally been confirmed experimentally that in the high plastic strain rate and/or low temperature region the constant hardness curve is concave upward. In the low plastic strain rate and/or high temperature region it is concave downward. Based on the experimental data such as those shown in Figs. 1-5 some specific questions can be raised in order to fully characterize quantitatively the basic behavior of plastic deformation in the grain matrix. The search for the answer for these questions are currently the main thrust of load relaxation experiments. These questions are:

1. If there is a different scaling behavior for the high plastic strain rate region and for the low plastic strain rate region separately, what will be the scaling behavior in the transition region where the constant hardness curve exhibits an inflection point?
2. Is the assumption based on which the composite curve in Fig. 4 is constructed valid? It has been assumed that the shape of the constant hardness curve is independent of temperature.
3. What is the mechanistic basis for the temperature dependence of the apparent activation energy shown in Fig. 5?
4. What is the mechanistic basis for the change in the shape of the constant hardness curve between the high and low plastic strain rate regions?

Hart⁴ recently has proposed a more mechanistically based model which is an extension of his original phenomenological

theory. He suggests that in the high plastic strain rate and/or low temperature region the dislocation glide is the predominate rate determining process for plastic deformation. In the low plastic strain rate and/or high temperature region the dislocation climb is the predominate rate determining process for plastic deformation.

In the region where dislocation glide is important, the plastic strain rate will not be strongly dependent on the temperature. On the other hand the activation energy for dislocation climb should be that for vacancy motion.

The concept introduced above is not a new one but the form of the equations and the physical significance of the parameters introduced by Hart are different from those in the literature. Based on the preliminary results of the data analysis, Hart's model seems to be confirmed by the type of experimental data described previously and offers considerable insight as to future experimental and theoretical work.

For dislocation glide controlled plastic deformation Hart suggests

$$\dot{\epsilon}_p = A (\sigma - \sigma^*)^M \quad (2)$$

For dislocation climb controlled deformation, the expression is

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

In the region where both processes are important, they combine in parallel. In these two equations, the parameter M is a temperature independent constant and equals to the inverse of the slope of the translation line in the high plastic strain rate region such as the one shown in Fig. 1. The parameter A is a rate con-

stant and is temperature dependent. The parameter σ^* is the value of σ as the $\dot{\epsilon}_p$ in Equation 3 becomes infinite and is a measure of the level of hardness. The parameter λ is a constant which is temperature independent and governs the shape of the constant hardness curve in the low plastic strain rate and/or high temperature region. The parameter $\dot{\epsilon}^*$ is a materials constant and can be affected for example by heat-treatment. The activation energy for $\dot{\epsilon}^*$ is the same as that for vacancy motion.

At the present the analysis of the data such as those shown in Figs. 1-5 is in progress with the following objectives.

1. To determine the values of the parameters in Equations 2 and 3.
2. To use the model to answer quantitatively the four questions raised previously.
3. To compare the results of the present work with those based on the existing theories of dislocation dynamics and creep.

As pointed out previously that the preliminary results of the data analysis are very encouraging. These results have shown that the model described previously can be used to account for the scaling behavior in the transition region. The shape of the constant hardness curve especially in the transition region is influenced by the temperature. The observed temperature dependence of the apparent activation energy and the change in the shape of the hardness curve are also consistent with the model.

The principal investigator feels that the present work is at the verge of making a major break through in the understanding of plastic deformation in the grain matrix. His collaboration with

Dr. E. W. Hart of G.E. Research and Development Center has accelerated the progress. It is expected that most of the data analysis will be completed before the end of 1975 calendar year.

The result of the present work is also important practically. For example the zircaloy data of interest to the light water reactor applications are in the temperature and strain rate range shown in Fig. 3. It is seen that much of the data are in the transition region where simple scaling relationships do not apply. The new development described previously can be used to account for these data and therefore extended the applicability of plastic equation of state.

b. Anelastic Deformation

Anelastic deformation is time dependent but recoverable in contrast to plastic deformation which is non-recoverable. During the last year considerable progress has been made in this laboratory in the understanding of anelastic deformation in the NSF sponsored work. Since these new findings are relevant to the present work and to the work to be proposed for the next fiscal year, some of the recent results and their relationship to the work described in the last section will be discussed.

Anelastic deformation is an important mode of deformation for the situation where the load and/or temperature are changing rapidly for example in the initial stage of creep and in cyclic deformation. It is recoverable that is when the load is removed, the strain will return to zero. It is time dependent in that the anelastic strain rate at a given load decreases with time suggesting the existence of a saturation anelastic strain.

The work on anelastic deformation is designed to test and to

obtain quantitative information on the following hypotheses.

1. Saturation anelastic strain is linearly related to the applied stress

$$\sigma = M \epsilon_a^s \quad (4)$$

where ϵ_a^s is the saturation anelastic strain and M is a modulus.

2. The driving force for anelastic deformation is the difference between the saturation anelastic strain and the current anelastic strain such that

$$\dot{\epsilon}_a = f (\epsilon_a^s - \epsilon_a) \quad (5)$$

where $\dot{\epsilon}_a$ and ϵ_a are current anelastic strain rate and anelastic strain respectively.

Experimentally using both single crystal and polycrystal aluminum specimens, it has been found that there are two separate anelastic processes. There is a very rapid one with relaxation time less than one second. It has been identified to be associated with the grain boundary anelastic deformation as proposed by Zener originally.⁵ The magnitude of the saturation grain boundary anelastic strain is found to be linearly related to the applied stress and is roughly a factor of three to five larger than that of the elastic strain. The grain boundary anelastic strain rate is shown to be a linear function of the difference between the saturation anelastic strain and the current anelastic strain.

The other anelastic process lasts much longer in time. It has been identified to be associated with dislocation glide in the grain matrix. The magnitude of the grain matrix anelastic

strain is also found to be linearly related to the applied stress and is roughly equal to that of the elastic strain.

The grain matrix anelastic strain rate is found to be a power function of the difference between the saturation anelastic strain and the current anelastic strain. Using Equation 4 this difference can be expressed in terms of the difference between the applied stress and a resistance stress. The latter difference in stress has the physical significance of an effective stress for dislocation glide such that the grain matrix anelastic strain rate $\dot{\epsilon}_a^m$, becomes,

$$\dot{\epsilon}_a^m = A (\sigma_{\text{eff}})^M \quad (6)$$

where the parameters A and M are the same as those in Equation 2. It is noted that the same parameters are used in Equations 2 and 6. The reason is that both equations describe dislocation glide controlled deformation. If the term $(\sigma - \sigma^*)$ in Equation 2 is also viewed as an effective stress, these two equations should have the same form.

At the present anelastic experiments are planned on type 316 stainless steel. It is planned to evaluate the parameters A and M in Equation 6 using anelastic deformation data and to compare with the values of the same parameters in Equation 2 evaluated using load relaxation data.

If the possibility discussed above is found to be true experimentally, it will represent a great simplification in the understanding of dislocation dynamics. It also offers the practical advantage in mechanical testing because one can use the load relaxation experiment to evaluate parameters governing the anelastic deformation or vice versa. Experimentally it is easier sometimes

to do the load relaxation experiment and sometimes the anelastic deformation experiment.

c. Grain Boundary Sliding

The load relaxation data of type 316 stainless steel at temperatures higher than 500°C clearly exhibit the contribution of grain boundary sliding which have been discussed in the last progress report. Typical $\log \sigma - \log \dot{\epsilon}_p$ curves at these temperatures are shown in Fig. 6. Efforts are being made to identify quantitatively the contribution and also the constitutive relations of grain boundary sliding. Some of the results will be described below.

Qualitatively the high plastic strain rate region of the curves in Fig. 6 corresponds to grain matrix processes controlled deformation. As the plastic strain rate is decreased the increased contribution of grain boundary sliding increases the slope of $\log \sigma - \log \dot{\epsilon}_p$ curves. At the low plastic strain rate end of the S-shaped curve, the grain boundary offers little resistance to deformation and the deformation is controlled again by grain matrix deformation.

The curves shown in Fig. 6 are influenced by the effects of thermal aging such that they are not constant hardness curves and that the separation in $\log \sigma$ between the high plastic strain rate end and the low plastic strain rate end of the S-curve is larger than that without the influence of thermal aging. Type 316 stainless steel is known to be sensitive to thermally induced microstructural changes at elevated temperatures. Considerable effort has been devoted during the past fiscal year in obtaining constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves in which the influence of

thermal aging is absent.

Efforts are made also to obtain the shape and position of constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves reflecting only the grain matrix deformation. Quantitatively this means to obtain the value of the parameters in Equation 3 which describes the grain matrix curve. The strategy adopted in data analysis is to use the measured constant hardness $\log \sigma - \log \dot{\epsilon}_p$ curves discussed in the last paragraph, the corresponding grain matrix curve and the grain boundary sliding model proposed by Hart to calculate the stress-plastic strain rate curves for grain boundary sliding and to investigate their physical significance.

A typical $\log \sigma - \log \dot{\epsilon}_p$ curve obtained without the influence of thermal aging is shown in Fig. 7. The absence of thermal effects during the period when the data of curve A are taken can be demonstrated by the reloading experiment. At the end of the run for the curve A in Fig. 7, the specimen is reloaded to the stress level represented by curve B for the second load relaxation run. After an initial transient region (flat portion), curve B merges into curve A suggesting that there is no significant thermally induced microstructural change during the run for curve A. It should be noted that lengthy efforts in the thermal-mechanical treatment are involved in producing the data such as those shown in Fig. 7.

At the present data analysis is still in progress preliminary results seem to support some of the conclusions from the previous work on lead.⁷

d. Grain Matrix Deformation in
Single Crystal and in Polycrystal

As mentioned previously that load relaxation experiments have been performed on single crystal specimens in an attempt to measure the grain matrix $\log \sigma - \log \dot{\epsilon}_p$ curve independent of the influence of grain boundary sliding. It has been discussed previously that grain matrix curve is required for the purpose of evaluating the contribution of grain boundary sliding.

Typical load relaxation data on single crystal and polycrystal nickel specimen are shown in Fig. 8. These curves have about the same hardness (same σ^*) and both are in the region where grain matrix deformation is controlling. Fitting of these curves to Equation 3 gives the same value of the parameter λ . This means that they are of the same shape. The values of the parameter $\dot{\epsilon}^*$ in Equation 3 are different, however, for these curves. From the viewpoint of plastic equation of state these curves belong, therefore, to two different families.

The results described above suggest that single crystal data can give only the shape of the grain matrix curve and cannot give the position of the grain matrix curve for the purpose of analyzing grain boundary sliding data. The data such as those shown in Fig. 8 have not been reported in the literature and are quite revealing for delineating the single crystal behavior vs. the polycrystal grain matrix behavior. At the present due manpower limitation, this potential research direction is not being explored.

GRAIN BOUNDARY GAS BUBBLE, GRAIN BOUNDARY CAVITY
AND INTERGRANULAR FRACTURE

Under applied stress grain boundary cavities or gas bubbles will grow either by grain boundary sliding or by stress-induced atomic transport for example in creep or in low cycle fatigue at elevated temperatures. The growth of grain boundary cavities or gas bubbles will lead to intergranular fracture and will reduce ductility. During the past fiscal year this problem is analyzed theoretically based on which experimental work is planned.

It is interesting to note that this part of the work is planned originally to investigate the effects of gas bubbles on grain boundary sliding. It turned out that the problem of the effects of grain boundary sliding on the growth of grain boundary cavities is a more relevant one and is being pursued.

Grain boundary cavity can be nucleated during deformation due to the incompatibility between grain boundary sliding and grain matrix deformation. This is apparently an easy process based on experimental observations. It can be nucleated also in the form of gas bubbles in the absence of deformation by collecting helium atoms and by the formation of methane at the grain boundary.

Once the grain boundary cavity is formed, under applied stress it will grow. It can grow by continued grain boundary sliding which will shear the cavity. With the knowledge developed on grain boundary sliding in this laboratory it is concluded that this process is important only at high plastic strain rates (near the high strain rate end of the S-curve for example in Fig. 7) and will not be investigated at the present time.

Stress induced atomic transport is another process by which

the grain boundary cavity can grow. This is exactly analogous to diffusional creep suggested by Herring⁸ where the driving force is the normal stress at the grain boundary and the grain boundary serve as the source of vacancies and the cavity as the sink.

Atomic transport controlled grain boundary cavity growth has been analyzed extensively in the literature. However, the following two possibilities have not been given proper attention. First, the grain boundary normal stress can be reduced or significantly relaxed depending on the extent of the contribution of grain boundary sliding in plastic deformation. This possibility is consistent with experimental observation in that cavity growth has been found to occur predominated on the grain boundary which is normal to the applied stress where grain boundary sliding is absent. In life time predictions, the influence of grain boundary sliding can therefore play an important indirect role.

The absorption of vacancies at the cavity surface can be controlled by the local reaction rate there especially in the presence of point defects supersaturation produced by irradiation. This is the second possibility which has not been considered in the literature.

During the past fiscal year the formulation of the rate equation for stress induced atomic transport is developed for the situation where the rate limiting step is the absorption reaction at the sink. One of the important consequences of the second possibility is that the growth rate of the grain boundary cavity may be slowed in the reactor. The results of theoretical considerations of the two possibilities described above are summarized as mentioned previously in a paper for publication.

Experiments are planned to confirm the effect of grain boundary sliding in relaxing the normal stress at the grain boundary. Grain boundary cavity will be produced in the absence of applied stress by the formation of methane bubbles. The growth rate will be measured at stresses, plastic strain rates and temperatures such that the contribution of grain boundary sliding is varied. The cavity growth rate as a function of grain boundary angle relative to the applied stress axis will provide additional information to test the possibility proposed above. This experimental work is just being initiated.

SUMMARY

(1) Load relaxation experiments have been performed in the region where the grain matrix plastic deformation is important on type 316 stainless steel and a zircaloy specimen. The experimental data are being analyzed in terms of a mechanistically based model and plastic equation of state.

(2) Anelastic deformation in pure aluminum has been investigated. A dislocation glide controlled anelastic component is identified which may be governed by the same laws as those for plastic deformation at low temperature and/or high plastic strain rate.

(3) The investigation of grain boundary sliding in type 316 stainless steel is being continued. Experimental data without the influence of thermal aging are successfully obtained.

(4) Load relaxation data on single crystal and polycrystal nickel specimens show that the constant hardness curves of these specimens belong to two different families.

(5) The growth of the grain boundary cavity under applied stress and irradiation has been analyzed. It is found that the normal stress at the grain boundary which is the driving force for atomic transport controlled cavity growth can be relaxed by grain boundary sliding.

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Fig. 1, Load relaxation data of type 316 stainless steel at room temperature in the form of constant hardness curves.

Fig. 2, Load relaxation data of type 316 stainless steel taken at different temperatures but of the same hardness.

Fig. 3, Load relaxation data of a zircaloy in the form of constant hardness curves at different temperatures.

Fig. 4, Composite curve by translating the curves in Fig. 2 along the $\log \dot{\epsilon}_p$ axis to overlap each other. The coordinates shown are for the room temperature curve.

Fig. 5, Apparent activation energy for plastic deformation in type 316 stainless steel as a function of temperature calculated based on the data in Fig. 4.

Fig. 6, Load relaxation data of type 316 stainless steel at different temperatures showing the contribution of grain boundary sliding.

Fig. 7. Load relaxation data of type 316 stainless steel at 600°C in the form of a constant hardness curve. Curve B is obtained in a reloading run to show the absence of thermal aging.

Fig. 8, Load relaxation data of single crystal and polycrystal nickel in the form of constant hardness curves.