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New observations on high-temperature creep at very low stresses

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ABSTRACT

Creep tests were conducted in compression to evaluate the flow behavior of aluminum at very high temperatures and low stresses. The experiments used two types of specimens: single crystals of 99.999% purity and oligocrystalline samples of 99.97% purity. Results obtained for the single crystals lie consistently between the conventional region of Harper–Dorn creep and the anticipated behavior based on an extrapolation of conventional 5-power creep. Using etch pit studies with the single crystals, it is shown there is no evidence for subgrain formation and the measured dislocation densities are consistent with data extrapolated from the conventional 5-power region. The creep results on single crystals suggest the stress exponent is close to ~3 at low stresses. It is demonstrated these results are consistent with earlier data including the results of Harper and Dorn when their data are plotted in terms of the true applied stress without incorporating a threshold stress.

1. Introduction

The creep behavior of polycrystalline metals at very high temperatures T was generally attributed to the occurrence of Nabarro–Herring diffusional creep [1,2] until 1957 when Harper and Dorn [3] reported unusual results when testing pure (99.99% purity) aluminum with a grain size of 3.3 mm. In tests conducted at a temperature of 923 K, corresponding to a very high homologous temperature of ~0.99 T_m where T_m is the absolute melting temperature, they observed creep rates at very low stresses ($\leq$0.1 MPa) which were more than two orders of magnitude faster than those anticipated for conventional Nabarro–Herring creep. The characteristics of this new creep behavior included a stress exponent, n, equal to 1, an activation energy for creep equal to the anticipated value for lattice self-diffusion and, based on a test using a single crystal, creep rates which were independent of the grain size. Whereas the first two of these characteristics are consistent with Nabarro–Herring creep, an independence of grain size provides a clear demonstration that Nabarro–Herring creep is not the rate-controlling mechanism. From these experimental data, Harper and Dorn [3] concluded they had observed a Newtonian viscous type of flow which probably occurred through some unidentified dislocation process. Subsequently, several additional investigations confirmed the general characteristics of Harper–Dorn creep in aluminum and Al-based alloys in creep testing at very high homologous temperatures [4–9]. Two recent reports provide broad overviews of these early data [10,11].

Despite these consistent results, the occurrence of Harper–Dorn creep has not been without controversy. McNee et al. [12] conducted experiments using pure Al and were unable to reproduce the original data of Harper and Dorn [3]. Using samples of 99.99% purity aluminum, Blum and coworkers [13–15] performed creep tests in compression and obtained a stress exponent of ~6.6 instead of 1 at low stresses, thereby suggesting the occurrence of creep flow within the conventional five-power creep regime. Based on these results, they suggested the earlier tests at very low stresses may have failed to achieve a genuine steady-state behavior so that the creep rates were over-estimated.

More recently, Mohamed and coworkers [16–19] conducted creep investigations on polycrystalline Al and Pb using samples of both metals having two different purities (99.99% and 99.9995% purity Al and 99.95% and 99.999% Pb) and they reached several significant conclusions. First, they reported the occurrence of conventional Harper–Dorn creep in the two sets of samples having the highest purities whereas they failed to observe Harper–Dorn creep in the two materials having lower purity. Second, in the samples of highest purity they noted the occurrence of periodic and cyclic accelerations in the strain–time behavior which, they suggested,

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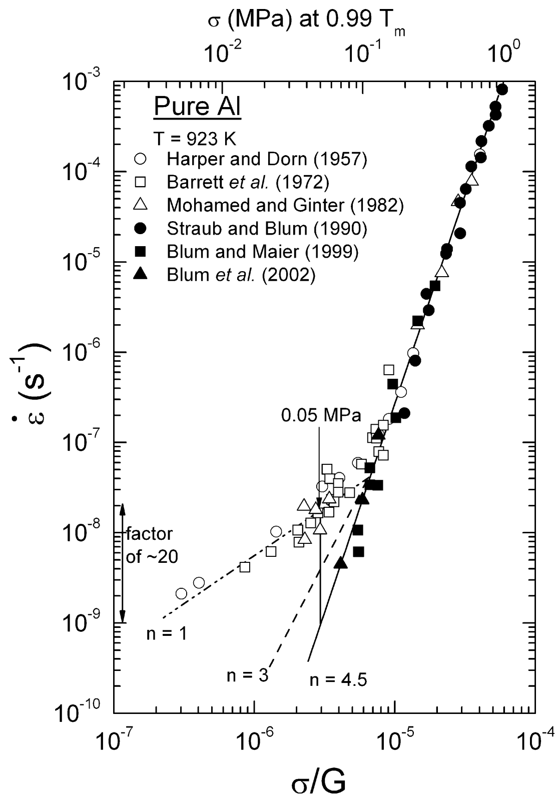

Fig. 1. Strain rate vs. normalized stress for pure Al at 923 K showing the transition to traditional Harper–Dorn creep at low stresses [3,6,9,13,14,21].

were consistent with the occurrence of dynamic recrystallization as a restoration process. However, it was subsequently noted that the accelerations in the creep behavior failed to occur at the regular intervals anticipated for the advent of dynamic recrystallization and therefore grain growth may provide an alternative explanation [20]. Third, and of considerable importance, the experiments suggested that a stress exponent of ~1 occurred only at the lower creep strains whereas there was a transition to a stress exponent >1 at higher strains [19].

These results demonstrate there are ambiguities in the creep data reported for the creep of aluminum at very high temperatures. The data plotted in Fig. 1 confirm these differences where the creep data have been normalized to a temperature of 923 K using the coefficient for lattice self-diffusion and the results are plotted logarithmically as the steady-state strain rate, $\dot{\varepsilon}$, versus the normalized stress, σ/G, where σ is the applied stress and G is the shear modulus [3,6,9,13,14,21]. Thus, there is a very clear difference in these data at the very lowest stresses and the vertical line at a stress of 0.05 MPa suggests a difference by a factor of ~20 between the creep rates in conventional Harper–Dorn creep and the extrapolated dislocation creep line with $n = 4.5$.

The present study was undertaken to examine these differences by conducting creep tests within the anticipated region of Harper–Dorn creep using large samples of high purity and testing these samples to relatively high strains.

2. Experimental materials and procedures

Tests were conducted using aluminum samples in two different forms: (i) single crystals having a 99.999% purity and with a [1 0 0] orientation and (ii) oligocrystalline samples of 99.97% purity where these samples are polycrystalline but contain only a very small number of grains. Major emphasis was placed on the testing of single crystals because the absence of grain boundaries necessarily precludes the occurrence of Nabarro–Herring diffusional creep or any possible erroneous effects due to grain growth. All of the single crystals were tested with large diameters (~25 mm) in order to minimize possible surface effects such as softening through dislocation loss or hardening because of the presence of an oxide layer [15]. The oligocrystalline samples had square cross-sections with dimensions of 12 mm × 12 mm.

All of the tests were conducted in compression at a temperature of 913 ± 1 K. Prior to applying the load, specimens were annealed at the test temperature for ~50 h under a very low stress corresponding to ~0.002 MPa where this low stress was used to maintain contact between the sample, the compression platens and the loading rods of the creep machine. An alumina-based liquid lubricant or boron nitride was used between the samples and the loading platens to reduce any frictional effects. Careful monitoring showed the temperature variation along the lengths of the samples remained within ±2 K. Strains were recorded in most tests using a laser-based extensometer but in some tests the strains were monitored using an LVDT (linear variable displacement transducer) attached to the creep machine close to the ends of the specimens. To provide an easy and direct comparison with published data, all results were subsequently normalized to a temperature of 923 K.

Selected specimens were electropolished after creep testing using a mixture of 90% acetone and 10% perchloric acid and they were then etched for 6–8 s using a solution of 50% HCl, 47% HNO_3 and 3% HF. The dislocation density, ρ, was calculated for each of these specimens using the expression [22]:

$$\rho = \frac{2N}{A} \tag{1}$$

where N is the number of etch pits recorded within area A.

3. Experimental results

Many of the tests were conducted using stress changes in which the stress is changed abruptly to a higher or lower level. An example is shown in Fig. 2(a) where the strain is plotted against time for different levels of the applied engineering stress and the same data are recorded in Fig. 2(b) as the instantaneous strain rate versus strain. Inspection of Fig. 2(b) shows no evidence for work hardening or softening at these extremely low stresses although the total strain interval of ~0.012 is sufficient for the occurrence of significant changes in the dislocation structure. Furthermore, the creep rates recorded at a stress of 0.03 MPa within the first strain interval at strains <0.002 are effectively matched by the creep rates recorded at the same applied stress near a strain of ~0.01 after the occurrence of intermediate creep at higher stresses. This reproducibility over a wide strain interval confirms that creep is occurring under reasonably steady-state conditions. There is also no evidence in Fig. 2(b) for a primary transient stage where the creep rate decreases.

It is important to note that the strains in Fig. 2(a) were recorded using a laser extensometer focused at the specimen surface so that any extraneous effects due to elastic and/or plastic adjustments between the specimen and other parts of the loading train were effectively excluded from the strain measurements. It is apparent from Fig. 2(b), and was evident also in other tests, that the creep rates measured at different strains under the same level of the applied stress are in agreement to within a factor of ~2. Thus, it is reasonable to conclude that any transient effects in these tests are negligible, the measured rates are very close to true steady-state conditions and any minor variations in the measured creep rates within small strain intervals probably reflect inhomogeneities in the evolution of strain within the samples. Similar strain rate–strain plots were obtained for all tests to ensure the development of true steady-state conditions.