# Synopsis

Name of the student: Shobhit Pratap Singh

Department: Materials Engineering, Indian Institute of Science, Bangalore, India – 560012

Title of the thesis: Evaluation of Low-Stress High-Temperature Creep: Insights into the Harper-Dorn Creep

........................................................................................................................................

The theory of creep behavior of single crystals and polycrystalline materials having large grains at very low-stresses and at very high temperatures, as originally proposed by Harper and Dorn in 1957, has been under constant debate since its proposition. The major dispute over the initial observations of Harper and Dorn generally concern the value of the reported creep exponent: While Harper and Dorn reported it to be 1, later many observed it to range from 1 to 3 (**Fig. 1**).
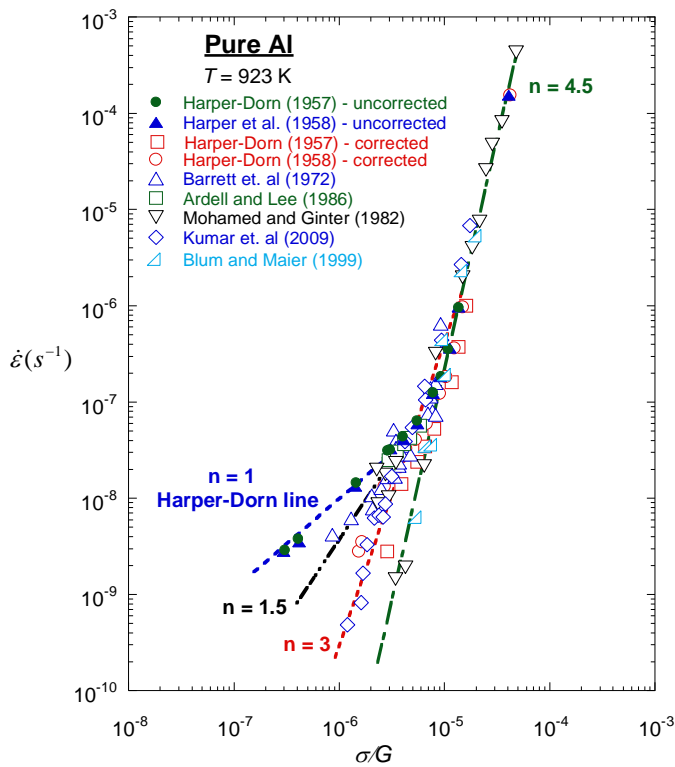


Fig. 1: Variation of the steady state strain rate as function of the normalized stress, as reported in several studies on Al, illustrating the dispute over the value of creep stress exponent in the low stress-high temperature regime, which is often known as the Harper-Dorn creep regime. Given the large span in which the steady-state strain rate may lie (e.g., over a factor of 50 at a stress of $10^{-6}G$), it is critical to resolve the dispute pertaining to the creep behavior of materials in the Harper-Dorn creep regime.

The variation of the dislocation density with the applied creep stress was later reported to be independent of the applied stress by a few investigators, while a few reported an increase in the dislocation density with stress in the "so-called" Harper-Dorn creep regime. Since its advent, the mechanism of creep in the Harper-Dorn creep regime has been studied using numerous metals and ceramics; however, often inconsistent results have been reported. Hence, a critical examination of the reported creep stress exponents and the dislocation density dependence on the applied stress is imperative to resolve the creep in the Harper-Dorn creep regime, which is perhaps one of the most debated and unresolved issues in the general area of the mechanical behavior of materials. Accordingly, the principal objectives of this work are to study the creep behavior of high purity single crystals in the Harper-Dorn creep regime.

Primarily, creep tests are extensively conducted using high purity (100)-oriented LiF single crystals, which has never been examined in the Harper-Dorn creep regime. However, the creep response of LiF in the "five"-power law regime is comprehensively reported, which proffers an easy comparison of current study with the literature. In addition, iso-thermal annealing of LiF crystals is conducted up to 10,000 h at a homologous temperature of 0.92 (i.e., 0.92 $T_m$, where $T_{\mathrm{m}}$ is the melting temperature) to observe the evolution of dislocation density. The dislocations were observed using etch-pit method and the evolution of dislocation density was recorded by observing the iso-thermally annealed sample at regular intervals. The dislocation density decreases to ~3 × $10^9$ m$^{-2}$ after 150 h of annealing, and remains saturated at this value during subsequent annealing (see **Fig. 2**). NaCl, another well-studied material having the crystal structure same as of LiF, where Harper-Dorn creep has been previously evaluated, is also isothermally annealed at 0.92 $T_m$ to investigate the evolution of dislocation density using etch-pit method. Here also, a saturation in the dislocation density, to a value ~$10^9$ m$^{-2}$, is observed under isothermal annealing condition (see

**Fig. 3**). These observations are consistent with a recent work on Al single crystals that utilized etch-pit method to show attainment of a saturation dislocation density (to a value close to $10^9$ m$^{-2}$) in these samples due to the iso-thermal annealing at a temperature near the melting temperature. As a part of this study, the dislocation density values reported for Al single crystals were reexamined using x-ray topography at a synchrotron, and it is confirmed that the numbers obtained using etch-pits are reasonably accurate. Hence, it is concluded that isothermal annealing of high purity single crystals at temperatures close to melting temperature ($>0.9\ T_\mathrm{m}$) cannot lead to a decrease in the dislocation density to an arbitrary low value (e.g., making it dislocation free). The existence of a finite value of the saturation dislocation density attainable through isothermal annealing confirms the idea of the frustration of the dislocation network.
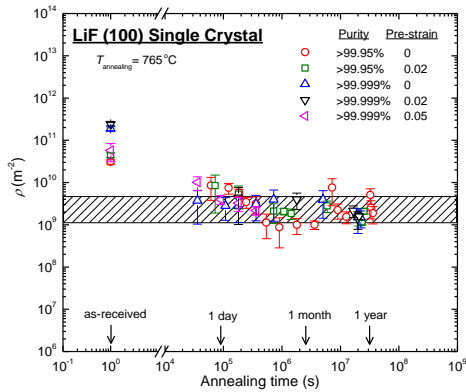


Fig. 2: Single crystalline LiF annealed at 0.92 $T_m$: Evolution of dislocation density for materials with different purity and pre-strain history with annealing time. The hatched region shows the saturation dislocation density. During intermittent observation, they were not polished metallographically and the surface layers were removed by chemical etching only.
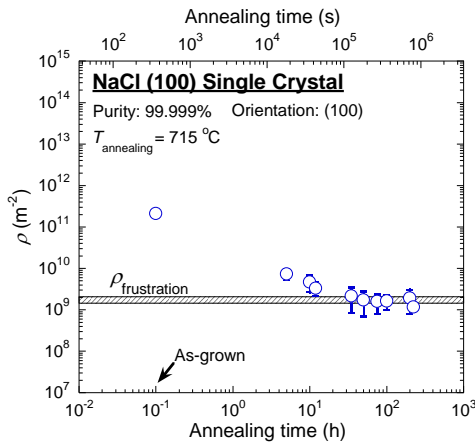


Fig. 3: Single crystalline NaCl annealed at 0.92 $T_m$: Evolution of dislocation density with annealing time. The hatched region shows the saturation dislocation density. The samples were furnace cooled. During intermittent observation, they were not polished metallographically and the surface layers were removed by chemical etching only.

The results demonstrate that the creep stress exponent of LiF at 0.95 $T_m$ decreases from ~3.5 (i.e., "five"-power law for ionic crystals) to ~1.5 at stresses below $10^{-5}G$, where $G$ is the temperature compensated shear modulus; Interestingly, this is the typical stress value at which creep behavior transitions to the Harper-Dorn creep in other materials also (see **Fig. 4**). Furthermore, the dislocation density in the Harper-Dorn creep regime in LiF remains independent of the applied stress, with a value only slightly higher than the saturation dislocation density measured under static annealing conditions (see **Fig. 5**). A re-examination of the creep data of NaCl also reveals a creep exponent of ~ 1.5 in the Harper-Dorn creep regime. Consistent with LiF, NaCl also showed stress independent dislocation density in the Harper-Dorn creep regime. Previously, the creep stress exponent of Al has been reported to range from 1 to 3. Hence, it appears that observation of a creep stress exponent of 1 in the Harper-Dorn regime is rather a coincidence than a universal fact.
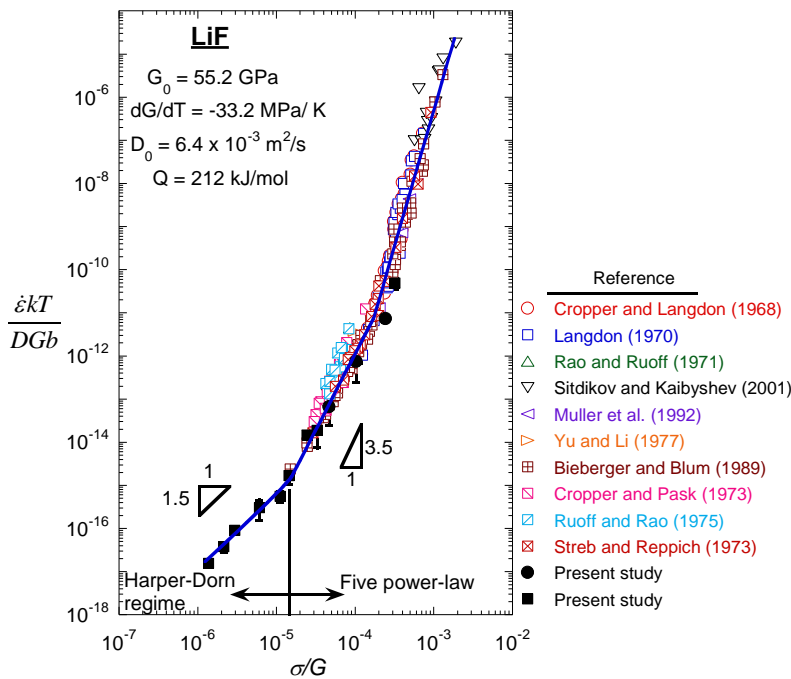


Fig. 4: A normalized stress versus normalized steady-state strain-rate plot for LiF. The solid line, whose slopes are shown by triangles next to them, are the best fit curves. The vertical solid line, corresponding to a stress of $10^{-5}G$, demarcates the boundary between the "five"-power law and the Harper-Dorn creep regimes.
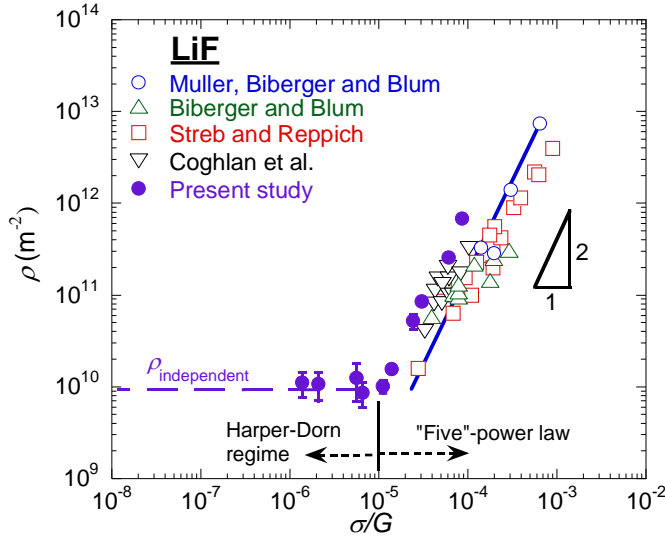
Fig. 5: A composite plot showing variation of the steady-state dislocation density as function of the normalized stress. The solid line, whose slope is shown by triangle next to it, is the best fit curve for the data in the "five"-power law. The vertical solid line, corresponding to a stress of $10^{-5}G$, demarcates the boundary between the "five"-power law and the Harper-Dorn creep regimes.

The typical constant dislocation density observed in the Harper-Dorn creep regime may be a consequence of "frustration" of the dislocation Frank dislocation network. Frustration means the minimum saturated dislocation density below which the dislocations are unable to recover. The implications of the initial and the frustration dislocation density on the observation of a creep exponent of $> 1$ in the Harper-Dorn creep regime is discussed in context of LiF, NaCl and Al, using a dislocation-network based model. If the initial dislocation density is *lower* than the frustration value, then a creep stress exponent of 3 would be observed in the Harper-Dorn regime (see **Fig. 6a**). On the other hand, if the initial dislocation is larger than the frustration dislocation density then the creep stress exponent of 1 to 2 and a stress independent dislocation density will be observed in the Harper-Dorn creep regime (see **Fig. 6b**). The developed dislocation-network based model interprets the creep response in both the Harper-Dorn and "five"-power law creep regimes and justifies most of the earlier observations made in the Harper-Dorn creep regime. One of the major implications inferred from such results could be a non-linear dependence of the dislocation climb velocity on the applied stress at such high temperatures in pure systems.
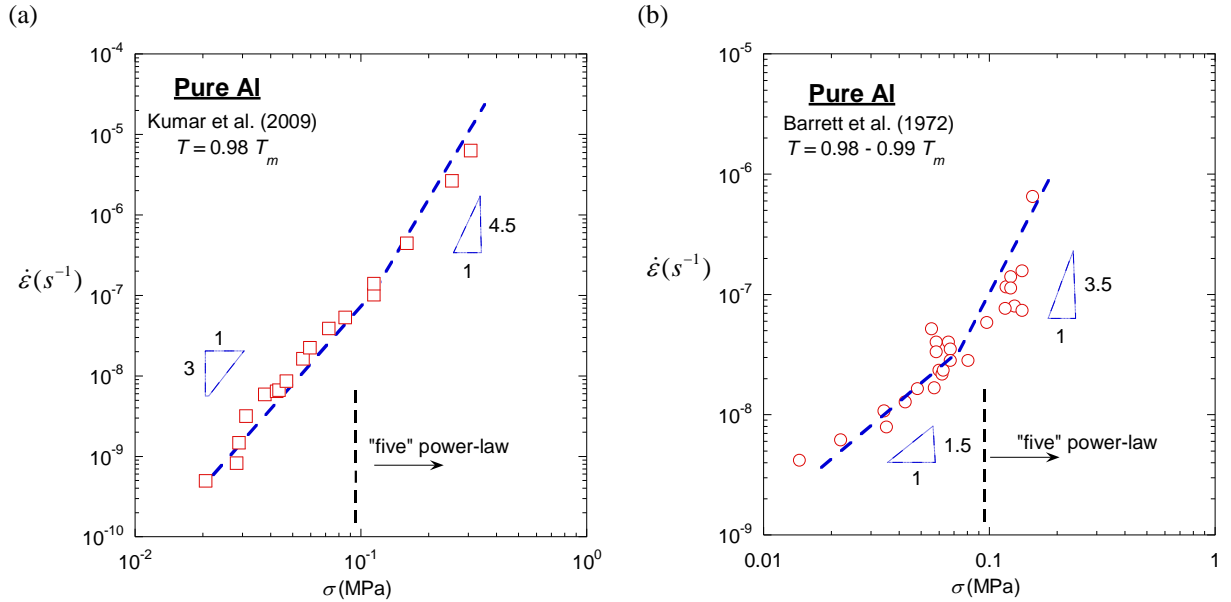
Fig. 6: (a) Stress versus the steady-state strain rate from the earlier study by Kumar et al. illustrating a change in the creep exponent from 4.5 to 3 with transition of creep regime from "five"-power law to Harper-Dorn creep. (b) Creep plot showing transition of the creep exponent from 3.5 to 1.5 in the Harper-Dorn creep regime. The dashed line, in both (a) and (b), represents the best fit of the data and the slope is shown by the triangles next to this line which is the creep exponent. The dislocation density was observed to depend on the applied stress in the Harper-Dorn creep regime in (a), whereas it was independent of the applied stress in (b). The case (b) is similar to the observations of the present study on LiF which is shown in Fig. 4 and Fig. 5.