A Research Proposal

Investigation of the Microstructure after Creep Deformation of Metals at Very Low Stresses

April, 2006

Summary

Creep refers to the time-dependent plastic deformation of materials under constant stress or constant load. It assumes major importance in the high-temperature range; namely above one-half of the melting temperature. Detailed studies on the creep behavior of materials, for both ultra-high purity and commercial purity, at low stresses are of scientific and practical significance. In this research, focus will be placed on studying the microstructure associated with a creep process that leads to acceleration in creep rates at low stress. This process is relevant to the prediction of geological deformation but also because of its relevance to many design considerations.

It is the purpose of this proposal to describe a program in which clarification of microstructural features related to low-stress creep in metals is sought. The proposal includes recent development, the issue to be addressed, approach to be adopted, and the procedures to be applied, are described.

The proposed research program is fundamental in scope and involves materials preparation, mechanical testing, microstructure characterization, and analysis. As a result, the program will lead to gaining knowledge and training in areas that would enhance my opportunities for a good professional career.

Table of Contents

1. Introduction

Creep refers to the time-dependent plastic deformation of materials under constant stress or constant load. It assumes major importance in the high-temperature range; namely above one-half of the melting temperature. Figure 1 shows a typical creep curve observed in metals. The slope of this creep curve is referred to as the creep rate. Following an initial rapid elongation, the creep rate decreases with time (primary creep or stage I) then reaches a steady state in which the creep rate changes little with time (secondary creep or stage II), and finally the creep rate increases rapidly with time (tertiary stage) until fracture occurs.

More attention has been paid to the steady-state stage of creep curve, basically because (a) primary creep is a transient stage with relatively short duration; (b) tertiary creep is a stage in which materials could fail in any time without warning, and hence no components or parts can be working in this stage; and (c) steady state creep is a stable stage of relatively long duration and most of engineering designs are based on it. At high strain rates (high-stresses), a log-log plot of steady state creep rate *vs*. stress at constant stress results in a straight line. This line is consistent with the prediction of creep theories based on defects (such as dislocations) motion that leads to the following equation:

$$\dot{\gamma} = A(b/d)^s (\tau/G)^n \exp(-Q_c/RT) \qquad (1)$$

where $\dot{\gamma}$ is the steady-state shear creep rate, A is a constant, b is the Burgers vector, d is the grain size, s is the grain size sensitivity, τ is the applied shear stress, G is the shear modulus, n is the stress exponent, T is the absolute temperature, and Q_c is the activation energy for creep. According to Eq. 1, n represents the slope of the straight line in the log-log plot of steady state creep rate, $\dot{\gamma}$, *vs*. applied stress, τ.

Detailed studies on the creep behavior of materials, for both ultra-high purity and commercial purity, at low stresses are of scientific and practical significance. From a scientific point of view, data obtained from such studies are critical to the characterization of creep behavior in terms of deformation mechanisms. From a practical point of view, information inferred from such studies is useful not only because of its relevance to the prediction of geological deformation but also because of its relevance to many design considerations. The importance of a detailed knowledge of the low stress

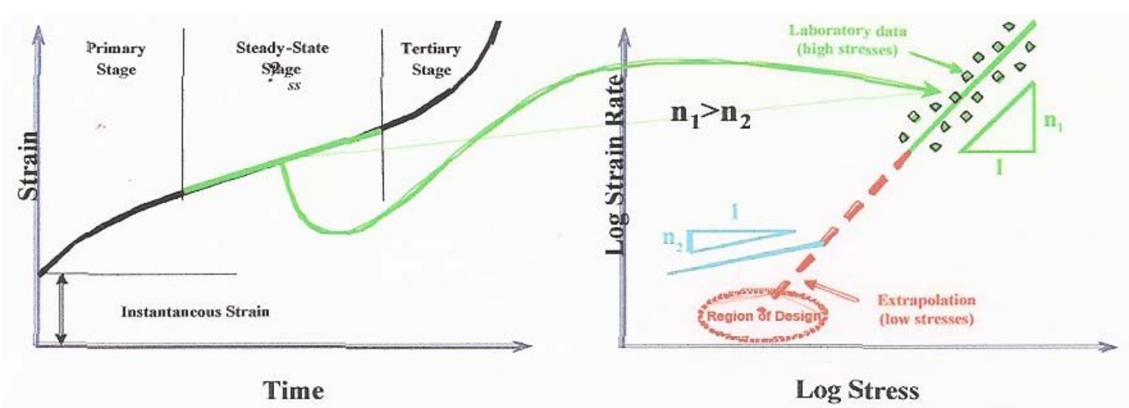


Figure 1(a): Typical creep curve **Figure 1(b): Extrapolating creep data to low stresses**

behavior to design considerations may be appreciated when it is recognized that extrapolation of the short-term data obtained at high stresses to the long-term data needed at very low stresses may result, in some cases (ultra-high purity metals), in a substantial underestimation of the strain rate, and thus lead to premature in-operation failures. This situation is encountered under two combined conditions: (a) the mechanism for creep at low strain rates differs from that at high strain rates, and (b) when the mechanism at low strain rates is characterized by a straight line whose slope (= ***n***) is less than that for the mechanism at high strain rates (Figure 1(b)).

Accordingly, it is no surprise that the very low-stress regime of high-temperature deformation has received considerable attention over the past four decades, and this has resulted in three major achievements. First, pertinent deformation mechanisms, such Nabarro Herring creep [1, 2], were formulated. Second, experimental data (mechanical and substructural) were obtained to examine the validity of the proposed deformation mechanisms. Third, a new type of creep behavior, referred to as Harper-Dorn creep [3], was reported. Despite this good progress and its potential impact on our knowledge of low-stress creep behavior, several issues remain unsolved. Primary among those issues is the issue related to the microstructural details corresponding to the low stress creep mechanism that is responsible for the enhancement of the creep rates in large-grained metals such as Al. (Figure 2).

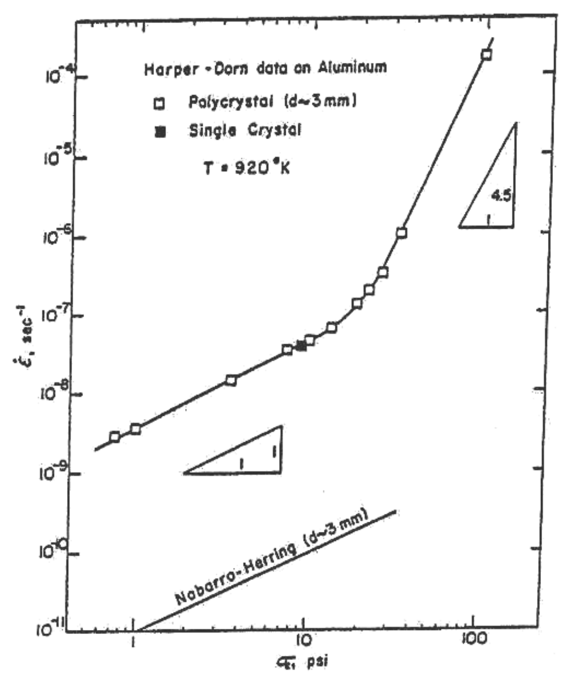


Figure 2***: Original creep data for Harper-Dorn creep in Al [1] plotted as strain rate vs. effective stress on a logarithmic scale, included is the prediction of Nabarro-Herring creep [4,5]***

The lack of reliable information and analysis that can be used to address aforementioned issue represents a major deficiency in our present fundamental knowledge on creep.

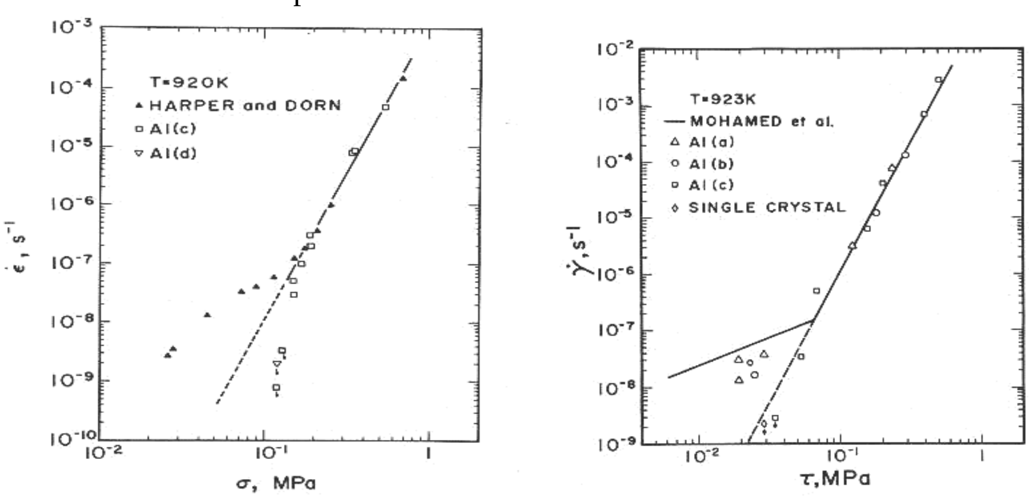
It is the purpose of this proposal to describe a program that should make a major contribution to the clarification of microstructural features related to low-stress creep in metals. In the following sections of the proposal, details of the proposed program, including recent development, the issue to be addressed, approach to be adopted, and the procedures to be applied, are described.

2. Proposed Program

2.1. Recent Developments

Recent research using Al has focused on performing long-term tests in which emphasis was made on the amount of strain and not the duration of the test. The data obtained as a result of these long-term tests have led to the following findings [4, 5, 6]:

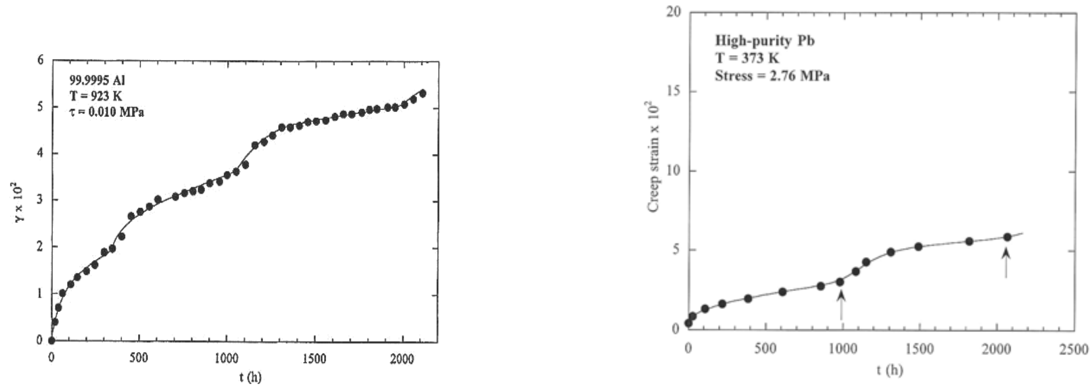
- Harper-Dorn creep does not always occur in Al when crept using large grain sizes and very low stresses. As shown by the data of Figure 2, 99.99 Al, unlike 99.9995 Al, does not exhibit the accelerated creep rates associated with Harper-Dorn creep despite the fact that both grades of Al were crept under the same conditions of temperature and stress.



(a) (b)

Figure 2: ***Strain rate versus stress for Al at 920 K (logarithmic scale)*** *[18]*
(a) Al(c) and Al(d) of 99.99 purity and whose samples have initial dislocation density of about 10^6/cm^2 do not exhibit Harper-Dorn creep (tensile data).
(b) Al(a) and Al(b) of purity of 99.9995 and whose samples have initial dislocation density of about 10^4/cm^2 exhibit Harper-Dorn creep; this contrasts with behavior of Al(c) and an Al single crystal, which do not exhibit this type of creep behavior (shear data).

- The creep curves associated with Harper-Dorn creep are not smooth (Figure 3a). Following transient strains of the order 0.01 to 0.02, the creep curves exhibit regular, periodic accelerations. Such periodic accelerations are similar in trend to the accelerations that were reported for Pb [7] (Figure 3b).



(a) **(b)**

Figure 3: creep curves showing regular, periodic acceleration

(a) High purity Al (99.9995) crept at 923 K and 0. 01 MPa [28, 29] (the Harper-Dorn region)

(b) High-purity Pb crept at 373 K and 2.76 MPa [30] (high stress-region)

2.2. Issue

Figure 3 shows the creep curve for 99.9995% Al in the low-stress region. The curve shows abrupt changes (accelerated jumps) in creep rate at progressive values of strain along the curve, as contrast to 99.95% Al that shows continuous decrease in the creep rate even after very long periods of time [1]. It is suggested in the literature [8] that these periodic accelerations may be associated with the dynamic recrystallization. However it was reported [9] that dynamic grain growth might lead to the observation of periodic oscillations similar to those noted in the creep curve of Al as reported by Ginter et al. (Figures 3a). While Ginter et al. [5, 6] reported that the cross sections of most of the samples tested represented single crystals (no boundaries were), one cannot rule out the possibility that there might be boundaries at planes inclined to the cross section of the sample gage. Accordingly, it is necessary to perform new experiments that would establish whether dynamic grain growth contributes to the occurrence of oscillations in the creep curve of Al.

2.3. Experimental Approach

2.3.1. <u>Testing</u>. In measuring creep strains double shear technique [10] will be adopted and care will be exercised to use very sensitive, high resolution equipment along with reliable temperature control. Double shear will be used in most cases to avoid the necking problem, especially at large strains.

2.3.1. <u>Single crystals vs. Polycrystals</u>. In order to examine in an unambiguous manner whether or not dynamic grain growth is responsible for the occurrence of jumps (accelerations) in the creep curve of Al, single crystals of high-purity Al will be tested in double shear under conditions that are identical with those used by Ginter et al. [5, 6] in testing high-purity polycrystalline Al; T = 920 K and shear stresses < 0.06 MPa. Also, the characteristics of the accelerations (shape, magnitude, strain intervals between successive accelerations, and initial strain before the first jump) will be carefully examined. Furthermore, the effect of varying the grain size or sample thickness will be investigated. The results of such an investigation will have implications in terms of the occurrence of dynamic growth and the multiplication of dislocations by surface sources.

2.3.2. Microscopy. Substructural examination of the as-received and crept samples will be conducted using (a) light-optical microscope (LOM) and (b) Philips CM 20 transmission electron microscope (TEM) operating at 200 kV.

2.4. Materials and Procedures

Experiments and microscopy will be conducted on the following materials of high purity and commercial purity.

2.4.1. Aluminum. A systematic study of A1 that involves long-term creep tests will provide definitive information on the characteristics of the creep behavior of A1 under the condition of very low stresses.

2.4.2. Pb. Pb has a low melting (T_m for Pb = 600 K) and, therefore, its creep study at temperatures close to the melting point can be performed in a simple manner. Testing Pb will serve the following purposes: an investigation of the occurrence of low-stress creep at large strains; data are available for Pb at small strains [5]. In addition, the large difference in the value of the staking fault energy between Al (200 erg/cm^2) and Pb (30 erg/cm^2) would provide an important parameter that can shed light on the nature and origin of creep.

2.5. *Impact of research on my education*

The proposed research program is fundamental in scope and involves materials preparation, mechanical testing, microstructure characterization, and analysis. Also, in carrying out research experiments, I will be using state-of-the-art materials research facilities. As a result, the program will allow me to gain knowledge and training in areas that enhance my opportunities to join a good research university as a graduate student. This training will pave the way for me to attain my goal of becoming a leader in the industry, a scientist in national laboratories, or a scholar in universities. In addition, the proposed program will allow us to attract young engineering undergraduate students, expose them to state-of-the-art materials research, and possibly interest them in joining the graduate program on materials.

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