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Long-term annealing of high purity aluminum single crystals: New insights into Harper-Dorn creep

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ABSTRACT

Single crystals of 99.999% and 99.9999% pure aluminum were annealed at high elevated temperatures ($0.98T_m$) for relatively long times of up to one year, the longest in the literature. Remarkably, the dislocation density remains relatively constant at a value of about $10^9\ m^{-2}$ over a period of one year. The stability suggests some sort of "frustration" limit. This has implications towards the so-called "Harper-Dorn creep" that generally occurs at fairly high temperatures (e.g. $> 0.90T_m$) and very low stresses. It is possible that ordinary five-power-law creep occurs within the tradition Harper-Dorn regime with very low initial dislocation densities in aluminum. Higher initial dislocation densities, such as with this annealing study, may lead to Harper-Dorn (Newtonian) creep.

1. Introduction

Long-term annealing of < 100 > and < 111 > oriented 99.999 (5 N) and 99.9999% (6 N) pure aluminum single-crystals at $0.98T_m$ was investigated in order to determine the change in dislocation density with various times up to one year.

Note that, from Table 1, that the short-term annealed dislocation density values from other studies range from $10^8\ m^{-2}$ to a much higher value of over $10^{11}\ m^{-2}$. The dislocation density values across different studies do not show and noticeable trend with purity as well as the annealing conditions. As-received dislocation densities were reported to be $3 \times 10^5\ m^{-2}$, $6.0 \times 10^{10}\ m^{-2}$ and $6.5 \times 10^7\ m^{-2}$. Overall, the starting dislocation densities (either annealed or as-received) vary by six orders of magnitude and these values will be shown to be of the order of those observed within the so-called Harper-Dorn regime. This observation will become important in later discussions. Again, the question remains as to whether longer annealing times (up to one year) can lead to lower dislocation densities. Certainly, from pure energy considerations, we expect the dislocation density to decrease with annealing time.

As Table I indicates, one year, by far, is the longest annealing time ever performed. The existence of a "frustration limit" of the dislocation density, suggested by Ardell and coworkers [7,10,11,34,38] for Harper-Dorn creep {low-stress and generally very high temperatures (e.g. $0.98T_m$) [1]}, in which the dislocation density does not decrease below a certain value (even at very low stress), is, thus, also examined in this work.

Harper and Dorn [1] suggested low stress-exponent (n) creep at very low stresses according to

$$\dot{\varepsilon}_{ss} = A_{HD}\left(\frac{D_{sd}Gb}{kT}\right)\left(\frac{\sigma}{G}\right)^n \tag{1}$$

where A_{HD} is the Harper-Dorn coefficient, D_{sd} the lattice self-diffusion coefficient, G is the shear modulus, b is the Burger's vector, σ is the stress (a threshold stress was subtracted by Harper and Dorn from the applied stress to give this σ value [2]) and n has a value of 1. {Interestingly, had this (probably fictitious) threshold stress not been subtracted, three-power law creep is observed [2] instead of Newtonian creep}.

Theoretically, a dislocation network creep model developed by the authors [15] in an earlier article, suggests that if the dislocation density varies with the steady-state stress as roughly $\rho_{ss}^{1/2} \propto \frac{\sigma_{ss}}{G}$, (as with classic five-power law creep) in the low stress regime (see Fig. 1), n is slightly larger than 3. On the other hand, for a constant dislocation density, n is about 1. This is roughly justified by the Orowan equation,

$$\dot{\varepsilon} = \rho_m b v \tag{2}$$

If ρ_m is constant,

$$v \propto \sigma^1, \tag{3}$$

$$\dot{\varepsilon} \propto \sigma^1 \tag{4}$$

But if ρ_m changes with stress,

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Table 1
Average dislocation densities of aluminum in the as-received and annealed conditions. PX – polycrystal, SX – single crystal.

Material	Dislocation Density (ρ)	Conditions	Reference
1. 99.99% coarse-grained PX Al	6.0×10^{10} m^{-2}	As received	[3]
	5.0×10^{10} m^{-2}	Annealed 773 K for 10 h	
	6.0×10^{9} m^{-2}	Annealed 823 K for 1 h	
	4.5×10^{9} m^{-2}	Annealed 903 K for 10 h	
5. 99.99% PX Al	2.4×10^{11} m^{-2}	Annealed (in vacuum) 773 K for 25 min	[4]
6. 99.994% SX Al	1.3×10^{7} m^{-2}	Annealed (in creep machine under < 0.00275 MPa) 823 K for 36 h	[5]
7. 99.9995% SX Al	1.0×10^{8} m^{-2}	Annealed 926 K for 50 h	[6]
8. 99.99% SX Al	3.0×10^{11} m^{-2}	Annealed 923 K for 48 h	[7]
9. 99.999% SX Al	6.0×10^{10} m^{-2}	Annealed 926 K for 50 h	[8]
10. 99.999% PX Al	4.3×10^{11} m^{-2}	Annealed 698 K for 1 h	[9]
11. 99.999% SX Al	6.5×10^{7} m^{-2}	Annealed 913 K for 50 h	[32]
12. SX Al *(purity not reported)*	2.0×10^{6} m^{-2}	As-received	[30]

$$\dot{\varepsilon} \propto \sigma^{n} \tag{5}$$

(where 4.5 > n > 1)

Hence, the observations of a constant dislocation density with decreasing stress, and a stress exponent of 1, are self-consistent for Harper-Dorn proponents. Opponents of the classical Harper-Dorn creep expect the observation of a stress-dependent dislocation density. Therefore, the nature of dislocation density variation with the stress in the Harper-Dorn regime may be the key to resolution of the existence of classical Harper-Dorn creep in aluminum. It should be mentioned that it is assumed that the strength of the structure within the Harper-Dorn regime is expected to be provided by the Frank dislocation network, just as at higher stresses and lower temperatures within the five-power-law regime [29,33].

Fig. 1 is compiled from creep studies by several investigators both supporting and refuting the existence of the classic Harper Dorn creep [5,8,9,16,18–20]. Lin et al. [16] and Barrett et al. [5] both suggested that the dislocation density remains fixed with decreasing stress, leveling out at about 10^{8} m^{-2} and Harper-Dorn Creep was suggested to be observed. "Network frustration" was suggested by Lin et al. to explain the observations. Some earlier work by the authors [2,12,13,15,19] suggests that the dislocation values, in fact, continue to decrease with stress in a manner as with lower temperature and higher stress five-power-law creep of aluminum, as also did Nes [14]. Thus, an ancillary purpose of this annealing study is to check the concept of a "frustration limit" for low stress, high-temperature, creep deformation.

As just mentioned, Ardell et al. suggested that the dislocation density in the in the Harper-Dorn regime is constant with changing applied stress due to a dislocation network frustration. This was suggested to be due to an inability of the dislocation network to coarsen due to Frank's rule [17], which may not be satisfied with coarsening of the dislocation network at lower values of the Frank network dislocation density.

$$\sum_{1}^{n} b_i = 0 \tag{6}$$

where b_i is the Burgers vector of the *i*th dislocation (link) meeting at the node in the network. Long-term annealing experiments could confirm or refute the existence of this frustration limit for the starting dislocation density. The proposed annealing experiments are simply the limit of the change is dislocation density as the stress decreases to zero.

2. Experimental procedure

Single crystal aluminum samples for this study were purchased from Material–Technologie & Kristalle GmbH (MaTecK), Jülich, Germany of 5 N purity (99.999% pure) and cylindrical dimensions of 127 mm length × 25.4 mm diameter and an orientation of < 100 > or < 111 > along the length of the cylinder. 6 N purity (99.9999% pure) Al single crystals were also used with a < 100 > orientation and 19 mm

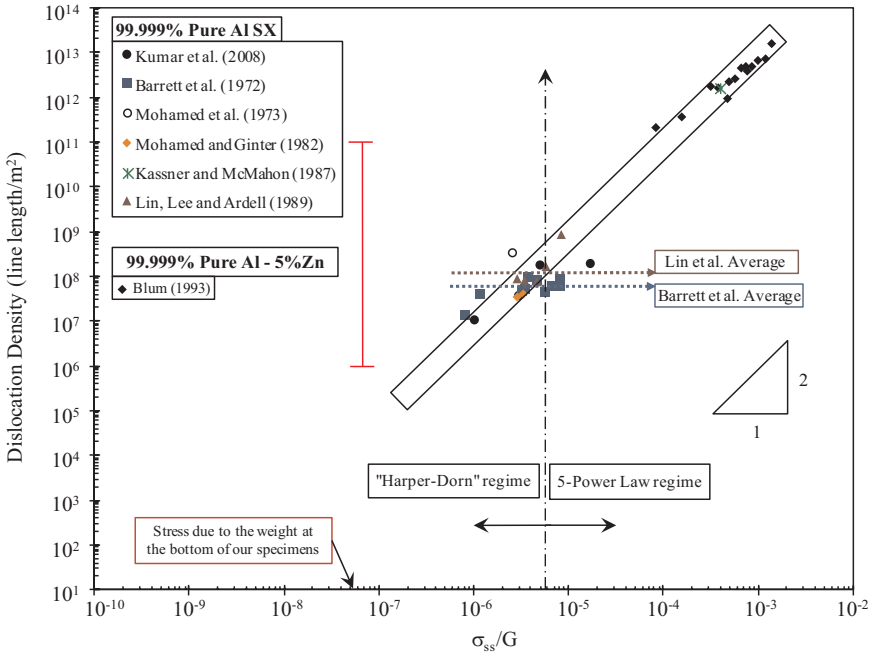

Fig. 1. Steady-state dislocation density versus the modulus-compensated steady-state stress at an elevated temperature of 923 K (0.99 T_m) based on earlier work and the authors' previous work in [2]. The data of Lin et al. [16] and that of Barrett et al. [5] suggest a lower limit of the dislocation density (ρ). The work by Barrett et al. [5] and Kumar et al. [2] may suggest a continual decrease in the dislocation density with decreasing stress. {The shear modulus (*G*) used was 16.96 GPa at 920 K (0.98T_m) [21]}. Note that the initial dislocation densities of Table I are of the same order as the steady-state dislocations densities at low (e.g. Harper-Dorn) stresses.