Kinetics of Dynamic and Static Recovery During High-Temperature Deformation and Annealing

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Abstract

Published data on the distributions of dislocation link lengths generated during the steady-state power-law creep of monocrystalline NaCl and Harper-Dorn creep of monocrystalline Al are analyzed to obtain information on the equation governing the growth rates of individual links in the network. It is shown that the rate of growth, g(L), of an individual link of length L is adequately described by the equation

$$g(L) = \frac{\overline{K}}{L^{m}} \left(\frac{L}{L^{*}} - 1 \right).$$

where K is a rate constant, L* is a critical link length and m (> 1) is a constant. This equation is not valid over the entire range of link lengths, but becomes increasingly accurate for small values of L. For NaCl m = 1.387 ± 0.046 for the five sets of data analyzed, while for A1 m = 1.533 ± 0.059 for three sets of data. It is argued that this equation should be valid for static recovery by the process of network coarsening, and leads naturally to an equation describing the variation of the dislocation density, ρ , with time, t, of the type $\rho^{-n} \propto t$, where n is an exponent related to m as n = (m + 1)/2. This recovery equation differs from those proposed previously in that $n \neq 1$. It is tested by regression analysis of two sets of published data on static recovery, one on previously crept samples of an austenitic stainless steel and the other on single crystals of iron deformed 9.8% by cold-rolling. The value of n for the austenitic steel is 1.311 (m = 1.623), while that for the iron samples is n $= 1.210 \pm 0.117$ (m = 1.421 ± 0.233). Despite some uncertainties, including those in existing network theories of deformation which are discussed, the results suggest that recovery during creep and static recovery share much in common, and are different manifestations of the same fundamental process.

1. Introduction

The dislocation-network model of creep was recognized as potentially important in the mid-1960's by Mitra and McLean /1/ and quantified in later years by Öström and Lagneborg /2/ and Ardell and Przystupa /3/. It provides the most realistic and self-consistent picture of the elevated-temperature deformation of simple metals and alloys of all the models that have been proposed to

this date. Its principal asset is that it breathes life, on the microstructural level, into the established idea that creep of a crystalline solid is a competition between hardening and recovery. In the framework of the dislocation network model, hardening is a consequence of the interactions between the dislocations that glide freely under the influence of an applied stress and the nearly immobile dislocations in the network. The process is assumed to be statistical in nature and driven by an unlimited supply of suitable dislocations which interact either by collisions or as a result of shrinking of some of the network links to zero length. The primary interactions are collisions, which tend to refine the network. Recovery comes about by coarsening of the network, and steadystate creep obtains when the processes of network refinement and coarsening achieve dynamic equilibrium.

The attractive features of the dislocation network model are numerous. It is entirely consistent with observed creep microstructures in pure metals, and in many alloys and ceramics as well. In these materials a dislocation network is ubiquitous, whether or not subgrains are also present. The model accounts in a natural way for primary creep behavior and the principal findings of stress-drop tests without the need to invoke the troublesome implications of internal stresses, some of which are discussed in the recent paper by Ajaja /4/. The model consigns subgrain boundaries to their rightful role as low-energy dislocation structures that provide the final resting place for the dislocations that run into them. The mental gymnastics required to explain why, for example, normal primary creep is observed irrespective of whether or not subgrains form, even in the same material (e.g. Fe-Si alloys /5/ and pure MgO /6,7/ are thereby eliminated. The model leads naturally to the idea that there should be such a phenomenon as Harper-Dorn /8/ creep, the kinetics of which are controlled entirely by recovery of the dislocation network /9/.

Despite its attractive features, the dislocation network theory has not been widely accepted. This is due in part to its relatively late arrival as a quantitative theory, at which time conventional wisdom regarding the roles of subgrains and internal stress was so entrenched that new ideas eschewing their importance were easier to ignore than to address. Additionally, the extant quantitative theories contain parameters which are difficult to

estimate, do not appear in other problems, and so are totally unfamiliar. A good example of this in the theory of Ardell and Przystupa /3/ is the collision rate between gliding dislocations and dislocation links in the network, M, which is a state variable. Since M depends on the velocities of the gliding links, hence on the stress, and also on the probabilities of encounters between links of various lengths, thus on the network geometry, it is extremely difficult to estimate.

Another problem with the theories is that there is no fundamental quantitative understanding of network coarsening, which is the cornerstone of recovery during creep. At issue here are mathematically expedient static recovery laws which are commonly used, but which have never been thoroughly tested against the available network coarsening data. The predictive capabilities of these laws must be tested before they can be used with confidence. The principal objective of this paper is to provide some insight into these issues.

2. Background

One of the earliest suggestions regarding a kinetic law governing static recovery of the dislocation network was offered by Lagneborg et al. /10/, who proposed the use of the equation

$$\frac{dL}{dt} \equiv g(L,t) = \overline{K}_1 \left(\frac{1}{L^*} - \frac{1}{L} \right) = \frac{K_1}{L} \left(\frac{L}{L^*} - 1 \right), \quad (1)$$

where g(L,t) is the instantaneous rate of growth of an individual link of length L in the network, K_1 is a temperature-dependent constant and L* is a critical link length, the physical significance of which is that links in the network with length exceeding L* grow while those with length smaller than L* shrink. The seeds of Eq. 1 were sown by Lagneborg /11/, who suggested that the kinetics of network coarsening were analogous to those of grain growth. If L is replaced by the grain diameter R in Eq. 1, the theory of Hillert /12/ leads to a law for grain growth of the form $< R > 2 \propto t$. The same mathematics lead to the kinetic law for the growth of a link of average length < L > given by

$$\langle L \rangle^2 - \langle L_0 \rangle^2 = K_2 t, \qquad (2)$$

where <L_o> is the average initial link length and K₂ is another constant. Since the density of dislocations in the network, ρ , is related to <L> by <L> \propto $1/\rho^{1/2}$, the dislocation density should decrease during static recovery according to the equation

$$\rho^{-1} - \rho_{0}^{-1} = K_{3}t, \tag{3}$$

where ρ_0 is the initial dislocation density and K_3 is another constant. Eq. 3 was first derived by Li /13/ using a different approach involving the rate of annihilation of excess dislocations of one sign. Li presented some unpublished data of Sankaran and Li on static recovery in LiF which suggested that Eq. 3 was accurately obeyed.

Eq. 1 predicts that $g(L,t) \to L^{-1}$ as $L \to 0$. However, Lin et al. /14/ have shown recently that this prediction is not consistent with the results of creep experiments on monocrystalline NaCl and Al. Instead, g(L,t) approaches L^m , where $m \approx 3/2$ for NaCl and $\approx 4/3$ for Al. This finding arises from consideration of the limiting behavior of the dislocation link-length distribution, which approaches L^m as $L \to 0$. The implication of this result is that Eq. 1 should be replaced by a more general equation of the form

$$g(L) = \frac{K}{L^m} \left(\frac{L}{L^*} - 1 \right). \tag{4}$$

where K is another constant. It is not difficult to show that the application of the procedures in Hillert's theory /12/ leads to a static recovery law for the dislocation density of the type

$$\rho^{-n} - \rho_0^{-n} = \kappa t, \qquad (5)$$

where κ is yet another constant and m and n are related by

$$n = \frac{m+1}{2}.$$
 (6)

Eq. 1 may be regarded as a special case of Eq. 4, with m = 1. Just as Kocks and Mecking /15/ have questioned the validity of Eq. 1 in the case of dislocation network coarsening, we may also question that of Eq. 4; there is no firm theoretical foundation for either one. Nevertheless, we may evaluate Eq. 4 in an empirical sense to see if it is consistent with observed behavior. With this objective in mind, the present paper examines two distinctly different types of sets of data, one on the

link-length distributions generated during the steady-state power-law creep of monocrystalline NaCl /14,16/ and the steady-state Harper-Dorn creep of monocrystalline Al /9,17/, and the other on static recovery in two different types of steels.

3. Analysis of Data

A. Steady-State Creep

If data on the distributions of dislocation link lengths are available it is possible to obtain the function g(L) experimentally (the dependence on t vanishes during steady state creep). This is done by numerical integration of functions involving the dislocation link-length distribution. The details of the integrations differ depending on whether creep occurs in the power-law or Harper-Dorn regimes because the equations of the dislocation network theory differ for these two regimes. Also, the integrations are easiest to perform for steady-state creep behavior. The necessary steps have been published for Harper-Dorn creep /17/; for power-law creep they have been derived by Lin /18/. The numerical integrations directly yield the product $C\phi(L)g(L)$, where $\phi(L)dL$ is the number of links with length between L and L + dL (at steady-state this function is also independent of time) and C is a constant (equal to unity for Harper-Dorn creep but not for power-law creep). Once the product $C\phi(L)g(L)$ is generated, Cg(L) can be obtained simply by dividing the product by the experimentally measured values of $\phi(L)$.

After g(L) is calculated, a procedure must be adopted to estimate the value of m that best characterizes the function. The procedure used here was to choose a value of m and plot the product CLmg(L) vs. L, which according to Eq. 4 should produce a straight line of slope K/L* and intercept -K. This was repeated for several values of m, and the best value of m was determined by regression analysis of the linear plot, which yields a value of the goodness of fit of the data, R². The error estimate, R², was then plotted against m and fitted to a cubic equation. The value of m producing the maximum value of R² was chosen as the best value. (In principle, the self-consistency requirement that the values of K determined from the slope and intercept of the linear plot should be identical can also be used as a criterion, but L* cannot be measured independently).

Harper-Dorn Creep of Al

The data analyzed were generated at applied stresses of 0.05, 0.06 and 0.08 MPa for monocrystalline Al tested at 920 K. Under these test conditions the creep of Al is well within the Harper-Dorn regime /9,17/. The steady-state link-length distributions $\phi(L)$ are shown in Fig. 1a, and a trial function $L^m g(L)$ with m = 4/3 is plotted vs. L in Fig. 1b. This value of m was chosen as

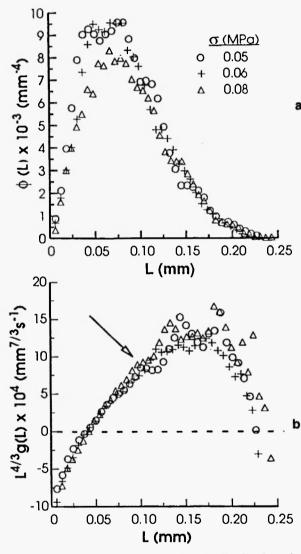


Fig. 1: (a) Dislocation link-length distributions in monocrystalline Al deformed at 920 K in compression in the Harper-Dorn creep regime to steady state at the stresses indicated; (b) Illustrating the behavior of the trial function L^{4/3}g(L) for the same sets of data.

representative of the results of the previous analysis /14/ of the limiting behavior of $\phi(L)$. The plots in Fig. 1b exhibit slightly negative curvature for links up to roughly 0.1 μ m. For links longer than this the fit is not good and the scatter is quite large. To determine the best value of m it was necessary to limit the data analyzed. The range chosen included only those link lengths smaller than that indicated by the arrow in Fig. 1b, and for some of the stresses the smallest two links were also excluded.

An example of the regression analysis for $\sigma=0.05$ MPa is illustrated in Fig. 2. It is seen that while the values of R^2 are all close to unity, the curve of R^2 vs. m passes through a sharp maximum, clearly indicating that one value of m, in this case 1.493, best represents the data. The curves of $L^mg(L)$ vs. L for the three stresses and values of m are shown in Fig. 3. The linearity displayed in Fig. 3 is excellent, and while the three values of m are not identical, the evidence suggests that m is independent of the applied stress. The value of m resulting from this analysis, 1.533 ± 0.059 , is greater than 1.387 ± 0.110 , obtained from the previous analysis of Lin et al. /14/.

Power-Law Creep of NaCl

Analysis of the data on the steady-state creep of NaCl is somewhat more difficult than that on Al because the link-length distributions have a long tail, extending into the so-called "glide" regime, i.e. the part of the

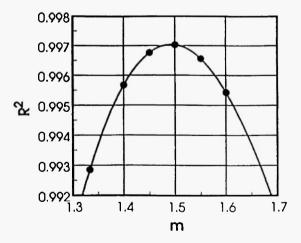


Fig. 2: Plot of R^2 vs. m for the data on Al at $\sigma = 0.05$ MPa.

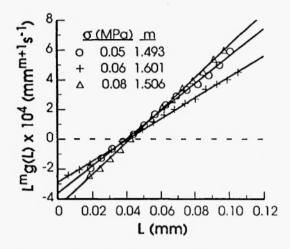


Fig. 3: Curves of L^mg(L) vs. L for the data on Al using the indicated values of m.

distribution where the links are no longer required to remain in equilibrium with their line tension /17/. This region is characterized by a special link length, L_c, which is inversely proportional to the applied stress /3/. However, g(L) is defined only for that part of the distribution in which the links can change their length by coarsening, hence only the part of the distribution where $L < L_c$ is amenable to numerical integration. The problems here are computational, not conceptual, hence are not difficult to overcome. The data analyzed were for the steady-state creep of NaCl obtained under the test conditions 0.39, 0.59 and 0.78 MPa at 923 K and 0.39 and 0.59 at 973 K /14/. The steady-state link-length distribution, as well as the trial function CLmg(L) with m = 1.5 /14/ are shown plotted vs. L for $\sigma = 0.39$ MPa, T = 923 K in Fig. 4. The behavior of CL^mg(L) for NaCl is different from that of Al in that the deviation at large values of L is in the opposite sense. This behavior is representative of the entire set of data, which suggests that Eq. 4 does not adequately describe the data over the entire range $0 > L > L_c$.

A fit was attempted nevertheless at small values of L. The principal difficulty encountered was in choosing a cut-off, and it was found by trial and error that extending L to several points beyond $L = L^*$ produced reasonable and consistent results. This may appear to be overly restrictive, but $L = L^*$ occurs beyond the peak value of $\phi(L)$, so that at least half of the network-recovery region of the link-length distribution is sam-

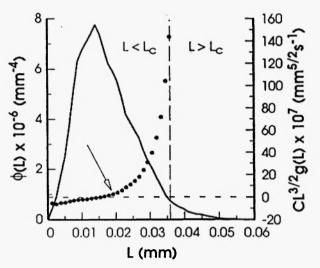


Fig. 4: Illustrating the dislocation link-length distribution observed during power-law creep of monocrystalline NaCl at $\sigma = 0.39$ MPa and 923 K. The function $CL^{3/2}g(L)$ derived from the data is also shown. The dashed vertical line indicates $L = L_c$ and the arrow indicates the maximum value of L used in the subsequent analysis.

pled. The relationship between R^2 and m for the five creep conditions is similar to that seen in Fig. 2, and the function $CL^mg(L)$ is shown plotted against L in Fig. 5, where the individual values of m are also indicated. It is evident that the linearity is very good over the range of L used in the analysis. The average value of m was found to be 1.387 ± 0.046 , compared to 1.480 ± 0.108 from the earlier analysis /14/, with no systematic dependence on σ or T.

B. Kinetics of Static Recovery

Data on the kinetics of static recovery were analyzed because there is no reason why Eq. 4 should not be valid for this process. The experimental evidence from the creep studies indicates that m is independent of the applied stress and temperature, which implies that Eq. 4 should hold when $\sigma=0$, as well. The difficulty in testing the validity of Eq. 4 for static recovery is the paucity of data. However, two investigations have produced data which are readily amenable to analysis. One is a study by Lee et al. /19/ of recovery in pure, cold-rolled mono-

crystalline iron by positron annihilation, and the other is a study by Odén et al. /20/ of static recovery of the dislocation network structure produced by creep deformation of an austenitic stainless steel. There are other data available, specifically those of Sankaran and Li /13/ on recovery in lightly deformed monocrystalline LiF, Keh /21/ on recovery in pure polycrystalline α-Fe deformed 16% in tension, and Hausselt and Blum /22/ on recovery in an Al-11 wt.% Zn alloy deformed in steady-state creep at three different stresses. The data of Sankaran and Li /13/ and Keh /21/ were analyzed, but the results will be mentioned only briefly herein. It was felt that the data of Hausselt and Blum /22/ could not be extracted from their published figures with enough accuracy to be analyzed reliably.

Lee et al. /19/ reported their data in the form ρ^{-1} vs. t, and the values of these quantities were taken from their published figures.* The data of Odén et al. /20/ were read directly from Fig. 3 of their paper. The data were then analyzed by plotting ρ^{-n} vs. t, in accordance with the predictions of Eq. 5, for various values of n and the linearity tested for goodness of fit, as for the analysis of the data on creep. It was found that R^2 passed through a sharp maximum as a function of n, and the value of n chosen for a given set of data was that yielding the maximum value of R^2 .

An example of this procedure applied to the data of Lee et al. /19/ for the annealing temperature of 679 K is shown in Fig. 6. The best fit is attained for n = 1.173, which implies m = 1.346 according to Eq. 6. The values of n and m for the other two temperatures investigated by Lee et al. are summarized in Table 1, and their data are displayed graphically in Fig. 7. The accuracy of the analysis is limited by the accuracy with which published figures can be read, but it was found that varying the input data within reasonable limits does not alter the finding that n, hence m, exceeds unity.

The published data of Oden et al. /20/ are more amenable to analysis because they reported their dislocation densities, hence obviating the need to extract them

The ordinates in the relevant figures of Lee et al. are missing the indications of the order of magnitudes for the reported values of ρ^{-1} . This, however, has no influence on the values of n obtained from the analysis.

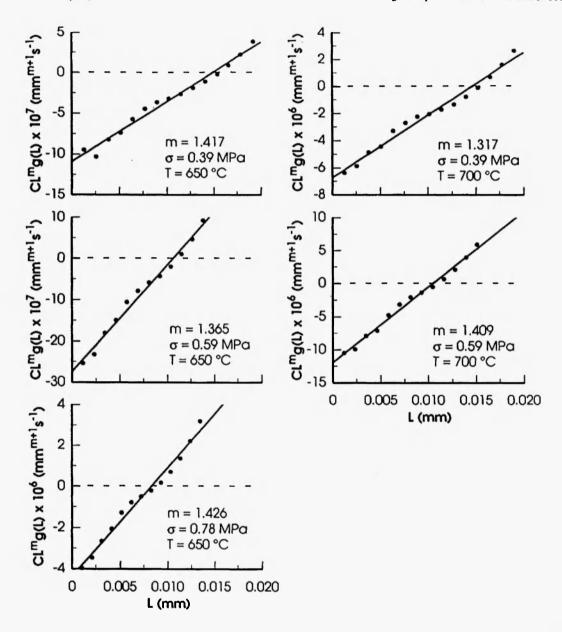


Fig. 5: Illustrating the linearity of the function $CL^mg(L)$ with L for the data on NaCl tested under the conditions indicated. The values of m obtained from the analyses are also shown.

Table 1

Values of n and m from the analysis of the data of Lee et al. /19/.

T (K)	n	m
373	1.117	1.234
518	1.341	1.682
679	1.173	1.346

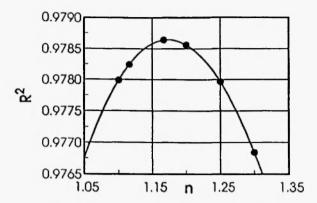


Fig. 6: Illustrating the dependence of R² on n for the data of Lee et al. /19/ at 679 K.

from their published figure. The analysis of their data is complicated, however, by the observation that ρ did not decrease indefinitely with annealing time, but asymptotically approached a limiting value. Some of the data of Hausselt and Blum /22/ appear to behave in an identical manner. Odén et al. were not able to explain this finding, but it will be shown later that a dislocation network cannot coarsen indefinitely because the possible combinations of Burgers vectors of the dislocations at the nodes of the network, which must sum to zero at any given node, eventually become exhausted. That a limiting value of ρ was found by Oden et al. is, therefore, perfectly normal (and might have eventually been found by Lee et al. as well had their experiments been extended to longer annealing times).

Of the eight data points reported by Odén et al., only the first five were included in the analysis to find the best value of n. The procedure used was identical to that for the data of Lee et al. The best value of n is 1.311 (m = 1.62). The variation of $\rho^{-1.311}$ with t is shown in Fig. 8, in which one extra datum (for t = 200 h) is also shown. The linearity displayed by the first five data points is excellent.

4. Discussion

One of the major objectives of this paper was to explore the possibility that dislocation network recovery during creep is fundamentally the same process as static recovery of the network that obtains during annealing. This has been accomplished by demonstrating that in all

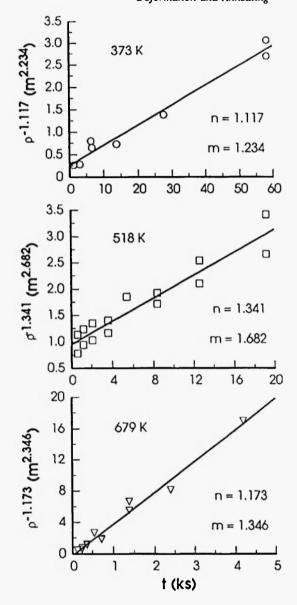


Fig. 7: The data of Lee et al. /19/ plotted in the form ρ^{-n} vs. t, using the values of n indicated in the individual figures.

likelihood the same fundamental equations govern both processes. In the case of creep, the basic relationship is Eq. 4; for static recovery, it is Eq. 5, which can be derived from Eq. 4 using the formalisms of the theory of coarsening. The important consequence is that neither m nor n is equal to unity, as predicted in earlier theories.

In evaluating the significance of the analyses presented herein it is important to consider their limitations. The principal limitation of the analyses of the

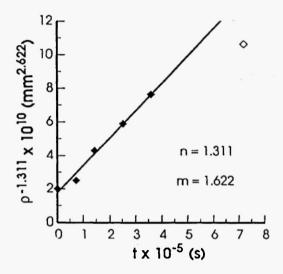


Fig. 8: Illustrating the linearity of the plot of ρ^{-1.311} with t for the data of Odén et al. /20/. The open symbol represents a datum at t = 200 h, which was not used in the analysis.

data on the creep of NaCl and Al is their reliance on theory to estimate the appropriate values of m. Specifically, the "experimental" function g(L) can only be obtained from the measured $\phi(L)$ by performing numerical integrations. These, in turn, rely on the theoretical equations derived by Ardell and Lee /17/ or Lin /18/ from the original equations of Ardell and Przystupa /3/. Therefore, if there is something amiss theoretically the derived functions can be expected to differ from those calculated and used to estimate m.

In fact, there are certain common assumptions regarding the geometry of a three-dimensional dislocation network that are not correct. The first of these is the so-called "constant volume" condition which leads directly to the relation between <L> and ρ. This condition assumes that the dislocation links in the network are equivalent to the edges of space-filling polyhedra. Filling space in this way requires, on average, that four edges meet at each corner. If the dislocation network were geometrically equivalent, we would expect that four dislocation links would meet at each node (corner) in the network. Frank's rule requires that the sum of the Burgers vectors at each node be equal to zero. Since the minimum number of Burgers vectors needed to fulfill

Frank's rule is three, we expect that only three dislocation links will generally meet at a node in the network. But this cannot satisfy the requirements of filling space, which means that the links in the dislocation network are not equivalent to the edges of an assembly of space-filling polyhedra. Hence the very concept of the constant volume condition loses its significance, and the relationship <L $> \propto \rho^{-1/2}$ owes its validity to a purely dimensional argument.

Frank's rule is also the key to a check on the selfconsistency of all the possible dislocation reactions assumed by the network theories. This is best illustrated within the framework of static recovery, i.e. pure network coarsening, where dislocation sources are inactive and the collision rates are nil, since the applied stress is zero. During coarsening certain links must shrink to zero length and disappear from the network. This process clearly depends on the availability of links that can physically contract, i.e. links connected to nodes at which the other dislocations have just the right Burgers vectors. A reaction involving the disappearance of a dislocation link is the exact opposite of the reaction of two dislocations that interact to produce an attractive junction /23/. Clearly, the supply of such links is bound to be exhausted, hence the process must stop, resulting in a stable configuration of links which cannot be annealed out. This is exactly the behavior observed by Odén et al. /20/ and Hausselt and Blum /22/. Existing network theories assume an unlimited supply of links that can shrink to zero, thus they cannot possibly account for the residual dislocation densities actually observed.

The opposite situation exists when the conditions are conducive for links to collide, i.e. under an applied stress. All the existing theories including ours /3/ are based on purely geometrical considerations in which it is assumed that colliding links join at the collision point and form a four-fold node, with no instantaneous change of the total length of the links involved. However, when the Burgers vectors are taken into account, a stable four-fold node is a remote possibility compared to the formation of jogs or creation of an attractive junction. These physically more appealing processes are not accounted for in the theory. It can, therefore, be expected that when these aspects of the problem are dealt with correctly, certain terms in the basic equations of the

theory will change.

Even given these qualifications, it does not seem likely that Eq. 4 can possibly describe the correct behavior of g(L) over the entire range of L ($0 < L < L_c$). However, many functions of the scaled link length $u = L/L^*$ other than Eq. 4 can also produce the limiting behavior $g(L) \to L^{-m}$ as $L \to 0$; for example, multiplying Eq. 4 by any arbitrary function of $1 \pm u$ would work. It is expected that advances in the theory will enable a search for the appropriate function to succeed. Whatever the form of this equation turns out to be, it is not expected to change Eq. 5, which would still describe the kinetics of static recovery.

For these reasons we expect that the most probable values of m for NaCl and Al have yet to be determined. However, there is little doubt that the values of m must be greater than unity because those obtained by Lin et al. /14/ from analysis of the limiting behavior of $\phi(L)$ are also greater than one. These values do not depend on the theory, only on the assumption that the function g(L) exists and has the form expressed by Eq. 4. Indeed, the fact that the values of m from the analysis in this paper differ from those obtained previously is most likely due, at least in part, to inadequacies in the theory.

The results on static recovery, particularly the data of Oden et al. /20/, also indicate that m must exceed unity. The value of n obtained from analysis of the data of Odén et al. is considered the more reliable of the two on static recovery because there is no doubt that it refers to static growth of a dislocation network generated during creep of the material. In the case of the data of Lee et al. /19/ the situation is not so clear because the dislocation densities in their material were generated by

cold-rolling 9.8% and they did not publish any micrographs of the recovered microstructures. It is thus more difficult to be certain that recovery occurred by network coarsening rather than a competitive process such as subgrain formation and growth. Interestingly, even for the data of Sankaran and Li /13/, which appear to obey Eq. 3 so convincingly, the value n = 1.033 (m = 1.065) provides the best fit (the data of Keh /21/ require a value of n far in excess of unity, but the number of data points is limited).

It is also necessary to be rather cautious in the interpretation of the recovery data because the differences between the results of the regression analyses for n=1 and n>1 are not very large. These differences are summarized in Table 2, where it is seen that the differences between the values of \mathbb{R}^2 for n=1 and that yielding the best fit with the data are generally less than 0.004, indicating that the fit for n=1 is not at all bad. If the goodness of fit had been equally divided for n<1 and n>1 the principal conclusion of this study would be far less convincing. However, since n>1 in all cases, the results appear to be significant, and suggest that network coarsening is common to both static recovery and recovery during steady-state creep.

5. Conclusions

Coarsening of the dislocation networks that are created during the Harper-Dorn creep of monocrystalline Al and power-law creep of NaCl is governed by Eq. 4, with m > 1, in the limit of small link lengths. This equation leads to a static recovery law given by Eq. 5, wherein the dislocation density decreases with time according to $\rho^{-n} \propto t$, with n = (m + 1)/2 > 1. The

Table 2 Comparison of the values of R^2 obtained from analysis of the data on static recovery for the optimum values of n (= n_{opt}) with those obtained for n = 1.

nopt	$R^{2} (n = 1)$	$R^2 (n = n_{opt})$	Ref.
1.117	0.9759	0.9768	19
1.341	0.9082	0.9094	19
1.173	0.9749	0.9787	19
1.311	0.9865	0.9880	20

similarity of the values of m that obtain during steady state creep and static recovery lends credence to the suggestion that the phenomena of coarsening of the dislocation network during creep and coarsening during static recovery are the same.

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