Strain Hardening at Large Strains in Aluminum Alloys

Los Alamos

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LA-11202-T
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*Contact NTIS for a price quote.
Strain Hardening at Large Strains
in Aluminum Alloys

Anthony David Rollett
ACKNOWLEDGEMENTS

The advice and criticism of Roger Doherty and Fred Kocks was essential to the work. Many others contributed heavily: Manuel Lovato to the mechanical tests, Michael Stout to the experimental program and data reduction, David Embury to a critical appraisal of the literature, for example. Thanks is due to the Aluminum Company of America (ALCOA) for providing all the materials and alloys used in this work. The support of the Los Alamos National Laboratory for Advanced Study Leave is also gratefully acknowledged.
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4.10 a) Commercial purity Al twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%.

4.11 a) Al-1Mg twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%.

4.12 a) Al-2Mg twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%.

4.13 a) Al-0.8Mn twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%. Note the lack of slip markings in either image.

4.14 a) Al-4.6Cu twisted to a shear strain of 1.6, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%. Note the lack of slip markings in either image.

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   b) commercial purity Al, (i) (111), (ii) (200)
   c) Al-0.8Mn, (i) (111), (ii) (200)
   d) Al-1Mg, (i) (111), (ii) (200)
   e) Al-2Mg, (i) (111), (ii) (200)
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   d) (200) pole figure calculated with LAPP code.

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   b) as a), (200) pole figure.
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   d) as c), (200) pole figure.

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7.5 Reduced hardening rate plotted versus reduced flow stress for commercial purity aluminum, data taken from Alberdi [7.9],deformed at 373K, 293K, 198K and 77K in order of ascending flow stress.

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7.7 Reduced hardening rate plotted versus reduced flow stress for copper deformed at 473K, 373K, 293K, 198K and 77K in torsion, data from Alberdi [7.9].
Strain Hardening at Large Strains in Aluminum Alloys

by

Anthony David Rollett

ABSTRACT

The strain hardening properties of high purity Al, commercial purity Al, Al-Mn, Al-Mg and Al-Cu alloys have been investigated at large strains in the temperature range 77-473K. Torsion of short thin walled cylinders was chosen as the deformation method because large strains can be achieved without such problems as friction and redundant work. The results of the tests indicated that the stage of strain hardening known as Stage IV occurs even in pure face centered metals in torsion. The term Stage IV is derived from studies of single crystal plasticity and is the nearly constant hardening rate observed at large strains in many metals. The hardening rate in Stage IV is approximately $2 \times 10^{-4}$ where the hardening rate has been reduced by the shear modulus and a Taylor factor in order to put it in terms of critical resolved shear stresses on a single slip system. A plot of hardening rate versus stress characteristically shows a sharp transition from steadily decreasing hardening rate in Stage III to constant Stage IV. Stage IV does not appear at high homologous temperatures. Alloying generally extends the occurrence of Stage IV to higher temperatures than in pure metals. A model has been developed that is based on the accumulation of dislocation debris such as dipoles and prismatic loops. This model is
able to account for the characteristics of Stage IV, such as the sharp transition observed on a plot of hardening rate versus stress.
Chapter I. Work Hardening at Large Strains

1.1 Introduction

1.1a Large-Strain Plasticity

The study of plasticity in metals has been pursued for many years in an effort to understand the phenomenon of irreversible shape change. This thesis represents an attempt to advance the understanding of events at large strains which is to say extreme changes in shape. One of the most basic metallurgical properties of a piece of metal is the flow stress required to achieve plastic flow. If a metal is deformed at a temperature that is less than one half of its melting point, this flow stress increases markedly as straining, or "working", proceeds. The rate at which the flow stress increases, known as the work hardening rate, decreases monotonically with increasing stress. (The terms "work hardening rate", "hardening rate" and "strain hardening rate" are interchangeable in this thesis.)

The work hardening rate, $\dot{\theta}$, $d\sigma/d\gamma$, often remains finite (and positive) to very large strains ($>1$). This behavior is not apparent at small to intermediate strains where the hardening rate falls smoothly with increasing stress in such a manner as to suggest that the flow stress will saturate at a finite strain. The principal aim of the work presented below has been to measure hardening rates at large strains over a range of homologous temperature and strain rate and to devise a physically based model for large-strain work hardening. The model predicts, for example, that all f.c.c. metals should exhibit a finite hardening rate at large strains, if deformed at a low enough temperature.
I.1b Motivation for Thesis

Comprehensive reviews are available for large-strain behavior of metals by Gil-Sevillano et al. [1.1] and Hecker and Stout [1.2]. These reviews concentrated on the data that were available and such specific topics as texture development. One of their main conclusions was that adequate models for large-strain work hardening were not available. The new models of large-strain hardening developed in this thesis were motivated by the conclusion that current models of large-strain hardening [1.3,1.4] do not give a good description of either the onset of what has come to be called Stage IV, or the work hardening rates in this regime, see Chapter II. This thesis concludes that two different types of model are required, depending on whether the material in question is dominated by single-phase behavior or by two-phase behavior. Single phase materials can be described by a model based on the accumulation of dislocation debris. Two phase materials can be described by a model for the dislocation mean free path being restricted by the second phase particles. A discussion of the applicability of these models to the experimental results is given in Chapter VII.

Although this thesis focuses on metals, it should be noted that Stage IV behavior has been found in non-metallic materials such as Ge and Si, Brion et al. [1.5].

I.1c Objectives

The objectives of this thesis are these: one, to add to the limited body of experimental data for stress-strain behavior at large strains; two, to develop a model for work hardening at large strains, commonly referred to as Stage IV work hardening; three, to explore the effectiveness of certain strengthening mechanisms at large strains, e.g.
solute hardening and precipitation hardening. The utility of these objectives relates to both constitutive laws for metals and to practical metal working processes that involve large strains, such as wire drawing, rolling, forging, etc.

This thesis explores large-strain work hardening as a function of three variables, temperature, strain rate and alloy. All three are essential to the development of a model for work hardening. The material class of choice is face centered cubic (f.c.c.) metals because work hardening at small to intermediate strains is well researched in this material class and successful models are available, e.g. Mecking and Kocks [1.6]. In this material class, aluminum, copper and nickel have been the subject of recent doctoral theses by Alberdi [1.7] and Hughes [1.8]. Aluminum and its alloys were chosen for this thesis as complementing the previous studies. Also aluminum is known to deform by dislocation motion only which avoids the complications introduced by such deformation mechanisms as twinning and phase transformation plasticity.

Many methods are available for strengthening metals at small strains, of which precipitation strengthening is perhaps the most significant in commercial alloys. Generally high strength as measured by the flow stress is aimed for while maintaining acceptable properties such as toughness, corrosion resistance etc. One aspect of strengthening that this thesis attempts to show is that the contribution of different mechanisms is radically altered at large strains. In particular, the point is reinforced that solutes can be as effective strengtheners as precipitates at large strains. This appears to be because the presence of solute lowers the rate of dynamic recovery during straining and
enhances the persistence of work hardening at large strains. A precipitation hardened alloy, however, does not appear to support as much work hardening at large strains.

I.1d Organization and Synopsis of Thesis

The thesis has these main chapters: review of literature, stress-strain tests, optical metallography, electron microscopy, models for large-strain work hardening and discussion of the results. Where individual points arise in the results that can be dealt with immediately, they are discussed with the results. The main discussion, however, focuses on application of the models proposed to the results obtained in this work and previous work.

The first chapter, "Work Hardening at Large Strains", sets the scene for why large-strain work hardening was chosen as a topic for investigation. Strain hardening at large strains is conveniently distinguished from small strain behavior by use of a plot of the hardening rate versus stress. On this plot, "Stage III" of work hardening, so-called from studies of single crystals, is a continuously decreasing hardening rate with increasing stress. In the low temperature limit, large-strain work hardening shows up as a horizontal line that represents a constant work hardening rate, called Stage IV. The two principal reasons for studying Stage IV work hardening were the need for more data and the lack of a model for Stage IV.

The second chapter, "Strain Hardening at Large Strains; a Review", contains a review of the existing literature on work hardening. The model for Stage IV that is presented in the sixth chapter uses the accumulation of dislocation debris as its physical basis. With this in mind, it was necessary to review a considerable amount of small-strain
behavior before considering the large-strain area. Stage I work hardening, "easy glide", appears to be dominated by the accumulation of dislocation debris in the form of dipoles. Although the debris accumulation dominates the work hardening, the major contribution to the flow stress is from forest dislocations that intersect the slip plane. Very early on in the work, this suggested that the low hardening rates observed in Stage IV might be another example of debris accumulation adding to the monopolar dislocation network developed at smaller strains.

Stage II work hardening is reviewed to demonstrate the geometrical basis for the essentially athermal and rate-insensitive hardening rate observed in this Stage. The dislocation networks that develop are shown to be heterogeneous in that dislocation tangles develop where the local dislocation density is higher than the mean density. Stage III of work hardening is where dynamic recovery of stored dislocation density occurs so that the hardening rate decreases as the flow stress rises. Dynamic recovery has been modeled on the basis of the rearrangement of dislocations into lower glide resistance configurations within the tangles. This is important for two reasons: one, the saturation flow stress that would arise in the absence of Stage IV is largely determined by the tangles; two, the dynamic recovery events in the tangles may lead to the formation of dislocation debris.

The second half of the review chapter is concerned with large-strain work hardening. If hardening rate data for different materials are normalized by the appropriate shear modulus (both hardening rate and flow stress) it appears that in the low temperature limit, Stage IV occurs at approximately the same level in all cases, viz. $\theta=2.10^{-4}G$. In
particular, Stage IV occurs in pure f.c.c. metals in all deformation paths provided a low enough deformation temperature is used. The consequence of this and much other literature is that all the explanations of Stage IV that have been proposed can be rejected.

The third chapter, "Experimental Investigation of the Stress-Strain Behavior at Large Strains in Various Aluminum Alloys", begins the description of the new work. Aluminum was chosen as a base material because plastic deformation occurs only by dislocation glide in the temperature and strain rate range of interest, i.e. 77K-473K and $\gamma = 7 \times 10^{-4} \text{ to } 7 \times 10^{-1} \text{ s}^{-1}$. Also alloying with Mg offered a range of solution strengthening without much change in stacking fault energy. This is in contrast to previous studies of alloying in Ni alloys by Hughes, for example. Torsion of short hollow tubes was used since it permits continuous tests to large strains without friction problems.

Variation of Stage IV behavior was studied as a function of alloy content and temperature. The effect of strain rate (constant strain rate during each test) was also studied more extensively than in previous investigations. This was motivated by the assumption in previous models of large-strain plasticity that some debris accumulates that could recover under diffusional control. This dependence on static recovery suggests a power law relationship between stress and strain rate at large strains that has a small exponent associated with it but this is not observed for Stage III. Qualitatively, such models predict a strain rate sensitivity of the evolution of the flow stress that increases at large strains. The question arose of whether such debris could be causing Stage IV. A significant result of the experimental tests, however, was that, in the range of temperatures and materials studied,
the strain rate sensitivity at large strains was similar to that at small strains. Only the end of Stage IV is (sometimes) more rate sensitive than at small strains.

The experimental results are summarized in Fig. 1.1. This figure shows only the locus of points where Stage III gives way to Stage IV, not differentiated stress-strain data. In the low temperature limit, the transition to Stage IV occurs at the same reduced hardening rate, no matter what the previous Stage III history was. In solute containing alloys, raising the deformation temperature or varying the solute content makes no difference to the behavior. In pure metals, however, the transition to Stage IV occurs at a decreasing hardening rate as the temperature increases. Moreover, whereas in the low temperature limit the hardening rate remains constant during Stage IV, in pure metals at raised temperature the hardening rate decreases.

The fourth chapter, "Optical Microscopy and Texture Results", documents various aspects of microstructure and texture. Scribing lines on the surface of the gauge length showed that the deformation was uniform. Slip line observations at a large strain showed that slip lines are obvious in single phase materials but not so in two-phase alloys. Reversing the sense of torsion did not change the slip line patterns significantly, suggesting that different slip planes are active during reverse deformation.
Fig. 1.1 Plot of the loci of the transition from Stage III to Stage IV work hardening. In the low temperature limit, the transition occurs at approximately $\theta = 2 \times 10^{-4} G$. As the deformation temperature is raised, the transition occurs at a smaller stress and a lower hardening rate in pure metals. In solution hardened alloys, however, the low temperature behavior is retained to higher temperatures than in the pure metals.
The texture development in monotonic torsion is as expected. After reversing the torsion to zero net strain, however, there remains a shear texture. On the other hand, the grain shape is apparently restored by the reversal of the deformation. This result suggests that there is an irreversible component of texture component. This is analogous to the work hardening behavior which is also irreversible, i.e. the alloys investigated here continued to harden on reversing the torsion.

The fifth chapter, "Transmission Electron Microscopy Results", shows how the microstructures vary with strain, alloy content and temperature of deformation. Evidence for the presence of dislocation debris in the form of loops is presented. The misorientation between subgrains is found to increase to very high levels at large strains; specifically, many high-angle boundaries can be found. This occurs without regard to alloy content or Stage IV behavior which suggests that the development of misorientation cannot be the controlling factor for Stage IV.

The sixth chapter, "Models for Stage IV Work Hardening", develops three models for Stage IV work hardening. The first model is for two-phase materials and shows that the commonly observed exponential hardening may be the result of the mean free path for dislocation storage being determined by the spacing of a hard second phase that deforms with the matrix.

The second model, for single-phase materials, explores the consequences for hardening of the accumulation of dislocation debris as a general second contribution to the flow stress. The third model, also for single-phase materials, takes the Kocks-Mecking model of Stage II and Stage III work hardening as its starting point. Dislocation debris
is taken to accumulate in the dislocation tangles as a result of dynamic recovery. The consequence of this accumulation is that the limiting stress that the material would reach if accumulation ceased (the saturation stress extrapolated from Stage III behavior), increases at the Stage IV work hardening rate. When Stage III has reduced the observable hardening rate to low levels then the debris hardening is apparent as a constant hardening rate in Stage IV.

The seventh chapter, "Discussion", discusses various aspects of large-strain hardening in the light of the models developed in the previous chapter. Two-phase materials appear to be characterized by a hardening rate in Stage IV that increases with stress. In single-phase materials, in the low temperature limit, the hardening rate is constant in Stage IV which means that the transition to Stage IV from III occurs at the same hardening rate regardless of the Stage III behavior. The model of Stage IV described in the previous chapter correctly describes this behavior. At higher temperatures in pure single materials, the hardening rate decreases which can be modeled by including the effect of dynamic recovery on debris, as discussed in chapter VI.

The thesis ends with a set of conclusions, suggestions for future work and some appendices.
1.2 References


1.2. S.S. Hecker and M.G. Stout, Deformation, Processing and Structure, ASM, St. Louis (1982).


Chapter II. Strain Hardening at Large Strains; a Review

II.1. Introduction

The topic of strain hardening at large strains is an important one for both our understanding of plasticity in metals and for practical applications of metallurgy, e.g. wire drawing, where large strains are attained. Here strain hardening is defined as the increase in flow stress observed during plastic deformation of metals. Large strains are taken to be strains greater than about 0.5 logarithmic strain in monotonic deformation. At temperatures greater than two thirds of the melting point and at conventional strain rates, metals generally exhibit a saturation flow stress after a strain that is of the order of a few per cent. This regime of high temperature plasticity (creep) will not be discussed here. A natural extension to low homologous temperatures is the expectation that a saturation stress should be observed if the material can be deformed to a large enough strain, Voce [2.1]. This tendency has been widely observed but primarily in tensile tests for which the strain rarely exceeds 0.5. Once the hardening rate has decreased to low values and at temperatures less than one third of the melting point, however, strain hardening has often been observed to persist to unexpectedly large strains. In many cases, the evolution of the strain hardening leads to an apparently linear stress-strain relationship at strains greater than one.

Langford and Cohen were among the first to show this effect clearly in a single phase material [2.2], wire-drawn iron. In the last ten years two major reviews have been published by Gil-Sevillano et al. [2.3] in 1981 and by Hecker and Stout [2.4] in 1982. Also two doctoral
theses have been published by Alberdi [2.5] and Hughes [2.6] in which this topic was the basis for their experimental programs. Much of the literature on large strains emphasizes the features that appear special such as the pronounced crystallographic textures that are developed and the macroscopic heterogeneities that tend to develop. A conclusion of this review, however, is that these phenomena are adjuncts of large strain plasticity but not necessarily the cause of the persistence of strain hardening. Instead, a more general review of strain hardening will be attempted in order to link behavior at large strains to the better characterized behavior at small strains.

II.2. The Stages of Strain Hardening

By 1956, a large amount of research into the mechanical properties of single crystals led Diehl [2.7] to divide the stress-strain curve into three stages, Fig. 2.1. Stage I occurs only during single slip in single crystals. Stage II is a linear hardening stage with a relatively high work hardening rate and occurs in both single crystals and polycrystals. This hardening behavior is best thought of as a limiting behavior at small strains since the extent to which it is observed decreases with increasing temperature and stacking fault energy.

Stage III corresponds to a steady decrease of work hardening rate and is sensitive to temperature and strain rate. The theory for Stage III discussed below assumes that a saturation stress is reached at the end of Stage III when recovery balances dislocation storage. When this discussion turns to consideration of large strain behavior, a further Stage IV is added to the list. This stage is the low level work hardening that persists in some cases to very large strains. The existence of a Stage IV in work hardening requires modification of any
theory of Stage III that predicts saturation of the flow stress at the end of Stage III.

Stage IV as discussed here is usually thought of as the small but sustained hardening that occurs at very large strains, e.g. as observed in wire-drawing of iron by Langford and Cohen [2.2], Fig. 2.2. The first reference to a Stage IV was by Kocks et al. [2.8] in 1966, who referred to it in a different sense as the stage of saturation of the flow stress that would have to be introduced when Stage III is treated as a parabolic stress-strain behavior [2.9]. The experimental evidence reviewed below strongly suggests that the persistent hardening at large strains is a real phenomenon in various metals and alloys and in various straining paths. This is the behavior to which the term Stage IV is now generally applied, [2.5,2.6]. It can only be significant, however, at low homologous temperatures in view of the large body of literature on creep plasticity which demonstrates the existence of a saturation stress under many different conditions of loading and temperature.
Fig. 2.1 The Stages of Work Hardening after Diehl [2.7], Stage I/Easy Glide, Stage II/Athermal Work Hardening and Stage III/Dynamic Recovery.
The various stages of work hardening are most clearly distinguished on a diagram of $\theta$ versus $\sigma$, where $\theta = d\sigma/d\varepsilon$, Fig. 2.3. Stage II, when present, is a constant, high, value of $\theta$ at about $G/200$ (for resolved shear stresses). Stage III can often be represented by a straight line where the hardening rate decreases linearly with stress towards a "saturation stress". Stage IV intervenes before the saturation stress is reached and is commonly thought of as another stage of constant, low $\theta$ at about $2.10^{-4}G$. 
Fig. 2.2  Stress-Strain curve for low carbon iron deformed by wire drawing and tested in tension at room temperature, after Langford and Cohen. [2.2]

Fig. 2.3  The Stages of Work Hardening for a polycrystal (no easy glide) represented on a plot of hardening rate versus flow stress.
The next section reviews the characteristics of Stages I-III in order to set the scene for the large strain regime. The review of small strain hardening is largely based on the discussion of Nabarro et al. [2.10]. The review of the intermediate strain regime follows the work of Lucke, Mecking and Kocks [2.11,2.12,2.13].

II.2a. Stage I.

II.2a.1 Easy glide

Single crystals of close packed metals will deform on a single slip system with little strain hardening to strains on the order of 0.6 when stressed along a crystallogeographic direction that is not related to the crystal symmetry. This stage of work hardening is known as easy glide, Andrade and Henderson [2.14], because large amounts of strain can accumulate without much work hardening. The order of magnitude of the work hardening rate is $2.10^{-4}G$, where $G$ is the shear modulus [2.10]. The end of Stage I is not a particularly reproducible phenomenon and is sensitive to material purity and stress raisers on the specimen surface, for example.

The terms "critical resolved shear stress" and "resolved shear strain" have particularly obvious meanings in single slip: the macroscopic stress on the material must be large enough that when the stress is resolved onto the active slip system, a critical shear stress is attained. The scalar ratio between the magnitude of the resolved shear stress and the magnitude of the applied stress is called the Schmid Factor, which is a simple function of the single crystal orientation. This same ratio connects the external strain with the shear strain on the active slip system. The shear strain, $d\gamma$, can be written
in terms of the mean number of mobile dislocations that traverse the slip plane, \( dn \), and the mean spacing of the active slip planes, \( d \)

\[
d\gamma = \frac{dn \, b}{d} \quad \text{(2.1)}
\]

II.2a.2 Stage I Hardening Rate

If each mobile dislocation could pass through the crystal without any portion of it being permanently stopped, the work hardening rate would be exactly zero. The experimental data all show a small but finite work hardening rate which means that some accumulation of dislocation line length occurs. Fourie and Murphy [2.15] showed by examination in the transmission electron microscope (TEM) that the predominant storage mechanism in Stage I is in dislocation dipoles. Parallel edge dislocations of opposite sign that are closer than \( Gb/8\pi(1-\nu)\tau \), where \( \tau \) is the current flow stress, form a stable arrangement, Gilman [2.16].

Argon and East [2.17] developed a statistical theory for Stage I work hardening based on the capture of mobile dislocation segments into dipolar and multipolar configurations. This theory produced the correct order of magnitude and sign of the strain rate sensitivity and temperature sensitivity but over-estimated the work hardening rate by a factor of about 5. It is noteworthy that the low hardening rates observed in Stage I are described with fair success by this theory that is based on the accumulation of dislocation debris.

Although Stage I is only relevant to single crystal behavior, it is worth noting that the low work hardening rate appears to be a consequence of the accumulation mechanism. That is, dislocation debris in the form of dipoles accumulates which gives a small but finite hardening rate. This hardening rate is of the same order as that observed in Stage IV. The combination of low hardening rate and debris
accumulation will be taken up again later in the thesis since the model of Stage IV developed in Chapter VI is based on debris accumulation. It is therefore necessary to understand small strain work hardening behavior in order to make progress in the large strain regime.

II.2b. Stage II / Athermal Hardening

After a certain amount of strain, single crystals exhibit a transition from a low hardening rate to a much larger rate. This transition is not well defined in terms of either stress or strain and is sensitive to chemical purity, prior handling and stress raisers such as surface blemishes, Nabarro et al. [2.10].

II.2b.1 Hardening Rate in Stage II

Stage II work hardening is characterized by a linear stress-strain curve whose slope is an appreciable fraction of the modulus. In terms of a resolved stresses and strains, the hardening rate is G/200 and varies little with either temperature or strain rate, Nabarro et al. [2.10]. For a given material and temperature, the hardening rate varies only slightly with single crystal orientation. It is important to note that this high hardening rate can occur even while a single slip system is supplying the imposed strain. That is, a single crystal can be hardening in Stage II while still deforming in single slip as demonstrated by Kocks [2.18]. Stage II also occurs in tensile tests of <111> oriented single crystals which deform on six slip systems, Kocks [2.19], in <100> single crystals which deform on eight slip systems, Hosford et al. [2.20], and in polycrystals where at least five slip systems must be active in order to accommodate the imposed strain. The significance is apparent from Burgers' vector analysis in the TEM of dislocation structures in single crystals deformed into Stage II, Steeds [2.21].
This shows surprisingly high dislocation density on systems other than the primary slip system. What appears to happen is that accumulations of dislocations on the primary slip system leads to the localized production of a critical resolved shear stress on secondary slip systems. This secondary slip presumably relaxes the stress concentration due to pile-ups on the primary slip plane.

II.2b.2 Forest Theory

The consequence of this secondary slip for the flow stress is that the dislocations produced are mostly "forest" dislocations with respect to the primary slip system. The term "forest" refers to the concept that the flow stress on a given slip plane is determined by the short range interaction of mobile dislocations on the slip plane with those static dislocations that intersect the slip plane. This theory has been extensively discussed by, e.g., Basinski [2.22,2.23]. It is supported by such experimental evidence as the flow stress in Stage I being determined by the density of dislocations that intersect the primary glide plane. That is to say, although a large rise in the density of primary dislocations occurs, the flow stress, \( \sigma \), hence the hardening rate, \( d\sigma/d\varepsilon \), is controlled by the much smaller increase in density of secondary dislocations, see e.g. Argon and Brydges [2.24]. The forest theory is also supported by experiments on latent hardening where tests of the flow stress on previously inactive slip systems show only small changes in the flow stress from that of the previously active slip system, Basinski and Basinski [2.23], see also discussion in section II.3c.5.

The fundamental relationship for flow stress is the Taylor equation [2.25],
\[ \tau = \alpha \sigma b \sqrt{\rho} \quad (2b.1) \]

Mecking and Kocks [2.26] reviewed the available data and concluded that Eq. 2b.1 holds over a wide range of flow stresses and dislocation densities. The term \( \alpha \) represents an average interaction parameter between dislocations and conceals much of the inherent complexity of detailed dislocation theory. For example, the interactions vary from entirely elastic between dislocations with perpendicular Burgers vectors, to energy storing when intersection leads to formation of a jog. Attempts have been made, Saada [2.27], to incorporate the details of dislocation interactions into a quantitative theory of work hardening but without notable success. Eq. 2b.1 illustrates a fundamental point for comparing flow stresses between materials which is that the shear modulus is the appropriate quantity to use in normalizing stresses. The Burgers vector, \( b \), is also material dependent but varies much less than the shear modulus.

II.2b.3 Models for Stage II

The work hardening rate in Stage II is essentially constant. Kuhlmann-Wilsdorf developed a theory for this Stage which assumed that the line length of dislocation stored per unit strain is proportional to the reciprocal of the mean free path between existing dislocations, Kuhlmann-Wilsdorf [2.28],

\[ \frac{d\rho}{d\gamma} = k \sqrt{\rho} \quad (2b.2) \]

Given that "self-similarity" holds in the structure, meaning that the constant \( k \) in Eq. 2b.2 is a purely geometrical factor, the above equations can be combined to show that

\[ \frac{d\tau}{d\gamma} = \frac{G}{200} \quad (2b.3) \]
The factor of 1/200 was the result of arbitrary selection of geometrical constants. Kuhlmann-Wilsdorf has attempted to extend the mesh-length theory [2.29] to Stage III and even beyond. She assumed, however, that the cell size, which determines the mesh length, is essentially static in Stage III. This assumption is not well founded experimentally as the literature indicates that the cell size varies inversely with the stress or even faster, see e.g. Thompson's review [2.30]. When strained metals are observed in the transmission electron microscope (TEM), the dislocation substructure frequently is observed to be non-uniform such that the dislocations are concentrated on certain planes. The term "cell" refers to the unit of dislocation structure of order 1μm that is defined by these planes of stored dislocations.

Other models exist for Stage II work hardening which have been well reviewed by Nabarro et al. [2.10] and by Mitchell [2.31]. Since this thesis is not concerned with small strain plasticity, only one model will be described in detail, the Areal Glide Model. This model appears to account for Stage II work hardening better than the older models. It also provides the basis for a model for large-strain hardening discussed in Chapter VI.

II.2b.4 Areal Glide Model

Kocks showed [2.32] that the spatial distribution of intersections of forest dislocations with a slip plane makes a crucial difference to work hardening. If the intersections were arranged on a perfect lattice, then at the critical shear stress, a mobile dislocation would be able to shear the whole slip plane. In reality, however, the distribution is non-uniform and yield occurs at a percolation transition. That is, the distance that a mobile dislocation can travel increases very rapidly as
the applied stress nears the macroscopic yield stress. A mobile
dislocation can cause macroscopic yielding even though it leaves small
concave loops behind its passage around local "hard spots". A hard spot
corresponds to a local (upward) fluctuation in the dislocation density.

A computer simulated microstructure, Fig. 2.4, illustrates what
happens. An important part of this process is that the concave loops do
not remain as Orowan loops, as if the hard spots were impenetrable
particles, but instead plastic relaxation occurs. This means that
additional dislocations are generated on secondary slip systems so that
the concave loop on the primary slip system is converted to dislocation
density on other slip systems. Without this relaxation process, tangles
would not form because continuing straining would pile up more Orowan
loops until enough stress concentration occurred to collapse the
innermost loop. At this point dynamic equilibrium has been attained and
each new loop will collapse the innermost loop.

Kocks later detailed this "Areal Glide" theory for Stage II work
hardening where the development of dislocation tangles develops from the
accumulation of concave glide loops around hard spots on a slip plane
[2.13]. The key quantity is the spacing between hard spots, λ, assumed
to be proportional to the current mean dislocation spacing,

\[ \lambda \approx \frac{10}{\sqrt{\rho}} \]  \hspace{1cm} (2b.4)
Fig. 2.4 Concave dislocation glide loops around "hard spots" on a slip plane, from the results of a computer simulation of dislocation glide across a slip plane with a random distribution of point obstacles that represent the intersections of forest dislocations with the slip plane, Hanson [2.33].
The constant of order 10 was justified on the basis of computer modeling of dislocation motion through a field of randomly positioned obstacles, representing the intersection of forest dislocations with the slip plane [2.32]. Then if the passage of a mobile dislocation across the slip plane leaves behind a loop of length $/rho$ at each hard spot, the strain per loop stored, $d\gamma$, is $b\lambda^2$, giving

$$d\rho/d\gamma = (/rho \ b \ \lambda^2)^{-1}$$

(2b.5)

from which it can be derived that

$$d\tau/d\gamma = \alpha \ G / (2 \ \lambda^2 \ \rho)$$

(2b.6)

$\lambda$ is inversely proportional to $/rho$ so from Eq. 2b.4 it follows that the geometrical storage of dislocation line length gives a reasonable value for the hardening rate (in Stage II) of about $G/200$. Kocks [2.13] went on to show that the Areal Glide model accounts, for example, for the difference between the observed long slip steps that can be observed on the surface of deformed metals in Stage II and the small mean free path for dislocation storage that is required to explain a high work hardening rate.

II.2b.5 Recovery Experiments

Experimental work has been reported by Hasegawa et al. [2.34] that supports the "two phase" character of slip, i.e. the difference between hard spots on the slip plane where geometrical storage of dislocations occurs, surrounded by softer areas where the mobile dislocations percolate across the slip plane. They measured the change in length during recovery of aluminum samples that had been predeformed in either tension or compression. In both cases the material continued to deform in the same direction as the previous straining though only after an initial reverse straining. Thus a tensile specimen will first contract
(on the order of 0.01%) and then lengthen. The initial reverse straining was ascribed to dislocations running back in the soft regions of their slip planes. The subsequent straining in the same direction as the pre-strain was ascribed to recovery in the hard spots or tangles. The direction of the recovery strain changes sign because the residual stress on each slip plane (on removing the applied stress that accomplished the pre-strain) is negative in the soft regions though still positive in the hard spots. Stated in another way, the fact that there is a measurable, signed strain during thermal recovery indicates that the dislocation networks stored during monotonic straining are polarized.

II.2b.6 Misorientations

The storage of concave glide loops around hard spots in the glide plane cannot easily explain the development of cell to cell misorientations. The production of secondary slip with short mean free paths for convex loops on the secondary glide planes, however, can explain the development of misorientation with increasing strain. Single slip in Stage II was investigated for copper by Steeds [2.21]. His work was the first to report TEM observations in sections not cut parallel to the slip plane. When copper deformed into Stage II is viewed so that the primary slip planes are edge on, alternating black-white contrast is seen between the "carpets" of dislocations that lie parallel to the slip planes, Fig. 2.5. The diffraction conditions and orientation shown for this image suggest that the misorientation across the carpets could not be due to dislocations of the primary slip system. Moreover dislocations lying in the carpets were shown by diffraction contrast experiments not to be primary dislocations.
Fig. 2.5 Mats of stored dislocations lying parallel to slip planes, from Steeds [2.21]

Reprinted with permission from J.W. Steeds, "Dislocation Arrangement in Copper Single Crystals as a Function of Strain," Proc. Royal Society, A292, 343, 1966. Figure 40 from the cited article.
For multiple slip in polycrystals, there are several active primary slip systems and therefore secondary systems. Therefore there are sufficient slip systems to cause the analogous development of closed boxes or cells, each of which is misoriented by a few degrees from its neighbors. It is known that the development of misorientations across cell walls is not an essential part of work hardening because of the work at high strain rates that has been performed by Follansbee and coworkers. Gray [2.35] and Gray and Follansbee [2.36] have shown that copper, for example, can be shock loaded without any net plastic strain. Yet the material has a high flow stress and a dislocation cell structure that lacks cell-to-cell misorientations. This suggests that misorientations between cells or subgrains are a byproduct of straining but are not required for dislocation storage (and therefore hardening) to occur. Misorientations are therefore due to small imbalances in the number of dislocations of one sign in a cell wall. The Hasegawa et al. [2.34] concept of cell formation (section II.2b.5 above) allowed for the formation of misorientations within cell walls but did not envisage net misorientations across the cell walls as a whole.

Stage II has been reviewed in some detail because the general nature of small strain work hardening forms the foundation of the modeling of large-strain work hardening to be described in a subsequent chapter. The essential features of Stage II are that dislocation storage occurs for purely geometrical reasons which leads to a constant work hardening rate that is nearly insensitive to temperature or strain rate. II.2c Stage III / Dynamic Recovery

Stage III is characterized by a hardening rate that decreases monotonically with increasing flow stress, leading to the much repeated
term "parabolic hardening" for the stress-strain curve. In contrast to the athermal storage of dislocation line length in Stage II, Stage III behavior is highly temperature sensitive and somewhat strain rate sensitive. Stage III is associated with "dynamic recovery" which means the recovery or loss of line length during plastic straining and which is generally only weakly time dependent. The time dependence can be stated more strongly than this because the rate of recovery is much greater than would occur by static recovery at the same temperature. Dynamic recovery is distinct from the time-dependent kinetics associated with static recovery during annealing treatments. This distinction will be seen to be important in subsequent discussions of large strain models of work hardening. The availability of a model for Stage III constructed from first principles of dislocation properties is perhaps even more remote than for the previous stages of work hardening.

II.2c.1 The Stage II-Stage III transition:

In polycrystalline aluminum at room temperature, for example, Stage III is the only observable stage of work hardening. At lower temperatures, however, Stage II can be observed in aluminum, Hosford et al. [2.20]. The transition from Stage II to Stage III is one of the few phenomena in work hardening that has been successfully modeled by a single dislocation behavior. Haasen pointed out [2.37] that the temperature dependence of the the stress at which Stage III starts is modeled by

$$\log(r_{III}) = A - BT. \quad (2c.1)$$

This is consistent with a model for thermally activated constriction of dissociated screw dislocation segments so that they can then cross-slip, Schoeck and Seeger [2.38]. Cross slip permits the
oppositely signed screw segments of dislocation loops in different slip planes to annihilate each other. The current flow stress enters through the fact that the applied stress can assist the constriction process, thereby lowering the apparent activation energy for the process. The activation energies determined from the experimental data for Cu and Ni by Haasen were of the correct magnitude. The presumption is that once cross-slip can occur at a high enough rate, dynamic recovery will begin. It is important to note however, that if work hardening models are constructed on the basis of a single dislocation phenomenon such as cross slip, the models will not fit all the experimental facts. For example, the lack of strong orientation dependence of the work hardening rate in either Stage II militates against any one mechanism being dominant. The justification of this remark is that different orientations should mean that a different type of dislocation-dislocation interaction is dominant, see e.g. Schwink and Gottler [2.39].

II.2c.2 The End of Stage III

The experimental evidence from much stress-strain data taken out to moderately large strains shows that the work hardening rate in Stage III decreases monotonically towards an apparent saturation stress. Accurate descriptions of stress-strain curves have been a topic of discussion for many years but this thesis will not attempt to discriminate between the many competing models in detail. Suffice it to say that the common power law relation has many times been shown to be adequate for only the limited range of the tensile test, e.g. Dorn et al. [2.40]. Voce [2.1] and Palm [2.41, 2.42] published an alternative
exponential stress-strain law that has the advantage of showing an asymptotic saturation stress. The differential form is

\[ \theta = \theta_0 \left( 1 - \sigma / \sigma_s \right) \quad (2c.2) \]

where the strain does not enter the relation explicitly. A result of this thesis is the demonstration that a modification of this equation can be made which is useful for describing large strain behavior. That modification is to make \( \sigma_s \) a quantity that increases (slowly) with strain instead of being constant for the particular combination of material, strain rate and temperature. Therefore it is useful to review the theoretical and experimental basis for Eq. 2c.2.

II.2c.3 Dislocation Model for Stage III

Kocks [2.12] studied the variation of the apparent saturation stress with temperature and strain rate in aluminum. He showed that the same dislocation kinetics apply to this as was found for the transition to Stage III, Eq. 2c.1. The physical basis for the Voce-Palm relation and the temperature dependence can be modeled as follows.

If the geometrically caused dislocation storage described for Stage II continues unabated throughout Stage III, the falling hardening rate implies an ever-increasing dynamic recovery rate. A simple assumption that can be made is that the amount of dislocation line length that is lost per unit strain is proportional to the current dislocation density. The physical concept is that of mobile dislocations sweeping through the dislocation network and providing a transient assistance to parallel dislocation segments that are nearly able to break past an obstacle. Quantitatively,

\[ d\rho / d\gamma = -L \rho \quad (2c.3) \]

By differentiating Eq. 2b.1, one obtains
\[ \frac{d\tau}{d\rho} = 0.5 \alpha G b \rho^{-0.5} \]  
(2c.4)

Combining Eqs. 2b.2, 2c.3 and 2c.4 leads to

\[ \frac{d\sigma}{d\epsilon} = \frac{M^2}{2} \left( \frac{k \alpha G b}{2} - \frac{\sigma}{2} \right) \]  
(2c.5)

which is equivalent to Eq. 2c.2 above, i.e. \( \theta_0 = \frac{M^2 k a G b}{2} \) and \( \sigma_s = \frac{2}{LM^2} \). The physical picture provided by Kocks is that the saturation stress is set in the hard spots where tangles form. The equation that describes the variation of the saturation stress is, by analogy with one form of stress-assisted, thermally activated motion of dislocations at a given structure, [2.43]

\[ \ln \left( \frac{\tau_s}{\tau_{s0}} \right) = -\frac{kT}{A} \ln \left( \frac{\epsilon_0}{\epsilon} \right) \]  
(2c.6)

where \( \epsilon_0 \) is a reference strain rate, and \( A \) represents an activation energy, or more correctly an activation work. The reason for the analogy is that, unlike the case of the stress at which Stage III commences, Haasen [2.37], the saturation stress is a result of the evolution of the dislocation structure, not the behavior of a fixed structure. The values of \( A \) reported by Kocks corresponded to a low activation energy, approximately 0.4 eV for aluminum. Therefore Eq. 2c.6 is a phenomenological description of thermal activated dynamic recovery that does not attempt to identify a particular controlling dislocation-dislocation interaction.

Equation 2c.6 suggests that the strain rate sensitivity of the saturation stress obeys a power law relationship between stress and strain rate. There are two important differences between this relationship and those derived on the basis of diffusively controlled recovery, examples of which are discussed below. One difference is that the exponent, \( m \), in the power law relation, decreases linearly with
temperature rather than being fixed by the diffusion mode considered, Eq. 2c.7,
\[ \dot{\varepsilon}/\varepsilon_0 = (\tau_s/\tau_{s0})^m, \quad m=A/kT. \] 
(2c.7)

The second difference is that at low temperatures, the exponent is very much higher than any possible exponent for a relation derived on the basis of diffusional recovery. For aluminum at room temperature, for example, the data of Kocks [2.12] indicates that \( m=16 \).

II.2c.4 Applicability of Kocks Model for Stage III

The model of Stage III discussed above predicts a linear decrease of hardening rate with increasing stress. Kocks [2.12] reproduced data for aluminum and stainless steel which showed that the model appears to work well at low homologous temperatures. For temperatures higher than about 0.3Tm, however, the \( \theta-\sigma \) plots become less linear such that a saturation stress extrapolated from data at medium strains is less than the flow stress eventually reached at large strains. Alberdi's data for copper and aluminum show the same trend of linear \( \theta-\sigma \) plots at low homologous temperatures giving way to curved plots at higher temperatures. Data for other f.c.c. metals, Mecking et al. [2.44], show that the Stage III model is a good description for Stage III at low temperatures in all unalloyed f.c.c. metals. The effect of adding solute to an f.c.c. metal appears to be analogous to a lowering of the temperature both in terms of raising the saturation stress at the end of Stage III and in terms of giving a linear \( \theta-\sigma \) plot. See, for example, the data of Hughes [2.6] for Ni-Co alloys.

Mecking et al. have recently investigated the application of this analysis to a number of f.c.c. metals tested over a wide range of temperatures [2.44]. They were able to show that Eq. 2c.6 applied over a
remarkably wide range of homologous temperatures and also that the
differences between materials, i.e. the variation of the parameter, A,
could be accounted for with a single parameter that varied in the same
way as the ratio of stacking fault energy to shear modulus and Burgers
vector. This scaling by the stacking fault energy is reasonable since it
strongly influences the spacing between pairs of partial dislocations
which in turn controls the activation energy for cross slip.

II.2c.5 Microstructural Evidence for Dynamic Recovery

The concept that saturation of the flow stress at the end of Stage
III represents the balancing of dislocation storage with dislocation
recovery may seem artificial. In other words, the simplest explanation
might be that the storage rate of dislocations simply drops to low
levels (zero at saturation of the flow stress). One important
microstructural point can be made here, however. If in a material with
well developed cells the dislocation storage rate tended to zero near
saturation, such that dynamic recovery were not necessary, then the cell
shape ought to change in the same way as the macroscopic shape change.
Langford and Cohen [2.2] showed many years ago that this was not the
case. As long as the cell structure remains even only approximately
equiaxed, a considerable amount of dislocation storage and dynamic
recovery must be occurring during straining.

II.2c.6 A Physical Picture for the Saturation Stress

Kocks [2.13] has provided a qualitative but useful physical
picture for the saturation stress at a given strain rate and
temperature. Starting with the Areal Glide model of Stage II work
hardening, dislocation storage occurs at the hard spots in the
microstructure. These hard spots develop into the cell walls typically
observed as straining proceeds. As the rate of dynamic recovery increases in Stage III, so a larger and larger fraction of dislocation segments stored in the hard spots or tangles can be recovered. The stored dislocation segments have a spectrum of lengths. The longer the segment, the smaller the (local) stress that is required to bow it out and recover it. As the flow stress rises as a consequence of strain hardening so the mean length of stored dislocation segment will decrease. The saturation stress is therefore determined by the longest dislocation segment that will not be recovered at the applied temperature and strain rate. A more detailed picture can be found in the statistical treatment of dynamic recovery by Kocks and Mecking [2.45].

II.2d. Summary of Small Strain Work Hardening

This completes the brief review of work hardening at "normal" strains. Review of Stage I demonstrated the possibility of dislocation debris controlling the low rates of work hardening in that Stage. The review of Stage II illustrated the success of a model for work hardening in Stage II that relies on the non-uniformity of dislocation distribution. The concept that there are places in a strained material where a saturation stress is determined will be used in the development of a model for Stage IV that will be described in a later chapter. Stage III shows that dynamic recovery occurs and that the rate at which dislocation line length is recovered increases as the stress increases.

II.3. Stage IV / Large Strains

Before reviewing the available stress-strain data at large strains, it is useful to summarize the results of treating plasticity on a crystallographic basis. Previous authors have placed considerable emphasis on the importance of texture development to understanding large
strain behavior in polycrystals. Although texture is relevant to understanding the differences between different deformation paths and between instability types in plastic flow, this thesis will focus on microscopic mechanisms for large-strain work hardening. The brief review of texture that follows is mainly intended to make the link between polycrystal plasticity and events on each slip system. It is assumed throughout this work that isotropic hardening applies, meaning that each individual slip system hardens to the same extent.

The underlying assumption in the above discussion is that the behavior of polycrystals only differs from that of single crystals by the geometry of slip. That is, the geometry of deformation of polycrystals precludes easy glide. Subsequent stages of work hardening, however, should be similar. These points have been extensively discussed in the literature, e.g., Kocks [2.46], with the conclusion that the assumption appears to be valid. This is the basis for comparing work hardening rates resolved onto a slip system.

Another assumption of this work is that the deformation PATH (direction in strain space) can be decoupled from the deformation HISTORY (total accumulated strain). This concept has been described in detail by, e.g., Tomé et al. [2.47], who used the geometry of slip in f.c.c. polycrystals to account for the differences between tension, compression and torsion strain paths applied to copper. Further investigation of the degree of dependence of strain hardening on strain path is the subject of current research at the Los Alamos National Laboratory by Stout and others. At the time of writing, however, it appears that there is a small difference in work hardening between straining in shear and other deformation modes. One of the conclusions
of this review, however, is that the large strain phenomena are independent of strain path, at least to first order.

II.3a. Crystallographic Texture

Any metal that undergoes large plastic deformation by dislocation glide on restricted slip systems will develop a crystallographic texture. This texture means that the orientation distribution function for the grains in the polycrystal becomes highly non-uniform. An essential feature of polycrystal plasticity is that the macroscopic shape change (strain) imposed on a polycrystal has to be accommodated by microscopic shears due to dislocation motion. In order to achieve any microscopic shear, a critical shear stress must be reached on the given slip system. Moreover five slip systems as a minimum must be activated in any given grain if Taylor's assumption is used that the shape change of any individual grain is fully prescribed by the surroundings. Many comparisons of simulated and experimental textures have shown that Taylor's assumption is a good one for large strain textures although minor modifications improve the fit in some cases. These modifications are known as "Relaxed Constraints" [2.48] because they model the relaxation of certain of the strain boundary conditions with the substitution of stress boundary conditions on individual grains.

The particular deformation path employed in this work, torsion, has a characteristic texture associated with it. Canova et al. [2.49] recently compared experimental torsion textures with simulated textures that included the effect of Relaxed Constraints. For any given texture or orientation distribution function, a ratio can be calculated between the magnitude of the applied stress required to attain yield (an upper bound) and the critical resolved shear stress in each grain. This ratio
is known as the Taylor factor and may be averaged over the ensemble of grains. The utility of such an analysis is that it provides a physical, crystallographic basis for the difference in strength of metals in shear and tension, for example. Equation 2b.1 is thus modified to read

\[ \sigma = M \alpha G b / \rho \]  

(3a.1)

where \( \sigma \) is the macroscopic applied stress for plastic flow and \( M \) is the Taylor factor.

Large strain experiments in different deformation modes conducted at Los Alamos have shown consistent differences in the stress-strain curves, Fig. 2.6, Stout [2.50]. The evolution of the Taylor factor for each deformation mode is different, Fig. 2.7. When the differences in texture evolution are taken into account by scaling each curve by the appropriate Taylor factor, the differences are seen to be much smaller, Fig. 2.8. The conclusion from this analysis is that work hardening is independent of the deformation mode, to first order. The rate of change of the Taylor factor decreases with increasing strain. In torsion the Taylor factor fluctuates within a certain range. Therefore changes in the Taylor factor are small at large strains and are not likely to be able to explain the sustained increase in measured flow stress observed in Stage IV in many cases.
Fig. 2.6 Stress-strain curves for copper tested in wire-drawing, compression and torsion, Stout [2.50]. The von Mises yield criterion has been used to calculate equivalent stresses and equivalent strains for the torsion data (stresses multiplied by √3 and strains divided by √3)
Fig. 2.7 Evolution of the Taylor Factor for the three strain paths, tension (wire-drawing), compression and torsion. The shear strain and the Taylor Factor for torsion have been divided and multiplied, respectively, by $\sqrt{3}$ to put them on a von Mises equivalent basis.
Fig. 2.8 Stress-strain curves for copper for the same three strain paths as plotted in Fig. 2.6 except divided by the appropriate Taylor Factors as calculated by the LAPP texture simulation code (\(\sigma/M\) plotted versus \(\Sigma M(\varepsilon)d\varepsilon\)).
This thesis does not discuss or report texture development in detail since for torsional deformation it has been well described both theoretically and experimentally, Canova et al. [2.49]. The brief review presented here of crystal plasticity is essential to the analysis presented in subsequent sections where it is important to be able to convert macroscopic stress-strain data to resolved shear stress-shear strain data appropriate to slip on any individual slip system. Therefore a limited set of texture data is presented in Chapter 4 to validate the arguments presented here.

II.3b. Large-Strain Work Hardening, Stage IV

A complete review of large strain experiments was provided by Gil-Sevillano et al. [2.3] to which the reader is referred for complete lists of references to large-strain studies. The classical large strain metal working process is rolling and the development of large strain studies began with tensile tests of rolled sheet. Such tests have obvious drawbacks: one, there is a major change of deformation mode in going from plane strain compression to tension in the subsequent test; two, there is generally a large change in strain rate from the typically greater than 1 s\(^{-1}\) (von Mises equivalent) strain rate of the rolling passes to the less than 10\(^{-4}\) s\(^{-1}\) rate of typical tensile tests; three, the stress-strain curve that is generated is discontinuous.

In the oft-cited work of Langford and Cohen [2.2], wire drawing was used to attain large strains and the composite stress-strain curve was rendered nearly continuous by careful tensile tests in which the effects of necking were taken into account. Therefore there was little strain path change between the wire-drawing and the subsequent tensile tests. The main result of their work was that hardening persists, albeit
at a low level, to strains as high as 10 without any sign of saturation of the flow stress, Fig. 2.2. Some years before this, Kovacs and Feltham [2.51] published continuous stress-strain curves obtained from torsion experiments on Ni and Ag which showed a clear Stage IV for the Ni, albeit to a smaller strain. The advantages of torsion for large strain studies will be discussed in more detail in the section on experimental work.

II.3b.1 Alloying

The work of Rack and Cohen [2.52] on iron alloys, Fig. 2.9, demonstrated that the Stage IV work hardening was essentially unaffected by alloying though the flow stress level at a given strain generally increased with alloy content. The same conclusion can be drawn from the more recent work by Hughes [2.6] on Ni alloys, Fig. 2.10. If these data from two different classes of crystal symmetry are replotted in terms of resolved shear stresses divided by the shear modulus appropriate to the material and temperature, Fig. 2.11, it can be seen that Stage IV intervenes at approximately the same (reduced) work hardening rate.
Fig. 2.9 Stress-strain curves for various Fe-Ti alloys, wire-drawn and tested in tension, after Rack and Cohen [2.52], showing that Stage IV occurs at the same strain hardening rate but at a stress level that increases with alloy content.

Reprinted with permission from H.J. Rack and M. Cohen, Mat. Sci. and Eng. 6, 320-326, 1970. Figure 2 from the cited article.
Fig. 2.10 Shear stress-shear strain curves for three nickel alloys tested in torsion by Hughes [2.6], again showing similar strain hardening rates at large strains but stress levels that increase with alloy content.
Fig. 2.11  a) hardening rate versus stress, divided by the shear modulus of iron (79,000 MPa) and resolved onto a slip plane using a Taylor Factor of 3.1, using the data of Rack and Cohen from Fig. 2.9.
Fig. 2.11 b) resolved hardening rate versus resolved stress for three nickel alloys, using a shear modulus of 79,000 MPa and a Taylor Factor of 1.55. Note that Stage IV occurs at a hardening rate of order $2.10^{-4}$. Hughes [2.6]
The effect of alloying with a dispersed second phase is shown in Fig. 2.12, taken from Gil-Sevillano et al. [2.3]. Stage IV is present in these materials at a level of about $2 \times 10^{-4}$G, similar to that of single phase copper. The effects of alloy addition that produce a continuous or near continuous second phase are discussed in section 3c.8 below.
Fig. 2.12 Stress-strain curves for various dispersion hardened copper alloys, from Gil-Sevillano et al. [2.3], showing that the presence of the dispersed second phase primarily affects the stress level in Stage IV but not the hardening rate.

Reprinted with permission from Prog. Mat. Sci., 25, 69. J.G. Sevillano, P. Van Houtte, and E. Aernoudt, "Large Strain Work Hardening and Textures," 1981, Pergamon Journals, Ltd. Figure 2.17 from the cited article.
II.3b.2 Temperature

Alberdi performed large strain torsion tests on copper [2.5] at
five different temperatures, Fig. 2.13. Plots of hardening rate versus
stress, Fig. 2.14 show clear transitions from Stage III behavior to
Stage IV behavior at all temperatures, despite the fact that the Stage
III behavior deviates from a linear decrease of $\dot{\theta}$ with $\sigma$ at higher
temperatures. Extrapolation of the Stage III behavior to zero hardening
rate yields the same exponential dependence, Eq. 2c.6, of nominal
saturation stress on temperature as discussed in section 2c above. The
work hardening rate and stress at which the transition to Stage IV occur
both decline slowly with increasing temperature. Embury and Mecking
[2.53] suggested a criterion for the transition of the form

$$\dot{\theta}_{IV} = c \tau$$  \hspace{1cm} (3b.1)

where $\dot{\theta}_{IV}$ is the work hardening rate at which the transition to
Stage IV occurs, not necessarily the work hardening rate in Stage IV.
This criterion is represented on Fig. 2.14 as a straight line with
c=0.05 and appears to fit the data reasonably well. Embury and Mecking
[2.53] noted the similarity of this criterion to those for diffuse
necking, $\dot{\theta}=\sigma$, and local necking, $\dot{\theta}=0.5\sigma$, and speculated that Stage IV
might be associated with microscopic instabilities such as the
microbands commonly observed large strains in copper, [2.54]. The onset
of instabilities in plastic deformation generally results in reduced
load carrying capacity in the material, however, whereas Stage IV
definitely results in increased load bearing capacity. This point will
be taken up in the Discussion chapter.
Fig. 2.13 Shear stress- shear strain curves for copper tested in torsion at five different temperatures by Alberdi [2.5].
Fig. 2.14  Resolved hardening rate versus resolved stress for copper tested in torsion at five different temperatures, after Alberdi [2.5], using a temperature dependent shear modulus and a Taylor Factor of 1.55.
II.3b.3 Strain Rate Effect on Stage IV

The effect of strain rate variations on large strain behavior has not been as extensively studied as the parameters of temperature and material. The only study known to the author where more than two strain rates were studied is that on 99.99% Al by Bailey and Singer [2.55] where plane strain compression was used to reach strains of 2. The work hardening plots, Fig. 2.15, show that the work hardening rate in Stage IV is not particularly sensitive to strain rate. This is the same behavior as found previously for solute variation, section 3b.1. Extrapolation of work hardening plots to zero work hardening at a (presumed) end of Stage IV is of interest. This is because the strain rate sensitivity of the actual saturation stress can yield information on the rate controlling step for recovery at large strains. This point is taken up in section 3c.6.
Fig. 2.15  Resolved shear stress versus resolved stress for 99.99% Al tested in plane strain compression by Bailey and Singer [2.55] at four different strain rates. Note the lack of transition to a Stage IV behavior.
II.3b.4 Constant Structure Strain Rate Sensitivity

The strain rate sensitivity at constant structure is an important parameter for studies of the rate of development of plastic instabilities. Most f.c.c. alloys exhibit a small positive rate sensitivity which tends to slow down the localization of plastic flow in necks and shear bands, e.g. [2.56]. The strain rate sensitivity can be quantified as $d\sigma/dln\dot{\varepsilon}$ which can be measured experimentally by performing strain rate changes during a stress-strain test. A plot of this measure of strain rate sensitivity versus flow stress, known as a Haasen plot [2.37], often shows a straight line through the origin for pure f.c.c. metals for Stage II of work hardening. Another test of the nature of the resistance to dislocation motion is to change the temperature and calculate the ratio of flow stresses measured before and after the temperature change, known as the Cottrell-Stokes ratio. This ratio remains constant over the same range of strain and flow stress as the Haasen plot remains straight. These characteristics can be explained on the basis of thermally activated flow of mobile dislocations past obstacles which are other (forest) dislocations. The Haasen plot shows a proportionality between rate sensitivity and stress because the stress is a direct measure of the inverse mean spacing between obstacles. The Cottrell-Stokes ratio remains constant because it is a measure of the fraction of the flow stress that is caused by obstacles at which thermal activation is possible, i.e. short range obstacles; work hardening increases the obstacle density but does not change the composition of
the obstacles. The above discussion applies to Stage II and the first part of Stage III of work hardening.

II.3b.5 Strain Rate Sensitivity at Large Strains

At high strains in Stage III, however, the strain rate sensitivity increases more rapidly than the stress and the Cottrell-Stokes ratio decreases. These departures from small-strain behavior occur earlier with increasing temperature. At low homologous temperatures the same departures are observed but at large strains. Alberdi conducted extensive experiments on both copper and aluminum. The copper data for strain rate sensitivity, Fig. 2.16, showed a marked departure from linearity only for the highest temperature, 473K. The two lower temperatures showed mildly concave upwards curves. The same plot for aluminum, Fig. 2.17, showed pronounced departures from linearity. Hughes obtained Haasen plots for nickel alloys at two temperatures, room temperature and 573K. The room temperature data, Fig. 2.18, shows similar results to those from copper, i.e. mild departures from linearity. The higher temperature data, however, Fig. 2.19, show dramatic increases in strain rate sensitivity. On all four Haasen plots the stress at which the transition to Stage IV occurs has been marked. It is clear that above a homologous temperature of about 0.3, the onset of Stage IV coincides with a marked increase in the strain rate sensitivity. Alberdi also measured the Cottrell-Stokes ratio for copper and aluminum with temperature changes between 53°C and 17°C. Although the ratio varied by more than the experimental scatter, there was no obvious correlation with the onset of Stage IV in either case.
Fig. 2.16 Strain rate sensitivity measured by strain rate jumps, for copper tested in torsion at three temperatures by Alberdi [2.5], plotted versus stress. The stress at which the transition to Stage IV occurs has been marked by a vertical arrow. This Haasen plot is expected to show a linear plot with zero intercept, for a pure f.c.c. metal.
Fig. 2.17 Strain rate sensitivity plotted in the same way as the previous figure for aluminum tested at 293K and 373K, from Alberdi [2.5].
Fig. 2.18 Strain rate sensitivity for three nickel alloys tested in torsion at room temperature by Hughes [2.6].
Fig. 2.19 Strain rate sensitivity for three nickel alloys tested in torsion at 573K by Hughes [2.6]. In contrast to the room temperature data, Fig. 2.18, the strain rate sensitivity increases markedly on entering Stage IV.

The departures from linearity of the Haasen plot and the changes in the Cottrell-Stokes ratio have been modeled by Mecking and Kocks on the basis of a multiplicative factor in Eq. 2b.1 [2.26],

$$r = s(\dot{\epsilon}, T) \alpha G b \sqrt{\rho}$$  

(3b.2)

The factor $s(\dot{\epsilon}, T)$ decreases slowly as the rate of dynamic recovery increases, which leads to a slightly less than linear rise in flow stress with inverse mean dislocation spacing. In effect, the parameter, $\alpha$, that averages all the possible dislocation-dislocation interactions is postulated to decrease as the rate of dynamic recovery rises. A small decrease in $s$ leads, however, to more pronounced increases in strain rate sensitivity and the model was shown to account very well for the variations of strain rate sensitivity with stress and temperature in copper and silver. The model predated the reports of marked increases in strain rate sensitivity observed at high temperatures in Stage IV by Alberdi and Hughes. The variation of the factor $s$ with stress was linked to the change in hardening rate after a change in strain rate. It is interesting to speculate that the high temperature behavior might be linked to changes in transient hardening behavior in Stage IV; the larger the transient change in hardening rate after a strain rate change, the larger the increase in strain rate sensitivity will be in the Mecking-Kocks model.

The conclusion of this brief discussion of strain rate sensitivity is that at low homologous temperatures, the evolution of the strain rate sensitivity is a smooth function of flow stress; no marked discontinuities are present at the transition from Stage III to Stage IV until the deformation temperature is well above 0.3Tm. The significance of this is that Stage IV occurs without marked changes in the character
of obstacles to dislocation movement. If, for example, Stage IV were due
to the accumulation of very small obstacles such as vacancies or vacancy
clusters, these obstacles should be surmountable by thermal activation.
The strain rate sensitivity would then be expected to rise significantly
in Stage IV. Large obstacles to dislocation motion such as low-angle
boundaries, however, are not surmountable by thermal activation and so
should not contribute to the rate sensitivity of a material.

II.3c. Possible Causes of Stage IV

Over the years many causes for the low level of work hardening
observed in Stage IV have been proposed. Mecking and Grinberg [2.57]
listed eight possible causes of Stage IV which are discussed below.

II.3c.1 Grain Size:

Many alloys exhibit an increase in flow stress with decrease in
grain size. Since the spacing between grain boundaries in at least one
direction decreases to very small dimensions at very large strains,
Stage IV might be a manifestation of grain boundary strengthening. The
analogy here is with the case of hard second phases discussed above,
Embry [2.58]. Jago and Hansen [2.59] have recently shown, however, for
pure iron that the grain size effect tends to vanish, even at the
relatively small tensile strain of 0.2. Hughes [2.6] compared the strain
hardening of two different grain sizes of Ni-30Co out to large torsional
strains, Fig. 2.20, and found also that the grain size effect tended to
vanish at large strains. The reverse torsion experiments described in
Chapter III also tend to contradict a grain size cause for Stage IV.

Mecking [2.60] reported the results of rolling a stable orientation of
single crystal to large strains. In this case there are no high angle
grain boundaries in the material but Stage IV does occur. Mecking
ascribed this to the proliferation of microbands in Stage IV. The result indicates that high angle grain boundaries are not essential to Stage IV hardening.

II.3c.2 Deformation Bands:

Hatherly and Malin [2.54] defined deformation bands as the regions of grain where slip has occurred homogeneously. Deformation bands are separated by transition bands where the lattice orientation changes from that of one deformation band to the orientation of the deformation band on the other side of the transition band. Mecking and Grinberg [2.57] hypothesized that when the deformation bands decreased in size to dimensions comparable with the cell size, the hardening in the cell structure might be affected.
Fig. 2.20 Shear stress- shear strain curves for Ni-30Co tested in torsion by Hughes [2.6], showing that different grain sizes have very little effect on Stage IV.

A related topic is that of misorientations between cells which tend to increase with strain. Alberdi [2.5] investigated both copper and aluminum and found that the misorientations increased up to 10° by a shear strain of 4 in Al. At larger strains, however, there appeared not to be any further increase in misorientation. It is possible that 10° misoriented cell walls might act as barriers to dislocation motion. This point is left for discussion in chapter VII since the available evidence from the literature is insufficient to resolve the matter here.

II.3c.3 Surface Effects:

Samples deformed to large strains often have large surface to volume ratios. It is known, however, that the presence of a surface tends to soften a metal, Fourier [2.61]. The anticipated effect of surfaces, therefore, would be to diminish the strain hardening at large strains. For torsion, the surface to volume ratio is constant and so could not affect the hardening.

II.3c.4 Stress induced transformations:

In certain materials, such as stainless steels which are only marginally stable as f.c.c., a phase transformation can occur at stresses elevated by strain, causing enhanced hardening. This is, however, not relevant to Stage IV in stable single phase metals such as the aluminum alloys studied in this work or the unalloyed Ni studied by Hughes [2.6] and the unalloyed copper studied by Alberdi [2.5].

II.3c.5 Changing strain path:

If the relative amount of redundant strain varies, as it might in a wire drawing process for example, it is possible that the hardening might vary. It is hard, however, to see how it could lead to the sustained hardening of Stage IV and it does not explain the occurrence
of Stage IV in torsion experiments. There is also experimental evidence that changes of strain path reduce the rate of work hardening, see for example Jackson and Basinski [2.62], Armstrong et al. [2.63], Martin [2.64], the reverse torsion experiments reported in this thesis, chapter III, and current research on variable strain paths by Takeshita [2.65].

II.3c.6 Plastic Instabilities:

This possible cause of Stage IV was discussed in section II.3b.2 and found to be unsatisfactory.

II.3c.7 Texture:

One possible effect of texture development is that the Taylor factor continues to change at large strains. In wire drawing, for example, theoretical calculations show that the factor increases monotonically with strain. The small hardening rates measured in Stage IV might then be due to this gradual change in texture. The same hardening rates are observed in torsion, however, where the changes in Taylor factor are small and the value fluctuates in around a mean value. Large plastic strains at low homologous temperatures lead to strongly developed crystallographic textures. It has been argued that as the orientation of each grain changes, so the combination of active slip systems changes. This argument depends on the latent hardening of inactive slip systems being greater than the currently active ones. Then when a change of active slip systems occurs, the flow stress will rise. Latent hardening, however, is known to be largely a small strain effect, Frangiosa [2.66]. Also most grains re-orient to a stable orientation by a strain of about 2 so this effect would be expected to saturate and not lead to any work hardening at very large strains.
II.3c.8 Second-phase particles:

The presence of second phase particles of high aspect ratio is a special case in large strain behavior. High strains lead to decreasing spacings between the second phase in at least one direction. If the second phase is hard enough to block dislocation flow in the matrix then the mean free path for dislocation motion decreases strongly with strain. As the mean free path decreases, the flow stress rises by the Hall-Petch effect. If strain is measured as logarithmic strain, then the decrease in spacing is an exponential function of the strain. Embury and Fisher [2.67] demonstrated this type of hardening behavior in a heavily drawn pearlitic steel, Fig. 2.21. Bevk [2.68] more recently demonstrated this in drawn Cu-Nb alloys where the second phase was in the form of filaments. Further discussion of the two-phase material case is provided in the chapter on Modeling.
Fig. 2.21 Semi-logarithmic plot of stress versus strain for pearlitic steel from Embury and Fisher [2,67], showing an exponential increase of stress with strain at large strains.

II.3d. Quantitative Models for Large-Strain Work Hardening

Two models have been proposed recently for large strain stress-strain behavior by Nix. et al. [2.70] and by Prinz and Argon [2.71]. Both models employ a "two-phase" structure model where each mobile dislocation must pass through both the soft and the hard parts, "phases", of the structure. This should be contrasted with the Areal Glide model described in section 2b.4 where the existence of hard areas in each slip plane was essential to the description of the high hardening storage rates in Stage II. Whereas the Voce-Kocks model for the effect of dynamic recovery on work hardening rate predicts a linear decrease of hardening rate with increasing stress, both the Nix and the Prinz models show a less rapid decay of hardening rate. This is in keeping with the experimental data for hardening rate versus stress from many cell-forming materials, see e.g. Embury et al. [2.69]. Neither model, however, predicts the sharp transition from Stage III to IV that is observed experimentally. The models are summarized below in order to provide a complete picture of the current state of understanding of large strain behavior. The Prinz-Argon (PA) model in particular introduces several useful concepts connected with dislocation debris such as dipoles and prismatic loops.

II.3d.1 Nix et al. model [2.70]

Nix. et al. recently published a model of large strain work hardening based on a two phase dislocation structure with cell walls and interiors. The cell walls are infinite slabs with finite thickness. Differences in flow stress between cell interior and wall are allowed for by including elastic strains in the model. Each mobile dislocation is presumed to traverse both the soft cell interiors and the harder cell
walls. The model supposes that edge segments are deposited in the cell walls when dislocations expand along the cell interiors. Dislocation storage in both the cell interiors and the cell walls is modeled on the same geometric basis as was described above for Stage II work hardening rates. Dynamic recovery in the cell interiors is modeled by the kinetics of cross-slip. Recovery in the cell walls is modeled by a climb controlled process which results in strongly time dependent kinetics for the attainment of a saturation stress. The set of equations that they obtained required numerical techniques for solution. The results of the model for three different temperatures are plotted as hardening rate versus stress in Fig. 2.22. Although the curves are of the correct general form, there is no sharp transition to Stage IV as found experimentally. Also the residual hardening rate at the lowest temperature, $4.10^{-4} \text{C}$ at 300K, for example, is larger than that found experimentally, Hughes [2.6].
Fig. 2.22 Theoretical shear stress–shear strain curves for nickel for three deformation temperatures, showing the results of a numerical solution of the model of Nix et al. [2.70].

Reprinted with permission from W.D. Nix, J.C. Gibeling, and D.A. Hughes, "Time Dependent Deformation of Metals," Met. Trans., 16A, 2215, 1986. Figure 7 from the cited article.
II.3d.2 The Prinz and Argon model [2.71]

The Prinz and Argon model supposes a similar cellular structure but with a different method of assessing the flow stress contributions of the walls and interiors. The work hardening behavior of the cell interiors is simply modeled by the Voce-Kocks model. At the same time, however, mobile edge dislocations interact to form dipoles, as they do in Stage I. These dipoles can break down to form prismatic loops, as was so clearly shown by Price in hexagonal metals, for example [2.72]. Once in the form of loops, the debris is driven into the cell walls where it is trapped. The loop debris makes a contribution to the flow stress, \( \tau \), in the walls that is simply related to the mean loop size, \( <r> \), and number density of loops, \( n \), Kroupa [2.73],

\[
\tau = \sqrt{2/8} G b \sqrt{<r> n}
\]  

(3d.1)

The results of this model are similar to those of the Nix et al. model where a plot of hardening rate versus stress fitted to data by Lloyd et al. [2.74], Fig. 2.23, showed a concave upwards curve. Again, the rate limiting step for recovery in the cell walls is presumed to be diffusion controlled climb.

Both this model and the Nix et al. model predict saturation stresses that follow a power law with a small exponent,

\[
\sigma_s = A \dot{\varepsilon}^{1/n}
\]  

(3d.2)

where \( n<10 \). This is a reasonable model for high enough homologous temperatures where bulk diffusion can operate, say \( T/T_m>0.5 \). Prinz and Argon assumed that pipe diffusion would be the rate limiting step in order to fit their model to the room temperature data for aluminum at 0.3 of the melting point. Even at this temperature, the observed value of \( n \) is approximately 16, Kocks [2.12]. The topic of recovery mechanisms
at large strains will be discussed further in later sections. If the form of the debris is prismatic loops then the work of Westmacott and coworkers, e.g. [2.75], has shown that the recovery of loops is controlled by bulk diffusion. Therefore it seems unlikely that either of these two models should apply at low homologous temperatures.

Both models are limited in that they are derived on the basis of a cellular structure. There exist materials such as Al-5Mg, however, that do not form cellular dislocation structures and yet follow the same qualitative Stage III and Stage IV behavior as pure nickel, Hughes [2.6], which forms a clearly cellular dislocation structure. That is, a \( \theta-\sigma \) plot shows an approximately linear decrease of hardening rate with increasing stress.
Fig. 2.23 Plot of resolved hardening rate versus resolved stress for the experimental data of Lloyd et al. [2.74], compared with the results (solid line) of a numerical solution of the model of Prinz and Argon [2.71].

II.4 Dislocation Loops and Dipoles

An idea that has been implicit in the discussion to this point is that debris such dislocation dipoles and loops are less effective obstacles to the motion of mobile dislocations than are "monopoles" or isolated dislocation segments. The stress fields of isolated dislocations are well known to decay rather slowly as the inverse of the distance from the dislocation core. For dipoles, however, Li [2.76] has shown that the stress exerted by one dipole on another falls off as the square of the separation. This seems reasonable as the shear stress fields of each component of a dipole should nearly cancel each other, leaving only a net dilatational stress field. For dislocation loops, Kroupa [2.73] has derived the displacement and stress fields around infinitesimal prismatic loops and shown that the stress decreases as the cube of the distance. For loops of finite size [2.73], the stress decreases as the reciprocal of the distance when close to the loop as again as the the cube when far from the loop. Kroupa has also considered the interaction of loops and monopolar dislocations [2.77] and shown that the primary interaction is a strong elastic interaction at short range. For estimating the effect of a population of prismatic loops on the flow stress of a material, Kroupa [2.73] offers no less than three different formulae, based on different methods of averaging the various possibilities for dislocation-loop interaction. All methods, however, yield approximately the same increment of flow stress for the loop densities and sizes commonly observed in quench and age experiments. Dollar and Thompson, for example, [2.78] observed densities of the order $10^{22} \text{ m}^{-3}$ and a mean size of order 10 nm. One of the formulae quoted by Kroupa is convenient because it employs the product of the mean loop
size and number density as an effective line density; this was used by Prinz and Argon for their model, see Eq. 3d.1. Note that the interaction parameter in this equation has the value $\sqrt{2}/8$, $-0.18$, which is significantly lower than the accepted value of about 0.5 [2.26] for monopolar dislocations.

II.5 Definition of Stage IV

An appropriate coda to this review is to define Stage IV. It has been observed that, when Stage IV occurs, persistent hardening is observed at a hardening rate of approximately $2.10^{-4} G$. Stage III, however curved the $\theta-\sigma$ plot may be, shows a smooth monotonic decrease of the hardening rate to zero as the stress increases. It therefore seems reasonable to define the onset of Stage IV as the departure in a plot of hardening rate versus stress (resolved quantities) from a smooth descent to zero hardening rate in the vicinity of $\theta=2.10^{-4} G$. Stage IV itself may be an almost constant work hardening rate, as in the low temperature limit, or it may be a vestigial prolongation of work hardening as at higher temperatures in pure metals. This phenomenological definition of Stage IV is least ambiguous in the low (homologous) temperature limit.

II.6 Summary

The salient features of large-strain work hardening are as follows.

* Single phase materials exhibit linear work hardening at strains greater than about 1 provided that the deformation is imposed at a low enough homologous temperature. The level of work hardening is about $2.10^{-4} G$. 
* Two-phase materials where the second phase is continuous, e.g. fiber composites, or semi-continuous, e.g. pearlitic steel, exhibit similar levels of work hardening at large strains. The work hardening rate slowly increases, however, and this can be modeled by a Hall-Petch model.

* Texture development tends to saturate at large strains and is therefore not expected to contribute to Stage IV work hardening. The differences in Taylor factor due to strain path direction, however, account for most of the differences observed in stress-strain behavior between different deformation paths.

* Existing quantitative models [2.57,2.70,2.71] for large strain behavior have two clearly identifiable drawbacks: one, they do not reproduce the sharp transitions in hardening rate commonly observed between Stages III and IV; two, they postulate a recovery mechanism at large strains whose rate controlling step is diffusion. This leads to a power law dependence for the saturation stress which is not observed.

* A satisfactory model for work hardening in Stages II and III is available that accounts for the hardening rates, slip line lengths, strain rate sensitivity and non-uniformity of dislocation distribution, [2.12,2.13,2.26]. This model provides a definition of Stage IV in terms of deviations from a smooth decrease of hardening rate with stress towards a zero hardening rate at saturation.
II.7 References


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Chapter III. Experimental Investigation of the Stress-Strain Behavior at Large Strains in Various Aluminum Alloys

III.1 Introduction

This chapter first describes the experimental techniques and some checks on the techniques. Then the results of the stress-strain tests are given, chosen in such a way as to emphasize different aspects of large strain behavior. Therefore certain sets of data appear more than once.

III.2 Materials

The material chosen for this study was the f.c.c. metal aluminum and certain of its alloys. Aluminum has only the f.c.c. solid allotrope and those elements with which it forms solid solutions do not change the stacking fault energy appreciably, Kritzinger et al. [3.1]. It does not twin except under such extreme conditions as the shock loading used by Gray on Al-5Mg [3.2]. Therefore at room temperature and below, the only hardening mechanisms in Al are solutes, dislocation multiplication and the effects of second phases. The maximum test temperature was half the melting point of aluminum so the dominant dislocation motion was presumed to be glide rather than climb. The lack of dynamic recrystallization in aluminum at elevated temperatures was a further reason for the selection of this alloy system. Hughes' [3.3] investigation of Ni showed that dynamic recrystallization intervened at high homologous temperatures before Stage IV behavior could be established.
III.2a Alloy choice

Six alloys were used in this study in order to survey as wide a range of strengthening mechanisms as possible. One was a 99.99% "pure aluminum", supplied by the ALCOA company. This purity was chosen as the highest purity that was readily available at reasonable cost. The composition of this material is given in the table below.

Table 3.1. Composition of 99.99% Al (wppm)

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
<th>Ga</th>
<th>Mn</th>
<th>Ni</th>
<th>Si</th>
<th>Ti</th>
<th>V</th>
<th>Zn</th>
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<tr>
<td>ppm</td>
<td>&lt;9</td>
<td>&lt;9</td>
<td>34</td>
<td>&lt;120</td>
<td>&lt;9</td>
<td>&lt;40</td>
<td>8</td>
<td>&lt;9</td>
<td>&lt;40</td>
<td>2</td>
<td>10</td>
</tr>
</tbody>
</table>

All values except Al are in parts per million by weight.

A second alloy was a "commercial purity" aluminum with 0.17w/oFe, 0.072w/oSi, 0.025-0.035w/oTi, 0.011w/oV and all others <0.002w/o (chemical analysis supplied by ALCOA). This alloy has been developed by researchers at ALCOA to be comparable to the 1100 grade of commercial purity aluminum, while having a well defined and controlled impurity content. The composition means that it is the "baseline" alloy with which many of the commercially valuable alloys can be compared, for example the 3000 series alloys. The term "commercial purity aluminum" will be applied to this material in order to clearly distinguish it from the 99.99% Al; in the figures it is referred to as Al-0.17Fe-0.07Si. The third alloy was based on the same "commercial purity" as given above but with 1 w/o Mg added. A fourth alloy contained 2.05 w/o Mg which is, approximately, the maximum amount of Mg that is soluble at room temperature. This alloy was made by casting 99.99% Al with the appropriate alloy addition. A fifth alloy contained 0.78 w/o Mn and was
also made with the 99.99% Al. This alloy was processed to precipitate as much of the Mn as possible in order to produce a coarse dispersion. The sixth alloy, also made from 99.99% Al, contained 4.6 w/o Cu and was tested in two conditions of heat treatment, solutionized and over-aged.

III.2b Thermomechanical Processing of Alloys

The commercial purity alloy and its 1%Mg counterpart were received from ALCOA in the form of extruded rod, 38mm in diameter. This rod was machined into the various forms required for mechanical testing and each machined part was annealed at 530°C for 5 minutes. This heat treatment produced a reasonably equiaxed recrystallized grain shape with a mean size of about 100 μm. The Fe content of the alloy was largely precipitated during the casting of the original ingot in the form of coarse eutectic. The extrusion processing broke up the second phase particles, producing stringers of particles which are very evident in electropolished metallography specimens viewed with Nomarski contrast, Fig. 3.1.
Fig. 3.1 undeformed structure of the commercial purity aluminum in a section containing the torsion axis and the prior extrusion axis, showing stringers of iron containing constituents.
The 99.99% Al and the three alloys based on it underwent a different processing. All four alloys were cast in the form of cylindrical billets 115mm in diameter. The castings were homogenized if they contained alloy additions. The Mg alloy was annealed for 2 days at 590°C. The Cu alloy was homogenized at 535°C for 24 hours and the Mn alloy was homogenized at 640°C for 24 days. Then the cylinders were compressed on axis to approximately 65% strain. The 99.99% Al and the 2%Mg alloy were recrystallized at 380°C for one hour. The Cu alloy was recrystallized at 535°C for 10 minutes, the high temperature being required to avoid precipitation. The Mn alloy was annealed at 350°C for 1 month to precipitate the Mn in the form of coarse particles of the equilibrium phase, Al₆Mn. The expected volume fraction of precipitate for 0.78w/o Mn is 1.8% since conductivity measurements indicated that 68% of the manganese was precipitated. It transpired during the course of this study that the ease with which Mn precipitates in Al depends very much on levels of third elements such as Fe and Si. The Al-Mn system is still under study and the results will be reported elsewhere.

High purity binary Al-Mn alloys require prior cold work for Mn precipitation to occur at reasonable rates. As noted above, even after very long annealing times an appreciable fraction of the manganese remained unprecipitated. The reason for this is that an annealing temperature that is low enough to ensure a small equilibrium solubility for Mn, ~350°C, results in a low diffusion coefficient, ~2.5.10⁻²⁰ m².s⁻¹. For this diffusion coefficient and 24 days, the expected diffusion distance is of the order of 0.3 μm, which is somewhat smaller than the scale of the cell structure on which the Al₆Mn precipitates appear (see chapter V).
After these annealing treatments, the cylinders were compressed at 90° to the initial axis in an effort to break up the prior structure. Most of this compression was accomplished by rolling which resulted in billets approximately 400mm long by 150mm wide by 28mm thick. After each specimen was machined, a final recrystallization treatment was given. For the 99.99% Al and the 2%Mg alloy the treatment was 1 hour at 380°C. For the Cu and Mn alloys a treatment of 5 minutes at 535°C was used. The Mn alloy would not recrystallize at lower temperatures, presumably because of the presence of the Al₆Mn precipitate. The Cu alloy was recrystallized at the higher temperature to avoid precipitation. In both cases the final grain size of approximately 200 μm was not fine enough to give 10 grains across the smallest dimension of the torsion specimen employed in this study. As a consequence, the results for the Mn and the Cu alloys should be treated with caution though the results do show the expected trends. Optical micrographs of the grain structure of the alloys are given in the section on optical microscopy.

III.3 Test Method

The method chosen for obtaining stress-strain curves at large strains was torsion of short thin-walled tubes. Torsion has the advantage that a continuous stress strain curves can be obtained to very large strains before plastic instabilities cause fracture. The characteristic fracture morphology is that of a crack running around the specimen in a plane perpendicular to the torsion axis. This instability corresponds to the instabilities noted by Tanaka and Spretnak [3,4] in solid bar torsion experiments on high strength steel. The characteristics of failure in torsion were not pursued in this investigation, however.
The specimen design is illustrated in Fig. 3.2 and is known as the Lindholm specimen [3.5]. This design is the same as that employed by Hughes' study [3.3] of nickel alloys except that in most cases a thicker wall was used to compensate for the coarser grain sizes that were obtained in the aluminum alloys as opposed to the nickel alloys. The thin wall tube design permits approximation of uniform strain in the gauge cross-section in contrast to the solid bar specimen where assumptions have to be made in order to convert torque-displacement histories to stress-strain curves. Normally the thin wall design requires the use of a mandrel inside the tube to prevent buckling instabilities and this mandrel renders the torque measurement uncertain because of unknown friction contributions. The short gauge length makes the specimen surprisingly stable in dimension, however. Torsion tests of f.c.c. metals usually lead to a small axial expansion, the Swift effect, which can be explained by the texture development, see for example Canova et al. [3.6]. In the case of the Lindholm tube, however, the constraint of the relatively massive grip ends prevents the hoop contraction that is required for the Swift effect. Therefore the test is effectively a fixed end test where a simple shear is applied to the material. This behavior was verified by measuring the specimen length before and after a torsion test to a shear strain of 3.45 (or a von Mises equivalent strain of 2) and finding no detectable length change, even though the test machine was run in such a manner as to maintain zero net axial load. A further check on the dimensional stability was made by recording shadowgraphs of the specimen before and after this large strain test. Again, no detectable change in shape was recorded. The exception to this is that at very large strains of 6 (von Mises
equivalent), only attained in the commercial purity aluminum, the center of the specimen tended to pull in. This effect is documented in the optical metallography section.
Fig. 3.2 Diagram of the Lindholm tube used for torsion tests.
III.3a.1 Test Machine and Procedures

The test machine used in this study was a multi-axial MTS hydraulic machine with capability for tension, torsion and internal pressurization of tubes. This machine has been fully described elsewhere, Stout et al. [3.7], and no modifications were necessary for the large strain tests described here. The torque was measured by a standard load cell which has a maximum capacity of 1000 Nm. For all the alloys except the Al-Cu alloy, the smallest range of 100Nm was used during the tests. The strain was measured as angular displacement, measured as the change in resistance of a potentiometer that is mechanically linked to the axle of the machine. The rotary actuator used for these tests was limited to about 90° maximum angular displacement which represented a von Mises equivalent strain of about 2. Therefore to reach higher strains it was necessary to unload the specimen, reset the machine and reload to continue the test. In almost all cases the reload transient was small and the flow stress was unchanged after the interruption. The same procedure has been used by Hughes on Ni alloys [3.3] with good success.

III.3a.2 Strain Rates

In what follows, strain rates are given in von Mises equivalent terms unless otherwise stated. The strain rate in shear strain terms can be obtained by multiplying by √3. The use of von Mises equivalent units was occasioned by links to other experimental programs which employ other deformation modes such as compression. For most tests, a single von Mises equivalent strain rate of 4.10^{-3} s^{-1} was used. This choice was largely one of convenience since testing at higher strain rates affords less time for detecting and correcting problems; tests at lower strain
rates take unreasonably long times to attain large strains. In the experimentally accessible range of strain rates between $1.10^{-6}$ s$^{-1}$ and 1.0 s$^{-1}$, no significant changes in material behavior were expected. For investigations of strain rate sensitivity in monotonic tests, the strain rates $4.10^{-4}$, $4.10^{-3}$, $4.10^{-2}$ and $4.10^{-1}$ s$^{-1}$ were used. Tests at strain rates much higher than the latter were not possible with the actuator available. Also, higher strain rates would lead to unacceptable temperature rises if the tests were to be regarded as isothermal tests, as discussed below.

Strain rate sensitivity at constant structure was investigated by changing the strain rate by a factor of 10. The length of time at each strain rate was adjusted so as to accumulate the same amount of strain, approximately, in each segment. At room temperature in unalloyed aluminum there is a noticeable transient in the work hardening rate so in most cases a strain of 0.1 was allowed to accumulate at each strain rate.

III.3a.3 Temperature Control

Tests at the boiling point of nitrogen were very simply achieved by placing a polymer dewar around the lower grip and immersing the specimen in liquid nitrogen. In one case a thermocouple was attached to the specimen near the gauge section and the temperature recorded during a test at the highest strain rate used, a von Mises equivalent of $4.10^{-1}$ s$^{-1}$: no temperature rise was detected. Tests at elevated temperature were achieved by making a hollow ring with holes in the inner cylinder. Hot air was generated by passing air through a coil in a Lindberg tube furnace; the hot air was then concentrated on the specimen by the ring, Fig. 3.3. Although some care was required to maintain the specimen at
the desired temperature, this somewhat crude method had the great merit that very rapid heat-up and cool-down could be achieved. For the tests on unalloyed aluminum at 200°C this was desirable since static recovery is known to be appreciable at half the melting point.
Fig. 3.3 Arrangement of annulus used to deliver hot air to the gauge length of a specimen tested at elevated temperature.
Temperature rises during deformation is a significant issue in large strain testing since the temptation exists to conduct tests at high enough strain rates that achieving high strains does not require long times! As discussed in section II.2c.3, for pure aluminum at least, the saturation stress at the end of Stage III is expected to vary exponentially with temperature. Aluminum is one of the better materials from this point of view because of its high thermal diffusivity and low flow stresses.

A simple calculation was performed to set an upper bound on the temperature rise expected for a given strain rate as follows. It was assumed that all the work of plastic deformation was converted to heat and that this heat was all liberated on a plane perpendicular to the torsion axis and located at the mid-plane of the gauge section. The rate of heat production is then (in Watts) simply the product of the current flow stress (Nm\(^{-2}\)) multiplied by the gauge section volume (m\(^3\)), multiplied by the strain rate (s\(^{-1}\)). It was assumed that the grip sections act as perfect heat sinks at the ambient temperature. Then the area over which heat flow occurs is the axial cross sectional gauge area and the length over which a temperature differential is developed is half the gauge length. The maximum temperature differential calculated in this way is listed as a function of strain rate in Table 3.1 below.
Table 3.2
Maximum Temperature Rise as a function of Strain Rate

<table>
<thead>
<tr>
<th>( \dot{\epsilon} ) (s(^{-1}))</th>
<th>( \Delta T ) (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.15</td>
</tr>
<tr>
<td>2</td>
<td>0.3</td>
</tr>
<tr>
<td>3</td>
<td>1.5</td>
</tr>
<tr>
<td>4</td>
<td>3.0</td>
</tr>
</tbody>
</table>

These calculations were verified by testing a specimen of the 1%Mg alloy at the highest strain rate possible in the machine, a von Mises equivalent of \( \approx 4 \) s\(^{-1}\). The recorded temperature-strain histories are shown in Fig. 3.4. The largest rate of temperature rise was recorded for the thermocouple attached to the gauge section which indicated that the temperature rise would have been 3°C. Since the expected temperature rise varies linearly with strain rate and the maximum strain rate for which data is reported is a factor of ten less than that used for the special test, the conclusion was that the data to be reported below are all isothermal. Aluminum has a higher thermal diffusivity than many of the b.c.c. metals and is therefore likely to permit isothermal tests at higher strain rates.
Fig. 3.4 Temperature rise measured by three thermocouples attached to a torsion sample tested at the highest available strain rate. The thermocouple attached to the gauge section fell off after a shear strain of about 1.6.
III.3b Data Reduction

The results of each test were recorded on a standard X-Y pen recorder as torque versus angular displacement and an example is shown in Fig. 3.5. Each chart was digitized on a Hewlett-Packard microcomputer, model HP86, and the data were converted to shear stress and shear strain as follows. The strain conversion factor is

$$\gamma = (\text{angular displacement, } ^\circ) \frac{<r> \pi}{180^\circ} \left/ \text{(gauge, mm)} \right. \quad (3.1)$$

where $<r>$ is the average of the inner and outer radii and $\gamma$ is the shear strain. For all specimens, the actual angular displacement was measured between two marks scribed parallel to the torsion axis in a lathe. The difference between the actual angular displacement and the chart displacement was as much $2^\circ$ in $80^\circ$ total for the softest alloys. This discrepancy was ascribed to deformation of the hexagonal grip ends during the test. A simple compensation was applied to the strain values by scaling all the strain values by the ratio of the actual to recorded displacements. The torque to shear stress conversion was calculated as

$$\sigma = 1000 \left( \text{Torque, Nm} \right) \frac{<r>}{\pi(r_o^4 - r_i^4)} \quad (3.2)$$

where $r_o$ is the outer radius, $r_i$ is the inner radius and $\sigma$ is the shear stress. It should be noted that the actual stress state at the gauge section must be more complex than a pure shear stress because the test is actually a fixed end test as discussed above. The other stress components are expected to be small compared to the shear stress, however, [3.8], and related to the texture. At large strains, the texture is already well developed and changes very little so little effect on hardening rate would be expected. Also, these stress components are not expected to influence the glide resistance in alloys
with an f.c.c. lattice, as they might in a material with a high Peierls stress.
Fig. 3.5 example of a torque-displacement curve as recorded during a torsion test. This curve was obtained from an Al-1Mg alloy that was subjected to strain rate changes (section III.4d) at the points indicated by the arrows. The figure illustrates the strain rate sensitivity of this alloy.
III.3c Hardening Rates

Calculation of the hardening rate requires a differentiation of the stress-strain curve. This was achieved in the following manner. The digitized curve is a series of about 40 stress-strain coordinate pairs, the number of digitized points depending on the shape of the curve. Groups of 5 or 7 points were taken, incrementing one point at a time, and a quadratic fitted to the group of points. At the central point of the group, a value for the stress was calculated from the fitted quadratic and plotted as a point on the graph of the actual digitized data. At the same central point, a value for the differential, $d\sigma/d\varepsilon$, was calculated from the fitted quadratic. The results of one such fitting is shown in Fig. 3.6. As with all such procedures it is easy enough to fit the curve itself but a subjective judgment is required as to whether the differentiated data is smooth enough to be useful but not so smooth as to have erased important, physically significant features. The beginning and end of each set of data required fitting to fewer points in the obvious way, i.e. if the group was 7 points, the first data point was fitted with only the first point and the next 3 points.

Although the hardening data is displayed in this thesis as hardening rate versus stress for reasons outlined in the review, it should be noted that for materials such 99.99% Al tested at room temperature, the transition from rapid decrease to slow decrease of hardening rate is much more apparent in the plot of hardening rate versus strain shown in Fig. 3.6.
Fig. 3.6 results of digitizing a torque-displacement curve; the continuous line is a digitized stress-strain curve. The set of individual points that track the stress-strain curve show the results of fitting quadratic curves to the data, taking sets of 5 points at each data point, 2 on either side of the data point. The set of points that describe the decreasing hardening rate with increasing strain show the results of differentiating the quadratic at each point.
III.3d Taylor Factors

The stress-strain data are reported as macroscopic shear stress versus shear strain for the reason that this is the measured data and avoids the assumptions that are necessary for calculating equivalent stresses and strains. In order to be able to compare one material with another and to remove the effect of variation of shear modulus with temperature, all stresses are divided by the shear modulus before plotting the hardening data. The stress-strain data are also resolved onto critical shear stresses on a slip system in order to facilitate eventual comparison with other deformation paths, such as wire-drawing. This is done by dividing the shear stress by the appropriate Taylor factor and multiplying the shear strain by the same Taylor factor. The Taylor factor is found to vary so little in simulation of torsion texture development, Canova et al. [3.8], that a single value of 1.55 was used to convert measured stress-strain data to resolved values. The evolution of the Taylor factor for torsion is shown in Fig. 3.7 as calculated by the current version of the Los Alamos Polycrystal Plasticity code; 500 individual grains were used to simulate a polycrystal. The simplification of using a single value for the Taylor factor is not valid for, e.g., wire drawing to moderate strains where the evolution of the texture leads to Taylor factor changes on the order of 10%. Note also that hardening rates calculated from resolved quantities vary as $H^2$ rather than $M$. The resolved stresses and hardening rates are divided by the temperature corrected shear modulus, for which values were taken from Kocks [3.9] and Mecking [3.10]. It was found that a maximum value of $\sigma/G$ of 0.01 and a maximum value of $\theta/G$ of 0.05 provided useful plots for plotting the Stage III behavior and the
transition to Stage IV behavior. A reduced hardening rate of 0.05 is approximately that expected for Stage II work hardening rates in f.c.c. metals.
Fig. 3.7 Theoretical calculation of the Taylor Factor in torsion as a function of strain, as calculated by the Los Alamos Polycrystal Plasticity texture simulation code.
As a check on the use of a single Taylor factor, the resolved hardening rate was plotted as a function of resolved stress for the Al-1Mg alloy, tested at room temperature at a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$, Fig. 3.8. One line shows the data for which a single Taylor factor was used and the other curve shows the results of taking account of the variation of Taylor factor with strain, Fig. 3.7. It appears that the two curves are close enough to be within the scatter of the experimental data.
Fig. 3.8 Comparison of the results of calculating hardening rate versus stress with either a constant Taylor factor or with a Taylor factor that evolves with strain.
III.4 Results of Torsion Tests

III.4a Small Strain Behavior

Two torsion tests were performed with strain gauges on the commercial purity and the 1% Mg alloys as a check on the small strain behavior. The resulting stress-strain curves are plotted in Fig. 3.9 and show the expected difference in yield stress between the 1% Mg alloy and the soft commercial purity alloy. The 0.2% offset yield strengths were 14.3 MPa for the commercial purity Al and 29.3 MPa for the 1%Mg alloy. This illustrates that the alloy addition has only a small effect on the as-annealed strength of aluminum, by comparison with the effects of an precipitation hardening.
Fig. 3.9  stress-strain behavior at small strains from strain gauged specimens: a) commercial purity aluminum, b) Al-1Mg.
III.4b Reproducibility of Data

The commercial purity Al and the 1%Mg alloy were machined into Lindholm torsion specimens with two different wall thicknesses, 1.27mm (the standard thickness in this study) and 0.76mm. For each alloy the stress-strain curves are plotted in Fig. 3.10. In both cases the results were very close which suggests that the stress-strain data are not sensitive to the sample geometry.

The ideal mechanical testing program would include enough repeated tests that errors can quantified as the standard deviation of some measured quantity. The time, expense and, above all, limited supply of material prevented such an approach. For certain conditions, however, many tests were run with the same material at the same temperature and strain rate. Fig. 3.11 shows four shear stress-strain curves for the Al-1Mg alloy tested at room temperature and the standard von Mises equivalent strain rate of $4.10^{-3}$ s$^{-1}$. The similarity of the results suggests that the test is sufficiently reproducible that the results discussed below, where often only a single specimen was tested for each condition, are significant.
Fig. 3.10 Comparison of stress-strain curves obtained from Lindholm tubes with 0.762mm wall, "THIN", and 1.27mm wall, "THICK":

a) commercial purity aluminum, b) Al-1Mg.
Fig. 3.11 Four shear stress–shear strain curves for Al-1Mg tested at room temperature and a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$. 
III.4c Effect of Alloying at Constant Test Temperature

Monotonic torsion data were obtained for all the alloys at room temperature, Fig. 3.12A and at 77K, Fig. 3.13A, at the standard strain rate of $4 \times 10^{-3} \text{ s}^{-1}$. For certain alloys, the room temperature data shows a clear linear Stage IV over large strains. For the 99.99% Al and the commercial purity Al, however, the hardening rate appears to decrease continuously. At the lower temperature, all alloys show extended hardening at large strains. The hardening rate is plotted versus stress for the room temperature data in Fig. 3.12B. The Mg alloys and the Cu alloy show a clear transition to Stage IV behavior at about $\theta_0/\Gamma = 1.8 \times 10^{-4}$. Once in Stage IV, the hardening rate is maintained almost constant in the Mg alloys for a range of stress before starting to decrease. The unalloyed materials, however, do not show a clear Stage IV although there is a definite departure from Voce Law behavior at large stresses.

The effect of small amounts of solute is now dramatic: at the end of Stage III, a shear strain of 1, the data shows a difference of 43 MPa between the commercial purity and the 1%Mg alloy whereas the difference in 0.2% yield stresses was only 15 MPa. The effect of Stage IV is to magnify these differences. For the largest strains achieved in the commercial purity and the 1Mg alloys, the difference in flow stress was more than 60 MPa. The flow stress of the Al-2%Mg alloy rose to the same levels as observed at small strains in the age-hardening Cu alloy. The Al-5Mg illustrates the point that the hardening achieved with a solute addition is not simply proportional to the atomic concentration; the Al-5Mg has a similar flow curve to that of the over-aged Al-4.6Cu alloy.
The latter alloy, however, has only about 2a/o Cu as opposed to the 5a/o of the Mg alloy.

The Al-Cu alloy was used to investigate the effect of strain hardening on an age-hardenable alloy. Figure 3.12A shows that both conditions of the alloy reach high strength levels at small strains. The plot of hardening rate versus stress, Fig. 3.12B shows that the overaged alloy exhibits a distinct Stage IV with increasing hardening rate with increasing stress, in contrast to the solute hardened alloys where the hardening rate decreases slowly during Stage IV. The solutionized alloy was obtained by annealing at 210°C for five minutes and quenching after the normal high temperature solutionizing treatment (30 minutes at 530°C). This low temperature reversion treatment puts any GP zones that may have formed during room temperature aging back into solution without raising the vacancy concentration in the material. The over-aged condition was obtained by annealing at 225°C for 24 hours, followed by a furnace cool.
Fig. 3.12A Shear stress-shear strain curves measured at a shear strain rate of $6.9 \times 10^{-3} \text{s}^{-1}$ for various aluminum alloys as noted on the figure.
Fig. 3.12B Hardening rate plotted versus stress derived from the data of 3.12A, reduced by the appropriate shear modulus for the deformation temperature and by a Taylor Factor, =1.55, in order to obtain quantities appropriate to slip on a single slip system.
Fig. 3.13A Shear stress- shear strain curves measured at a (shear) strain rate of $6.9 \times 10^{-3}$ s$^{-1}$ for various aluminum alloys as noted on the figure, tested in torsion at the boiling point of liquid nitrogen, 77K.
Fig. 3.13B Hardening rate plotted versus stress derived from the data of 3.13A.
The results of various tests at 77K are shown in Fig. 3.13A. They show that strain hardening is more effective at the lower temperature and that the alloys are less affected by the change in deformation temperature than are the unalloyed materials. The plots of hardening rate versus stress, Fig. 3.13B show that Stage III is nearly linear for all the alloys; this in contrast to the room temperature data which showed curved plots for the unalloyed materials. The transition from Stage III to Stage IV is obvious in all alloys except the Al-Mn alloy. The reduced hardening rate at which the transition occurs is approximately $2.10^{-4}$ s$^{-1}$. The hardening rate decreases slowly as Stage IV proceeds, as observed at room temperature. The Al-1Mg alloy at both 293K and 77K exhibits a small increase in hardening rate at the transition; this phenomenon is visible on the torque-displacement graphs as a slight reverse curvature at the transition.

The phenomenon of increased alloy hardening as strain hardening is applied has been documented by Dorn et al. [3.11] and Schmidt and Miller [3.12]. The data presented here reinforce the idea that for solute hardening in particular, small strengthening effects in annealed materials are magnified to large flow stress differences at moderate strains, even near the end of Stage III. At 0.3Tm in aluminum, the differences are further magnified during Stage IV because of the weakness of Stage IV in unalloyed aluminum. No further discussion of this topic will be attempted in this thesis but it appears that there is a lack of theory to describe the synergistic effects of strain and solute hardening.
III.4d Strain Rate Changes at Constant Structure

Tests were performed at room temperature to determine the strain rate sensitivity at constant structure. The procedure used was that of switching between a strain rate of $2.10^{-3}$ and $2.10^{-2}$ s$^{-1}$ (von Mises equivalent) for a time at each strain rate such that the same strain elapsed at each rate and so that the mean strain rate was approximately that of the "standard" strain rate employed in other tests, $4.10^{-3}$ s$^{-1}$. The results of the tests are plotted as stress change divided by the logarithm of the strain rate ratio versus flow stress. The data for the 99.99% aluminum, Fig. 3.14, shows the anticipated trend of an initial linear rise, increasing more rapidly at large stresses. The results for the commercial purity Al are similar, Fig. 3.15.

The Al-Mn alloy, Fig. 3.16, showed a higher rate sensitivity at all stresses. Most of the manganese is precipitated and that which remains in solution is not expected to be mobile at room temperature. Since the flow stresses achieved in this material were higher than in the previous two alloys and the curve is simply an extension of Fig. 3.15, it appears that the higher rate sensitivity is just a consequence of the higher stresses.

The torque-displacement curves for the Al-Mg alloys at room temperature exhibited a roughness characteristic of serrated flow due to dynamic strain aging, Fig. 3.5. The results of strain rate change experiments from the two alloys containing magnesium are not given here because in both cases the change in flow stress upon changing the strain rate by a factor of ten was indistinguishable from the noise in the stress-strain curves. Moreover, this was the case up to the largest strain in the test of about 2. The apparent lack of rate sensitivity is
not surprising in view of the fact that Al-Mg alloys exhibit dynamic strain aging. The temperature range in which this occurs depends on the Mg content, Guillot [3.14], but the peak of the effect is generally close to or a little below room temperature. The result that the rate sensitivity was approximately zero even at large strains is somewhat surprising in that the dislocation structure is expected to make an increasing contribution to the rate sensitivity with increasing dislocation density, Kocks et al. [3.15]. The anticipated behavior, then, had been negative rate sensitivity at small flow stresses changing over to positive rate sensitivity at large flow stresses. Further strain rate change experiments are reported in the section on reverse torsion experiments, III.4f.
Fig. 3.14 Strain rate sensitivity of 99.99% Al, measured by jumps in strain rate from shear strain rates of $1.7 \times 10^{-3}$ to $1.7 \times 10^{-2}$ s$^{-1}$ and back again. The circles represent increases in strain rate and the squares represent decreases in strain rate.
Fig. 3.15 Strain rate sensitivity of commercial purity aluminum. The circles represent increases in strain rate and the squares represent decreases in strain rate.
Fig. 3.16 Strain rate sensitivity of Al-0.8 wt% Mn. The circles represent increases in strain rate and the squares represent decreases in strain rate.
III.4e Effect of Different Strain Rates

In discussions that follow of the rate sensitivity of the evolution of the flow stress, the definition of this quantity is taken as follows. Since comparisons of flow stresses at saturation are made implicitly at a constant (zero) hardening rate, the method used in this work to describe the strain rate sensitivity of flow stress evolution is to make comparisons at the same hardening rate. On a plot of hardening rate against stress, the graphical construction would be to draw a horizontal line at a given hardening rate and mark the intersections with the data. Where saturation stresses are of interest, for Stage III, extrapolate the Stage III data to zero work hardening rate and obtain a nominal saturation stress, $\tau_{s1}$. The rate sensitivity of the saturation flow stress at the end of Stage III is then $d(\tau_{s1})/d\ln(\dot{\varepsilon})$. For Stage IV follow the same procedure is followed except that the extrapolation is from the Stage IV data.

III.4e.1 at Room temperature

The effect of conducting constant strain-rate tests at different strain rates is shown for the commercial purity aluminum and the 1% Mg alloy at room temperature in Figs. 3.17 and 3.18 respectively. Four different strain rates were used, $4.10^{-4}$, $4.10^{-3}$, $4.10^{-2}$ and $4.10^{-1}$ s$^{-1}$. Each figure has a) the shear stress plotted versus shear strain and b) the reduced hardening rate versus the reduced shear stress, as discussed above. The commercial purity aluminum exhibits a positive rate sensitivity of the evolution of the flow stress which is the same direction as its constant structure rate sensitivity. The magnitude of the rate sensitivity is somewhat larger at high strains than it is in Stage III. The 1% Mg alloy, on the other hand, exhibits a negative rate
sensitivity which again is in the same direction as its constant structure rate sensitivity. The differences in Stage IV are so small as to be within the scatter of the test method.
Fig. 3.17A Shear stress-shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, $6.9 \times 10^{-2}$, $6.9 \times 10^{-3}$ and $6.9 \times 10^{-4}$ s$^{-1}$ for commercial purity aluminum tested in torsion at room temperature.
Fig. 3.17B Hardening rate plotted versus stress derived from the data of

3.17A. The inset plot shows a magnified view at low

hardening rates.
Fig. 3.18A Shear stress-shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, $6.9 \times 10^{-2}$, $6.9 \times 10^{-3}$ and $6.9 \times 10^{-4}$ s$^{-1}$ for Al-1Mg tested in torsion at room temperature. The curve for the highest strain rate intersects the data for the other strain rates.
Fig. 3.18B Hardening rate plotted versus stress derived from the data of 3.18A.
III.4e.2 at 77K

Figure 3.19 shows the results of tests at two different strain rates for commercial purity aluminum. The factor of 100 difference leads to appreciable differences in flow stress at the same strain level. In contrast to the room temperature data, there is a distinct transition to Stage IV behavior, albeit with falling hardening rate in Stage IV. It is immediately apparent that dynamic recovery is far less effective at this low temperature than it was at room temperature. Data for the same pair of strain rates for the 1% Mg alloy is plotted with that for the 2% Mg alloy at the lower strain rate only in Fig. 3.20. At this low temperature, the strain rate sensitivity (of the evolution of the flow stress) in the 1% Mg alloy, as measured by the tests at two different strain rates, is positive and of the same order of magnitude as the commercial purity aluminum. This change in sign of the rate sensitivity is easily understood in terms of the large body of work on dynamic strain aging in Al-Mg alloys, e.g. Guillot [3.14]. Guillot's work showed that the maximum effect of Mg in pinning dislocations occurs near room temperature. At low enough temperatures such as 77K, the Mg atoms are not sufficiently mobile to pin dislocations effectively. At high enough temperatures such as 473K, see below, enough thermal energy is available that the pinning is no longer effective.
Fig. 3.19A Shear stress - shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, and $6.9 \times 10^{-3}$ s$^{-1}$ for commercial purity aluminum tested in torsion at 77K.
Fig. 3.19B Hardening rate plotted versus stress derived from the data of 3.19A.
Fig. 3.20A Shear stress- shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, and $6.9 \times 10^{-3}$ for Al-1Mg, and $6.9 \times 10^{-3}$ s$^{-1}$ for Al-2Mg tested in torsion at 77K.
Fig. 3.20B Hardening rate plotted versus stress derived from the data of 3.20A.
III.4e.3 at 373K

The results of tests at three strain rates, \(4\times10^{-3}\), \(4\times10^{-2}\) and \(4\times10^{-1}\) s\(^{-1}\), are shown in Fig. 3.21 for the commercial purity aluminum. The behavior is similar to that at room temperature albeit showing lower (extrapolated) saturation stresses. Note that even at a homologous temperature of 0.4, very large strains are required to approach saturation in this high stacking fault energy material. Figure 3.22 shows the results of two tests on the 1%Mg alloy at strain rates of \(4\times10^{-3}\) and \(4\times10^{-1}\) s\(^{-1}\). The intermediate strain rate was not used since the material appeared to be essentially rate insensitive. The Stage IV behavior at this temperature is very similar to that observed at room temperature.
Fig. 3.21A Shear stress- shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, $6.9 \times 10^{-2}$, and $6.9 \times 10^{-3}$ s$^{-1}$ for commercial purity aluminum tested in torsion at 373K.
Fig. 3.21B Hardening rate plotted versus stress derived from the data of 3.21A. The inset plot shows a magnified view at low hardening rates.
Fig. 3.22A Shear stress- shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, and $6.9 \times 10^{-3}$ s$^{-1}$ for Al-1Mg tested in torsion at 373K.
Fig. 3.22B Hardening rate plotted versus stress derived from the data of 3.22A.
III.4e.4 at 473.

The same trio of strain rates was applied to both the commercial purity aluminum, Fig. 3.23 and the 1%Mg alloy, Fig. 3.24. The differences due to strain rate are small in Stage III but more noticeable in the large strain regime. The commercial purity aluminum does not show any pronounced Stage IV behavior. The Al-1Mg alloy shows the interesting result that the lowest strain rate exhibits saturation at moderate strains whereas the highest strain rate exhibits a Stage IV. The plots of hardening rate versus stress for this alloy also show some curvature in Stage III, as does the unalloyed aluminum at lower temperatures.
Fig. 3.23A Shear stress-shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, $6.9 \times 10^{-2}$, and $6.9 \times 10^{-3}$ s$^{-1}$ for commercial purity aluminum tested in torsion at 473K.
Fig. 3.23B Hardening rate plotted versus stress derived from the data of 3.23A. The inset plot shows a magnified view at low hardening rates.
Fig. 3.24A Shear stress-shear strain curves measured at (shear) strain rates of $6.9 \times 10^{-1}$, $6.9 \times 10^{-2}$, and $6.9 \times 10^{-3}$ s$^{-1}$ for Al-1Mg tested in torsion at 473K. Note the apparent saturation at the lowest strain rate.
Fig. 3.24B Hardening rate plotted versus stress derived from the data of 3.24A.
III.4f Torsion-Reverse Torsion Tests

The torsion test with the Lindholm tube is unusually suitable for performing strain reversals at large strains because of the stability of the specimen dimensions. Also the texture change on reversing the strain was shown by Backofen [3.16] to be small, in contrast to the differences between tension and compression textures. Several tests were conducted to investigate the sensitivity of the large strain hardening behavior to strain reversals. Figures 3.25-3.32 show the shear stress-shear strain curves for these tests. The hardening rate has also been plotted as a function of the strain in order to illustrate the effects of reversing the sign of the deformation. The two curves can be easily distinguished by the fact the stress generally increases with increasing strain whereas the hardening rate decreases. In each case the sign of the stress and the strain has been reversed for the reverse torsion part of the curves so that the reverse deformation appears as a continuation of the forward deformation. The strain at which the deformation was reversed is apparent as a reloading point at half the maximum strain. In most cases the maximum forward strain was a shear strain of 3.45 or a von Mises equivalent strain of 2.
Fig. 3.25 Shear stress-shear strain for 99.99% Al tested in torsion to a shear strain of 1.3 and then in reverse torsion, at a shear strain rate of $1.7 \times 10^{-2} \text{ s}^{-1}$. The hardening rate has been plotted as a function of strain on the same axes to illustrate the transient decrease of hardening on reversing the straining direction.
Fig. 3.26 Shear stress- shear strain for 99.99% Al tested in torsion to a shear strain of 2.6 and then in reverse torsion, at a shear strain rate of $1.7 \times 10^{-3} \text{ s}^{-1}$. The hardening rate becomes negative during the transient.
Fig. 3.27 Shear stress-shear strain for 99.99% Al tested in torsion to a shear strain of 3.5 and then in reverse torsion, at a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$. The hardening rate becomes negative during the transient. The roughness of the hardening rate curve is a consequence of the jumps in strain rate used to investigate the strain rate sensitivity.
Fig. 3.28 Shear stress- shear strain for commercial purity Al tested in torsion to a shear strain of 3.5 and then in reverse torsion, at a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$. The hardening rate becomes negative during the transient.
Fig. 3.29 Shear stress- shear strain for Al-0.8Mn tested in torsion to a shear strain of 3.5 and then in reverse torsion, at a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$. The hardening rate becomes negative during the transient and there is a small Bauschinger effect, i.e. drop in flow stress.
Fig. 3.30 Shear stress - shear strain for Al-1Mg tested in torsion to a shear strain of 3.5 and then in reverse torsion, at a shear strain rate of 6.9 \times 10^{-3} \text{ s}^{-1}. In contrast to the unalloyed aluminum, the hardening rate is low during the transient but no work-softening occurs.
Fig. 3.31 Shear stress- shear strain for Al-2Mg tested in torsion to a shear strain of 3.5 and then in reverse torsion, at a shear strain rate of $6.9 \times 10^{-3} \text{ s}^{-1}$. As with the Al-1Mg the hardening rate is low during the transient but no work-softening occurs.
Fig. 3.32 Shear stress- shear strain for overaged Al-Cu tested in torsion to a shear strain of 1.7 and then in reverse torsion, at a shear strain rate of $6.9 \times 10^{-3}$ s$^{-1}$. The hardening rate remains small during a transient that lasts for a large strain interval.
In all the Bauschinger tests, the hardening rate after the reversal returned to approximately the same value as observed before the strain reversal. There was also a transient decrease in the hardening rate observed in all the materials. For tests at a maximum forward shear strain of 1.3, Fig. 3.25, 2.6, Fig. 3.26 and 3.5, Fig. 3.27, of the 99.99% aluminum, the severity of the decrease in hardening rate appeared to increase with strain. The largest maximum strain, Fig. 3.27, shows irregular flow curves as a result of strain rate changes performed to measure the rate sensitivity before and after the change in straining direction. In this test, work softening occurred after the reversal. The same transient work softening occurred in the commercial purity aluminum, Fig. 3.28, and the Al-Mn alloy, Fig. 3.29. The transients in the Al-1Mg, Fig. 3.30, and the Al-2Mg alloy, Fig. 3.31, are less obvious and no work softening was observed. The Al-Cu alloy, Fig. 3.32, showed a large transient decrease in work hardening rate before eventually returning to the level observed in forward deformation.

In order to investigate the effect of strain reversal on the constant structure rate sensitivity, strain rate change tests were conducted on the 99.99% Al, commercial purity Al, 1%Mg and 2%Mg alloys. The magnesium containing alloys showed the same lack of stress changes on reversing the shear direction as they had in monotonic straining. The curve for the 99.99% Al, Fig. 3.33, showed that the rate sensitivity remains essentially constant after the strain reversal. This is in spite of considerable variation in strain hardening rate.
Fig. 3.33 Magnified view of the von Mises equivalent stress-strain curve for 99.99% Al with changes in strain rate between $1.7 \times 10^{-3}$ and $1.7 \times 10^{-2}$ s$^{-1}$. The torsion was reversed at the point indicated by the arrow.
III.5 Summary

The results of the torsion experiments can be summarized as follows:

a. For all the alloys investigated, with the possible exception of the Al-Mn alloy, an abrupt transition from Stage III to Stage IV behavior was observed. This transition occurred in some cases where the Stage III $\theta-\sigma$ plots were curved, meaning that a simple Voce Law behavior was not followed in Stage III.

b. There was a positive correlation between high strength and the extent of Stage IV, i.e. at low temperatures and high alloy contents, Stage IV continues until fracture intervenes. As the temperature of deformation is increased and/or the alloy content is decreased, there comes a point at which the transition to Stage IV is not sharp and the hardening rate during Stage IV decreases near to zero before fracture intervenes.

c. Alloying with a soluble element raises the maximum temperature at which Stage IV behavior can be clearly observed.

d. Alloying with an insoluble element where a precipitate has been formed has a variable effect on Stage IV that depends on the nature of the precipitate;

e. As found in many other experiments, the work hardening rate in Stage IV is approximately constant at a level of $2 \times 10^{-4}$; this value is a hardening rate reduced by a Taylor factor to a value appropriate to slip on a single slip system.

f. Strain reversals do not eliminate Stage IV or change the (constant structure) rate sensitivity, though transients do occur
in the work hardening rate. The transient reduction in hardening rate occurs over strains as large as 0.75 shear strain.

g. The strengthening effect of solute hardening increases with strain. This effect is magnified at room temperature in aluminum because pure aluminum exhibits only a vestigial Stage IV whereas Stage IV in the Al-Mg alloys continues until fracture intervenes.

h. For the alloys investigated and allowing for the dangers of extrapolation, the saturation stress in commercial purity Al is approximately proportional to the logarithm of the strain rate of the deformation that was used to arrive at that saturation stress.

III.6 References


Chapter IV. Optical Microscopy and Texture Results

This chapter describes the following aspects of the optical metallography that was performed as part of this work: 1. the initial grain structures of the materials used, 2. the homogeneity of deformation in the particular torsion specimen used for this work, 3. the deformed microstructures at the grain level, 4. a study of slip line markings on a polished surface at a strain level of 2, 5. a brief description of the crystallographic textures that are characteristic of the torsion test, both experimental and theoretical.

IV.1 Initial Grain Structures

The undeformed grain structures of the various alloys are shown in Figures 4.1-4.4. The degree of directionality in the structures varied between alloys. The grain shapes of the 99.99% Al, Fig. 4.1a and the Al-2Mg alloy, Fig. 4.1b, were the closest to equiaxed. Fig. 4.1b also shows the transition in grain structure from undeformed in the grip section to deformed in the gauge section. The commercial purity aluminum is shown in two different sections in Fig. 4.2. Figure 4.2a shows a section cut parallel to the torsion axis and parallel to the extrusion axis of the prior thermomechanical processing. Both the commercial purity aluminum and the 1%Mg alloy had an appreciable iron content (0.17%) which was precipitated during solidification, presumably as the Al-Fe eutectic. Subsequent extrusion "stringerred" the iron containing constituents as is evident in the sections that contain the extrusion axis. The stringers in Fig. 4.2a are parallel to the extrusion axis and parallel to the subsequent torsion axis. These stringers of particles were also observed in the transmission electron microscope (TEM). The specimens were
machined such that torsion axis was parallel to the extrusion axis. The
grain structure of the Al-1Mg alloy was very similar to that of the
commercial purity aluminum as might be expected for having the same iron
content, Fig. 4.3, except that the grains tended to be elongated in a
direction parallel to the torsion axis. The Al-Mn, Fig. 4.4a, and Al-Cu,
Fig. 4.4b, alloys had grain structures that were elongated in the plane
of the last rolling deformation. Since the torsion specimens were
machined with the torsion axis parallel to this plane, the effect of the
admittedly coarse grain size was minimized.

The significance of this observation is that for a mechanical test
to be a true test of a polycrystal, a reasonably high proportion of the
grains must be not on the surface. If the criterion is chosen as 80% of
grains being in the interior, i.e. not in contact with a free surface,
then there must be at least twenty grains through the thickness of a
tensile sample (4n²/n³). In the case of the Lindholm tube used in this
work, the number of grains across the width of the gauge section can be
relaxed to ten because of the smaller fraction of exposed surface. This
smaller fraction arises because any given "slice" of the gauge section
only its inner and outer sides exposed (two out of six) whereas an
equivalent "slice" of a (square) tensile sample has four out of six
sides exposed.
Fig. 4.1  a) Undeformed grain structure of 99.99% Al, section normal to radial direction, as electropolished, image taken using Nomarski contrast.

b) grain structure of Al-2Mg alloy, section normal to tangential direction, anodized surface, image taken using crossed polars. Note gradation of structure from undeformed in the grip section (right) to deformed in the gauge section (left), $\gamma=3.46$. 
Fig. 4.2 a) Undeformed grain structure of commercial purity aluminum, channeling contrast conditions in a CAMSCAM scanning electron microscope. Iron containing second phase particles appear as stringers of bright points. The stringers are parallel to the extrusion axis and to the torsion axis.

b) material as a), section cut normal to the torsion axis, anodized surface viewed at 50X with crossed polars.
Fig. 4.3 Undeformed grain structure of Al-1Mg alloy.

a) torsion axis and prior extrusion axis parallel to stringers

b) section cut normal to the torsion axis.
Fig. 4.4 a) Undeformed grain structure of Al-Mn alloy, channeling contrast conditions in an SEM.  
b) undeformed grain structure of Al-Cu alloy, channeling contrast, showing irregular grain shapes.
IV.2 Homogeneity of Deformation

A large amount of the literature on large strain studies in the last ten years has discussed such aspects of heterogeneous plastic deformation as microbands and shear bands. Microbands will be discussed further in the next chapter on TEM studies but, anticipating, they appear to be uncommon in aluminum alloys. Shear bands occur copiously in many heavily rolled metals, see e.g. Hatherly [4.1], and they have been observed in torsion by Tanaka and Spretnak [4.2]. The failure mode observed in the torsion tests used in this work was invariably the propagation of a sharp crack whose plane was perpendicular to the torsion axis (z-axis in some figures). Such cracks run in the same plane as the strain localization features observed by Tanaka and Spretnak. This failure mode was not investigated in this work though it is undoubtedly of interest. In general, the failure appeared to occur very rapidly and the microstructures shown below do not show obvious signs of cracks or precursors to cracks in specimens that were not strained to failure. Another general observation on failure is that the single phase alloys could be strained to larger strains than the two-phase alloys. The single phase alloys could be twisted to von Mises equivalent strains of 5 or more whereas the Al-Mn and Al-Cu alloys could not be twisted to strains much greater than 2.

Figure 4.5 shows the microstructure of the 99.99% Al deformed to a shear strain of 1.8. A section normal to the radial direction is shown in Fig. 4.5a and a section normal to the torsion axis in Fig. 4.5b. The grains have become elongated in the expected manner and slip markings are evident in planes that are perpendicular to the torsion axis (compare the two cross-sections). At higher strains in the same
material, Fig. 4.6a, the grains are highly elongated. The Al-2Mg alloy at a slightly higher strain, Fig. 4.6b, shows a very similar structure. At these large strains, the gauge section shape tends to "suck in" at a shear strain of 10.6 in the commercial purity alloy, Fig. 4.7. This change in gauge section shape suggests that the stress-strain results in the commercial purity aluminum should be treated with caution at extreme strains.

The uniformity of deformation can be checked to some extent by scribing a line on the specimen surface, deforming and then photographing the result. The results of such a test are shown in Fig. 4.8 as a sequence of strains, including an experiment where the specimen was reverse twisted to a net strain of zero as indicated by marks on the grip ends. The appearance of the line as scribed is shown in Fig. 4.8a, followed by a shear strain of 1.7, Fig. 4.8b, a shear strain of 3.46, Fig. 4.8c, and lastly zero net strain, Fig. 4.8d after the forward strain shown in the previous figure, Fig. 4.8c. The lines appear to be very straight at both forward strains with any irregularities being on the grain scale. The apparent angle of the lines on the shoulders is an artifact of the curvature of the specimen. At zero net strain, the line is not completely straight as there is some reverse curvature in the transition region between the grip sections and the gauge length. This occurs because a small amount of deformation inevitably takes place in the transition region, causing work hardening there; on reversing the sense of twist, this work hardened region does not untwist. When the possibilities for non-uniform flow are considered, it is perhaps surprising that the zero net strain structure is so uniformly strained.
Fig. 4.5 a) As electropolished surface of 99.99% Al, deformed to a shear strain of 1.7, section cut normal to the torsion axis.

b) as for a), section cut normal to the radial direction, showing slip markings parallel to the radial direction.
Fig. 4.6 a) Radial section of 99.99% Al, deformed to a shear strain of 6.9, anodized surface viewed with crossed polars.

b) Radial section of Al-2Mg, deformed to a shear strain of 8.7, anodized surface viewed with crossed polars.
Fig. 4.7 a) Commercial purity Al at a shear strain of 10.6, section cut normal to the tangential direction, showing pulling in of the gauge section.
Fig. 4.8a) Showing a line scribed on a torsion sample that is parallel to the torsion axis, initially.
b) showing the same specimen after a shear strain of 1.7; the hour-glass shape of the deformed gauge section is an artefact of the lighting conditions.
Fig. 4.8  c) A different specimen twisted to a shear strain of 3.46, showing uniform deformation.

d) the same specimen as a) and b) after being twisted to a shear strain of 3.46 and then untwisted to zero net displacement, showing that the line very nearly returns to its initial position.
IV.4 Slip Line Study

For all six alloys used in this work, a slip line study was performed near a von Mises equivalent strain of 2 or a shear strain of 3.46. The sequence of actions was a) strain to 1.95, b) machine and electro-polish a flat section on the gauge section, c) strain a further 5%, d) photograph the polished flats, e) reverse the strain by 5% to 1.95 net strain and f) photograph the same area(s). The exception to this was the Al-Cu alloy (overaged condition) where the strain levels were chosen as 0.95-1.00-0.95 because of the limited ductility of this material. The strain level was chosen as one that put all materials well into Stage IV and the large strain regime. The reason for performing the study was to gain information on the mean free path of the primary dislocations, that is, the dislocations that carry the bulk of the imposed strain. The effect of second phase particles is also apparent in this study: whereas long slip markings were evident in the single phase alloys, second phase particles tended to remove these markings. These points are discussed in more detail below.

The surface markings are shown in Figs. 4.9-4.14 for the alloys in the sequence 99.99% Al, commercial purity Al, Al-1Mg, Al-2Mg, Al-0.8Mn and Al-Cu, respectively. On each page, the structure for the forward strain step is displayed on the left and that for the reverse strain step is shown on the right. The pairs of pictures can most easily be registered by comparing the positions of blemishes. The surfaces examined are normal to the radial direction and the torsion axis is parallel to the long (vertical) edge of each image. Taking each alloy in turn;
99.99% Al, Fig. 4.9: both after the forward step and the backward step, pronounced slip markings are evident. In one grain, only markings perpendicular to the torsion axis can be seen. In another grain, however, markings can be seen that run approximately parallel to the torsion axis. Since the markings are probably the traces of the active slip planes, they indicate that the mean free path of the primary mobile dislocations is much larger than the cell spacing. The latter dimension will be established in the next chapter but is generally of the order of 1 \( \mu \text{m} \). The reverse strain step does not noticeably affect the slip markings.

Commercial purity Al, Fig. 4.10: this material did not show such obvious slip markings as the pure aluminum. The sets of parallel lines spaced approximately 5 \( \mu \text{m} \) apart, crossing the images at an angle are presumed to be grain boundaries and stringers. The lack of slip markings is presumed to be a consequence of the second phase particles in this material. In the one grain where they are most evident after the forward strain step, the reverse step appears to reduce the height of the slip steps. This suggests that the reverse strain occurred by dislocation flow along the same planes as in the forward step. There are a few locations where lines of polishing pits, parallel to the grain boundaries, are evident; these are presumed to be due to stringers of the Al-Fe second phase particles.

Al-1\%Mg, Fig. 4.11: This material is identical to the commercial purity Al with the exception of the 1\% Mg in solution. The stringers of particles are more evident in these micrographs, perhaps as a result of variations in polishing conditions. More importantly, the slip markings that were evident in the pure Al are once again clearly present in this
alloy in some grains. The alignment of the markings is the same as before, being dominated by traces perpendicular to the torsion axis. As in the 99.9% Al, the reverse strain step has little effect on the slip markings.

Al-2Mg, Fig. 4.12: The results for this alloy are similar to those obtained in the 1%Mg alloy, despite the absence of stringers of second phase particles. The slip markings appear more marked after the reverse strain step though the effect is slight and may be due to variations in lighting conditions.

Al-0.8Mn, Fig. 4.13: The micrographs of this alloy show no signs of the slip markings that were a feature of the alloys described above. This is presumably a consequence of the homogenizing effect of the Al₆Mn precipitates present in the material. The spacing of the dot contrast in the micrographs is compatible with the approximately 1 micron spacing of precipitates found in TEM examination.

Al-4.5Cu, Fig. 4.14: This alloy was tested in the over-aged condition. Here again, the large second phase particles have the effect of homogenizing slip so that no slip markings are evident. The hardness indentations served as reference marks.

The results of this study indicate a) that in single phase materials, the slip markings are very much longer than the characteristic size of the dislocation structures, described in the next chapter; b) that the presence of a dispersed second phase disrupts the tendency of dislocations to move along the same planes and produce obvious slip steps on the surface; c) that there is essentially no reverse flow on exactly the same slip plane.
Fig. 4.9 a) 99.99% Al twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings. b) as above after a reverse strain of 9%.
Fig. 4.10 a) Commercial purity Al twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings.

b) as above after a reverse strain of 9%.
Fig. 4.11 a) Al-1Mg twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings.
b) as above after a reverse strain of 9%.
Fig. 4.12 a) Al-2Mg twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings.

b) as above after a reverse strain of 9%.
Fig. 4.13 a) Al-0.8Mn twisted to a shear strain of 3.38, electropolished and strained a further 9% to develop slip markings.
b) as above after a reverse strain of 9%. Note the lack of slip markings in either image.
Fig. 4.14 a) Al-4.6Cu twisted to a shear strain of 1.6, electropolished and strained a further 9% to develop slip markings. 
b) as above after a reverse strain of 9%. Note the lack of slip markings in either image.
IV.5 Crystallographic Textures

IV.5a Initial Textures

The initial textures of each alloy are shown in Fig. 4.15. The strength of the initial texture varied with material. The textures of the Al-Mn and Al-Cu alloys are not well defined because of the coarse grain size in those materials. The texture of the commercial purity aluminum shows a distinct fiber symmetry which is reasonable in light of the extrusion processing that was used to produce the material.
Fig. 4.15 Initial textures of the alloys:

a) 99.99% Al, (i) (111), (ii) (200)

b) commercial purity Al, (i) (111), (ii) (200)
Fig. 4.15 Initial textures of the alloys:

c) Al-0.8Mn, (i) (111), (ii) (200)
d) Al-1Mg, (i) (111), (ii) (200)
Fig. 4.15 Initial textures of the alloys:

e) Al-2Mg, (i) (111), (ii) (200)

f) Al-Cu, (i) (111)
IV.5b Experimental Technique

As a check on the statements made in the literature review about texture and the use of Taylor Factors, the texture of the commercial purity alloy was measured at von Mises equivalent strains of 1 and 2. An automated X-ray texture goniometer was used for this and a standard reflection method was used. That is, a metallographic mount of the strained material, in the polished condition, is mounted in the goniometer and intensities from a given reflection such as (111) or (200) are measured as a function of angle. The gauge section of the Lindholm tube is small and it was necessary to mount three pieces from the gauge section side by side, see Fig. 4.16. This mounting technique also requires each piece of the gauge section to be flattened before mounting which introduces a bending strain which, however, is small compared to the accumulated shear strain in the material.

IV.5c Experimental and Theoretical Torsion Textures

The results of texture determinations on the commercial purity aluminum are displayed in Fig. 4.17, for von Mises equivalent strains $\kappa v M = 1$, and Fig. 4.18, $\kappa v M = 2$. Alongside each experimental pole figure is a theoretical pole figure calculated with the Los Alamos Polycrystal Plasticity (LAPP) code. This code has been described in detail elsewhere in various publications, see for example Canova et al. [4,3]. The experimental textures appear to match the calculated pole figures reasonably well. The lack of symmetry in the highest intensity contours in the experimental pole figures is presumed to be due to the coarse grain size of the materials tested which means that a few strongly reflecting grains can make a large difference to the positions of the
intensity maxima. There may also be an effect from the initial texture of the material.
Fig. 4.16 Sample for texture measurement, showing pieces from the gauge section flattened and mounted side by side to obtain sufficient surface area.
Fig. 4.17 a) (111) pole figure for commercial purity Al twisted to a shear strain of 1.7.

b) as a) except (200) pole figure

c) (111) pole figure calculated with LAPP code at the same simulated strain.

d) (200) pole figure calculated with LAPP code.
Fig. 4.18 a) (111) pole figure for commercial purity Al twisted to a shear strain of 3.5.

b) as a) except (200) pole figure

c) (111) pole figure calculated with LAPP code at the same strain

d) (200) pole figure calculated with LAPP code.
IV.5d Torsion Textures at Zero Net Strain

Textures were also measured at zero net displacement for certain of the materials. The experimental textures at zero net displacement are shown for the 99.99% Al and the 2% Mg alloy in Fig. 4.19. It is clear that the texture, though not strong, is still a torsion texture. This confirms Backofen's result that a torsion texture is still present at zero net strain [4.4]. These results are less straightforward to account for than for the monotonically strained material. The reason is that the theoretical texture at zero net displacement is exactly that texture with which the calculation started, as shown by the sequence of pole figures in Fig. 4.20. The pole figures displayed in Fig. 4.20 were calculated from data very kindly provided by T. Lowe of Sandia National Labs. at Livermore, using the code developed by Asaro and Needleman [4.5]. The IAPP code yields precisely the same result that reversing a torsional strain to zero net strain restores the texture to the initial texture. The comparison of the two codes was made because the latter code only takes account of plastic deformation. The Asaro-Needleman code requires much more computing time but takes account of both elastic and plastic deformation.

The same specimens used for these texture determinations were examined optically to determine the grain structure. Figure 4.21 shows that the grain structure apparently returns to something very close to the initial, approximately equi-axed structure after a total strain, forward and back, of 4 (von Mises equivalent). The torsion with reverse torsion experiments were undertaken primarily to investigate the effect of a change in strain path on the strain hardening properties of the various alloys. Nevertheless, the by-product of the experiments is of
some interest since it appears to pose a new challenge to the theoretical calculations of texture.
Fig. 4.19 a) (111) pole figure for 99.99% Al after a forward shear strain of 3.5, then untwisted to zero net strain.

b) as a), (200) pole figure.

c) (111) pole figure for Al-2Mg at zero net strain, forward and reverse strains of 3.5.

d) as c), (200) pole figure.
Fig. 4.20 a) initial texture of simulated polycrystal with 300 randomly distributed orientations, (111) pole figure.

b) (111) pole figure for the simulated polycrystal after a forward shear strain of 3.

c) (111) pole figure for the simulated polycrystal after reversing the strain to a net shear of 0.05, calculated by T. Lowe using the Asaro-Needleman texture simulation code. This figure also shows the positions of the idealized components of a torsion texture.
Fig. 4.21 Grain structure of Al-2Mg at zero net strain after forward and reverse shear strains of 3.5, showing that the grain structure returns to an approximately equi-axed shape. Anodized surface viewed with crossed polars.
IV.6 Summary

The results presented in this chapter have shown the following:

a. the deformation in the Lindholm tube torsion specimen is uniform to very large strains.

b. slip line studies show a strong effect of second phase particles; viz., tending to eliminate the coarse slip line markings evident in single phase materials.

c. texture development in torsion follows the expected course.

d. Backofen's result has been confirmed that twisting and untwisting to zero net strain does not restore the texture to the initial texture but instead a torsion texture remains.

IV.7 References


Chapter V. Transmission Electron Microscopy Results

V.1 Introduction and Foil Preparation

This part of the work was undertaken primarily to characterize the heavily deformed materials generated by the stress-strain tests at the scale of the dislocation structure. A companion aim was to characterize the dislocation debris generated by plastic flow since accumulation of this debris is the basis for the model of Stage IV work hardening in single phase metals presented in chapter VI. A length scale is provided on high magnification micrographs that indicates the mean dislocation spacing calculated from the current flow stress and equation 3.1 (chapter II). The electron beam direction is indicated as B=[hkl]. In some micrographs the straining direction is indicated by the conventional pair of arrows that show tangential directions in a plane normal to the radial direction. All the foils were cut perpendicular to the radial direction unless otherwise indicated. The arrangement of foil shape and direction markers is illustrated in Fig. 5.1
Fig. 5.1  a) Diagram of foil shape and directions for foils cut normal to a radial direction (inside surface of tube).

b) Diagram of foil shape and directions for foils cut normal to the torsion axis.
The foils were prepared by the conventional methods. That is, thin slices were cut on a diamond saw to approximately 250 \( \mu m \) thick and reduced to 125 \( \mu m \) by grinding both sides on 600 SiC grit paper with water as a lubricant. Then the foils were thinned to electron transparency in a Tenupol twin-jet electropolisher using a solution of 1/3 \( HNO_3 \), balance methanol, at -30\(^\circ\)C. A nominal voltage of 20\( V \) was used, which gave a current density of approximately 2\( A/cm^2 \). With careful adjustment of the sensitivity of the photoelectric system, the hole was of the order of 100 \( \mu m \) in size and the usable area extended a few hundred microns into the foil. Not all attempts to manufacture foils gave such good results.

Most of the images presented here were obtained on a JEOL 200EX electron microscope operated at 200\( kV \) or on a Philips 400 operated at 120\( kV \). Both instruments have STEM capability which is convenient for obtaining instant working prints of a dislocation structure before performing surveys of orientation. A double tilt goniometer stage was used that was equipped to maintain the foil at a low temperature during examination. By using liquid nitrogen as the refrigerant, it was possible to keep the foils at less than -160\(^\circ\)C during examination which, it was hoped, would maintain the heavily dislocated structures in an unrecovered state. Even with this device, it was noted that converging the beam on a particular location would result in dislocation motion. Convergent beam techniques were necessary in order to obtain clear Kikuchi line patterns for orientation determination.

V.2 Stage III

In this section a comparison of the different alloys is presented where each alloy has been deformed into Stage III until the hardening
rate had dropped to the same level. The convention has been to compare materials at the same strain level. In many cases, however, materials are compared at the same (zero) hardening rate, either explicitly in this work when discussing saturation stresses at the end Stage III or Stage IV, or implicitly in the literature on creep. All the single phase alloys examined in this work developed cells and, at large enough strains, subgrains. The distinctions between cells, subgrains and grains tend to blur at large strains. The general trend is for misorientations across cell walls to increase with strain until at large strains, high angle boundaries are developed. The original grain boundaries cannot be found at very large strains.

There is evidence, see for example Thompson [5.1] or Raj and Pharr [5.2], that the cell size is proportional to the mean dislocation spacing (therefore as the reciprocal of the flow stress). It has been shown by Hasegawa et al. [5.3] that the decreasing hardening rate as Stage III progresses is reflected in an increasing "cleanliness" of the cell walls. For a given hardening rate, the sharpness of the cell structure is expected to be the same given only that the characteristic size of the cell structure is scaled by the reciprocal of the flow stress. This suggested a comparison of the various alloys at the same hardening rate which in this case was arbitrarily chosen to be 100 MPa in von Mises' equivalent units or 33 MPa in shear terms. The shear stress- shear strain curves are shown in Fig. 5.2A and the \( \theta-\sigma \) curves in Fig. 5.2B.
Fig. 5.2A Plot of Shear Stress versus Shear Strain for the Stage III experiment. All tests were terminated when the hardening rate was approximately 33 MPa in terms of shear stress and shear strain or 100 MPa in von Mises equivalent terms, well into Stage III.
Fig. 5.2B Plot of reduced Work Hardening Rate versus reduced Flow Stress for the data plotted in Fig. 5.2A
Figures 5.3 through 5.7 illustrate the various dislocation structures obtained in this experiment. The alloys with small amounts of solute, i.e. the 99.99% Al, commercial purity and 0.8% Mn alloys, show rectangular cells where the cell walls tend to be aligned with (111) planes. This result is that found by other investigators, e.g. Swann [5.4]. Even qualitatively, however, it is clear that the "cleanliness" of the cell structure is not the same in the alloys. The dislocation density within the cells rises with solute content. Higher magnification views of the 1% Mg alloy, Fig. 5.8 for example, show that there is a significant density of dislocation debris in the form of prismatic loops which are most easily seen near cell walls. Such loops have been observed in deformed f.c.c. metals by several investigators, see Swann's review [5.4].

The commercial purity Al and its 1% Mg alloy counterpart both show the stringers of Al-Fe particles that were evident in the optical and SEM microscopy, Figs. 5.2 and 5.4. The stringers are reasonably straight which indicates that the deformation was uniform.
Fig. 5.3 Structure in Stage III of 99.99% Al, after a shear strain of 0.27 at which the shear stress was 32 MPa. B=[114].
Fig. 5.4 Structure in Stage III of commercial purity Al, after a shear strain of 0.36, at which the shear stress was 41 MPa.

\( B = [114] \).
Fig. 5.5 Structure in Stage III of Al-0.8%Mn, after a shear strain of 0.4 at which the shear stress was 59 MPa. B=[110], g=(002).
Fig. 5.6 Structure in Stage III of commercial purity Al with 1\%Mg, after a shear strain of 0.4, at which the shear stress was 88 MPa.
Fig. 5.7 Structure in Stage III of Al-2%Mg, after a shear strain of 0.54 at which the shear stress was 105 MPa. $B=[110]$, $g=(220)$. 
Fig. 5.8 Structure in Stage III of Al-1%Mg, after a shear strain of 0.4 at which the shear stress was 88 MPa. View at high magnification of dislocation loops near cell walls. The expected mean dislocation spacing is 68 nm.
In the 99.99% Al, a survey of misorientations was performed as shown on Fig. 5.9, by recording convergent beam diffraction patterns and using the Kikuchi line positions to calculate orientations. The misorientations were calculated with the aid of a computer program kindly donated by E.Koken of McMaster University which is listed in Appendix B. The method is based on identifying the orientation of each location from the position of two Kikuchi-line pairs and then choosing the smallest misorientation out of the 24 possible rotation axes that connect a pair of orientations. The results of the survey, Table 5.1, indicate that at this low strain level, the cell to cell misorientations are small and are not cumulative. The misorientation measurements recorded here are not intended as statistically meaningful studies of the cell structure. This has been performed before, see Langford and Cohen [5.6]. The models for large strain hardening presented in this work do not depend in any particular way on the details of the cell structure. The information presented on cell size and misorientation is therefore intended as a test of the more detailed work performed by others.
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Fig. 5.9 View of cell structure in 99.99% Al in Stage III with Convergent Beam Diffraction Patterns (CBDP's) for a number of cells.
V.3 Stage IV, Room Temperature

The alloys investigated here did not show identical behavior at large strains but instead, the extent of Stage IV increased strongly with increasing solute content. For this reason it was not reasonable to make comparisons at the same hardening rate. Therefore a single strain was chosen that would be high enough to be well beyond Stage III, whether or no Stage IV behavior was obvious. An arbitrary choice of a von Mises equivalent strain of 2, i.e. a shear strain of 3.5, was made, Figs. 5.10-5.18.

The dislocation structures observed at this large strain vary more strongly than was the case in Stage III. The 99.99% Al has developed a clean cell structure with very low dislocation density within the cells. The Mn alloy has a very similar structure, Fig. 5.13, except that the cell size is smaller in keeping with the higher flow stress in this alloy. Comparison with the undeformed structure, Fig. 5.12, shows that the particles do not deform to any great extent. The 1%Mg alloy, however, shows a cell structure whose directionality is still defined by {111} slip plane traces, Fig. 5.14. Also the dislocation density within the cells is clearly high. The Al-2%Mg alloy shows a similar structure, Fig. 5.15.

The undeformed over-aged Al-Cu alloy shows nearly spherical particles of Al-2Cu, Fig. 5.16. This indicates that the over-aging heat treatment, 24 hours at 225°C, did not produce the expected structure but in fact must have been performed at a higher temperature than intended. At a large strain the particles appear to have been heavily deformed and in places broken up, Fig. 5.17. This is in strong contrast to the lack of deformation of the second phase particles in the Al-Mn alloy.
At higher magnifications, the Mg containing alloys showed clear evidence of loops, e.g. Fig. 5.18. Estimating the density of such debris is difficult, however, because of the high dislocation densities present in the materials. The structure of the Al-5Mg alloy is similar to that of the other Al-Mg alloys. Although the dislocation density is high and nearly uniform, there are significant cell to cell misorientations. Figure 5.19 illustrates the structure at a low magnification with two selected area diffraction (SAD) patterns inset. The SAD taken with the largest aperture shows partial rings whereas the SAD taken with the smallest aperture shows only slight asterism. When the smallest aperture is superimposed on the image, it covers an area that is approximately the size of one of the cells.
Fig. 5.10 Structure in Stage IV of 99.99% Al, after a shear strain of 3.46 at which the shear stress was 60 MPa. B=[110], g=(002).
Fig. 5.11 Structure in Stage IV of commercial purity Al, after a shear strain of 3.46 at which the shear stress was 74 MPa. 
B=[114], g=(131).
Fig. 5.12 Undeformed structure of Al-0.8%Mn showing particles of Al$_6$Mn. The diffraction pattern was taken from an area that included one of the (dark) particles. B=[111].
Fig. 5.13 Structure in Stage IV of Al-0.8%Mn after a shear strain of 3.46 at which the shear stress was 85 MPa.
Fig. 5.14  Structure in Stage IV of Al-1%Mg, after a shear strain of 3.46 at which the shear stress was 122 MPa. $\mathbf{B}=[110]$, $\mathbf{g}=(111)$. 
Fig. 5.15 Structure in Stage IV of Al-2%Mg, after a shear strain of 3.46 at which the shear stress was 152 MPa. B-[114].
Fig. 5.16 Undeformed structure of over-aged Al-4.6%Cu.
Fig. 5.17 Structure in Stage IV of overaged Al-4.6%Cu, after a shear strain of 3.46 at which the shear stress was 200 MPa. 

B=[552], g=(220).
Fig. 5.18 Dislocation loops at a shear strain of 3.46 in Al-1%Mg at which the shear stress was 122 MPa. This corresponds to a mean dislocation spacing of approximately 50 nm. B=[110], g=(111).
Fig. 5.19 Al-5Mg at a shear strain of 3.46. The inset SAD pattern that shows heavy asterism was taken with the largest aperture. The other SAD pattern was taken with the smallest aperture and shows much less asterism. B-[110].
V.4 Very Large Strains, Room Temperature

Certain of the materials were examined at even larger strains. The 99.99% Al at a shear strain of 6.9 showed significant changes from the previous strain. Figure 5.20 shows that the cell size is smaller as expected from the small increase in flow stress of ~5MPa. The appearance of the micrograph is also qualitatively different in that the contrast conditions within each cell vary considerably from one cell to its neighbor. Many of the boundaries exhibit strong fringe patterns which suggest misorientations larger than ten degrees. These indications will be developed in a more quantitative way below but they suggest that the cells observed at small strains have developed into subgrains at large strains. A further indication is that even the smallest aperture for Selected Area Diffraction (SAD) produces diffraction patterns with pronounced asterism. A general characteristic of heavily deformed metals is that Convergent Beam Diffraction Patterns (CBDP) are required to determine accurately the orientation of a particular cell or subgrain. A micrograph of a foil whose plane is normal to the torsion axis (approximately equivalent to a foil parallel to the rolling plane for that strain path) showed that the structure tended to be elongated in a direction parallel to the radial direction, Fig. 5.21. The cell size is of the order of 0.5 μm.

The commercial purity aluminum at a strain of 10.6 shows a very similar structure to that of the previous example, Fig. 5.22. For this sample a number of CBDP's were taken across a series of subgrains with locations shown in Fig. 5.23. The results are tabulated below in Table 5.2 which gives misorientations not only for adjacent subgrains but also between each pair of locations. This permits some feeling for cumulative
misorientations to be obtained. Misorientations are only given in the upper triangle of the matrix because of the obvious symmetry (the misorientation between 1 and 2 is the same misorientation as between 2 and 1). The cell size is of the order of 0.3 μm.

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Fig. 5.20  Sub-grain structure in 99.99% Al at a shear strain of 7.2.

B=[123].
Fig. 5.21 Sub-grain structure in 99.99% Al at a shear strain of 7.2 for a foil cut normal to the torsion axis. B=[122].
Fig. 5.22 Sub-grain structure in commercial purity Al at a shear strain of 10.6 and a shear stress of 92 MPa. Note the similarity of the structure to previous Figure.
Fig. 5.23 Locations and CBDP's in commercial purity Al at a shear strain of 10.6.
The Al-1%Mg alloy did not reach such large strains as the commercial purity aluminum, fracture intervening at about a shear strain of 8.5. Figure 5.24 illustrates the dislocation structure for this material at a shear strain of 8.4. The development of subgrains with large misorientations is similar to that shown for the unalloyed materials. The dislocation density within the subgrains appears to be higher and dislocation debris is more evident. The stringers of Al-Fe second phase particles that were so evident at small strains are not visible though individual particles can still be identified. A survey of misorientations is presented in Table 5.2 with the locations shown in Fig. 5.25.

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Fig. 5.24  Sub-grain structure in Al-1%Mg alloy at a shear strain of 8.4 and a shear stress of 155 MPa.
Fig. 5.25 Locations and CBDP's in Al-1%Mg alloy at a shear strain of 8.4 and a shear stress of 155 MPa.
V.5 Complete Strain Reversal

The commercial purity Al was examined after a torsion-reverse torsion experiment where the forward and backward shear strains were 3.46, giving zero net displacement. The TEM observations are of interest because of the apparent conflict between the optical observation that the grain shape is restored and the X-ray observation that the texture does not return to the initial texture. The structure viewed at low magnification is similar to those for the forward torsion case at intermediate strains, Fig. 5.26. The directionality of the cell structure is approximately parallel to the shear direction. The cell size is of the order 0.5 μm. Examination at higher magnifications shows that the cell interiors have some dislocation content, including debris in the form of loops, Fig. 5.27. A brief survey was made of orientations, Fig. 5.28, and the data is presented in Table 5.4 with the caveat that this is intended to give an impression of the range of orientation present in the structure. The results suggest that large misorientations exist on the sub-grain level despite the apparent return of the grain structure to an approximately equiaxed structure.
Table 5.4

Misorientations (degrees) in commercial purity Al at γ=0

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Fig. 5.26  Dislocation structure at low magnification in commercial purity Al at zero net displacement and a shear stress of 82 MPa. B=\langle 110 \rangle.
Fig. 5.27 Dislocation structure in commercial purity Al in one cell, showing presence of debris, a) bright field, b) dark field weak beam. $B=[110]$, $g=(002)$. 
Fig. 5.28 Locations and CBDP's in commercial purity Al at zero net displacement.
V.6 Temperature of Deformation

Up to this point all the structures considered have been of materials deformed at room temperature, about 293K. As the deformation temperature changes, the rate of dynamic recovery changes markedly. As Kocks [5.7] and others have shown, for pure f.c.c. metals at least, the saturation stress extrapolated from Stage III behavior varies exponentially with temperature. The effects on Stage IV have been demonstrated in the chapter describing the results of the mechanical tests. Lowering the temperature of deformation strongly reduces dynamic recovery and enhances work hardening in Stage III. The commercial purity Al at 77K, Fig. 5.29, shows a markedly refined subgrain structure as compared to room temperature, in keeping with the much higher flow stress. The dislocation density within each cell is also comparable with that observed in the Mg alloys at room temperature, i.e. the cell interiors are not as "clean". This result is in keeping with the more pronounced Stage IV observed at 77K than at room temperature. The small cell size and high dislocation density made a survey of misorientations impracticable. The variation of contrast with tilting of the specimen stage, together with the pronounced asterism evident in SADP's, suggested that the misorientations between cells was as large as shown for other heavily strained materials in this work. The specimen from which Fig. 5.29 was obtained, was maintained at 77K until it was sectioned for foil preparation. The foil itself was at room temperature for approximately three hours before being examined in the microscope. Although there is a small amount of rapid recovery on warming to room temperature, as shown by small decreases in flow stress in the mechanical tests, it was assumed that the effect on the observable
dislocation structure was small. Figure 5.30 shows a comparable image obtained from a foil of the same specimen of commercial purity aluminum deformed at 77K, where the foil had been at room temperature for at least two weeks before being examined in the TEM. Evidence for dislocation debris was found at this deformation temperature as shown in Fig. 5.31. Three views of the same cell are shown, showing dislocation loops. The three views were obtained with various zone axes and diffraction vectors and show the expected variation of visibility of the dislocation loops. Two of the views were obtained with the same zone axis but opposite diffraction vectors (g=±111); the apparent size of a few of the loops changes in the expected manner, Edington [5.5].

Higher deformation temperatures in commercial purity aluminum have the expected effect of coarsening the cell structure as the flow stress at the end of Stage III drops with increasing temperature. Figures 5.32 and 5.33 illustrate the structures for 373K and 473K respectively. Even at the highest temperature of 473K, evidence of dislocation debris was easily found as shown in Fig. 5.34. A survey of misorientations, Table 5.5 and Fig. 5.35, shows that equally large misorientations develop at this deformation temperature as at lower temperatures.
| Misorientations (degrees) in commercial purity Al deformed at 473K |
|-------------------|---|---|---|---|---|---|---|---|---|---|
| 1                 | 42.4 | 22.9 | 40.7 | 31.5 | 37.8 | 26.8 | 46.1 | 34.3 | 33.4 |   |
| 2                 | 56.5 | 3.9  | 42.6 | 34.3 | 54.5 | 27.9 | 51.3 | 52.1 |   |   |
| 3                 | 60.1 | 22.9 | 32.2 | 10.7 | 48.7 | 20.4 | 19.5 |   |   |   |
| 4                 | 39.2 | 30.9 | 51.2 | 30.5 | 53.4 | 54.0 |   |   |   |   |
| 5                 | 9.5  | 13.1 | 49.1 | 30.0 | 30.4 |   |   |   |   |   |
| 6                 | 22.6 | 46.3 | 38.4 | 39.1 |   |   |   |   |   |   |
| 7                 | 55.1 | 19.8 | 19.8 |   |   |   |   |   |   |   |
| 8                 | 53.5 | 53.9 |   |   |   |   |   |   |   |   |
| 9                 | 1.7  |   |   |   |   |   |   |   |   |   |
Fig. 5.29 Structure of commercial purity Al deformed at 77K to a shear strain of 5.75 and a shear stress of 197 MPa. B=[110],
g=(111).
Fig. 5.30 Structure of commercial purity Al deformed at 77K to a shear strain of 5.75 and a shear stress of 197 MPa but in a foil that had been annealed at room temperature for more than two weeks before being examined in the TEM.
Fig. 5.31 Three views of dislocation loops in the same cell in Al-0.17Fe-0.07Si deformed at 77K to a shear strain of 5.75 and a shear stress of 197 MPa. Each micrograph was recorded under different diffraction conditions as shown by the diffraction patterns, (a) B=[011] g=(220), (b) B=[123] g=(111) and (c) B=[123] g=(111).
Fig. 5.32  Structure of commercial purity Al deformed at 373K to a shear strain of 8.7 and a shear stress of 69.5 MPa. B=[123], g=(111).
Fig. 5.33  Structure of commercial purity Al deformed at 473K to a shear strain of 6.2 and a shear stress of 42 MPa. B=[112], g=(111).
Fig. 5.34 Dislocation debris (loops) in commercial purity Al deformed to a shear strain of 6.2 at 473K. B=[112], g=(111).
Fig. 5.35 CBDP's in various locations in commercial purity Al deformed at 473K.
V.7 Strain Rate Effects

Perhaps the most dramatic differences in large strain behavior were exhibited by the 1%Mg alloy at high temperature, 473K, where the lowest strain rate appeared to saturate but the highest strain rate continued to harden. Figures 5.36 and 5.37 compare the lowest and highest strain rates for this situation. The result of the comparison is that the higher strain rate clearly has a smaller sub-grain size as one might expect from its higher flow stress. Otherwise the two structures appear to be similar in terms of sub-grain shape and dislocation density within sub-grains.
Fig. 5.36 Structure of Al-1%Mg deformed at 473K at a shear strain rate of $7.10^{-3}$ s$^{-1}$ to a shear strain of 5.5 and a shear stress of 82 MPa. $\mathbf{B}=[110]$, $\mathbf{g}=(111)$. 
Fig. 5.37  Structure of Al-1%Mg deformed at 473K at a shear strain rate of $7 \times 10^{-1} \text{s}^{-1}$ to a shear strain of 6.2 and a shear stress of 108 MPa. B=[110], g=(002).
V.8 Summary

The results of the TEM examination of heavily strained aluminum alloys can be summarized as follows:

a) A subgrain structure is developed with approximately equiaxed shapes and large misorientations across the boundaries. The subgrains tend to be slightly elongated parallel to the macroscopic elongation direction. The term "subgrain" is used in this thesis in preference to "cell" for structural units where misorientations in excess of 20° were measured.

b) The presence of an obvious Stage IV hardening behavior correlates with a high dislocation density within subgrains; if, on the other hand, Stage IV is weak or absent, the subgrain structure tends to be "clean", i.e. the subgrain interiors exhibit low dislocation densities, as at high temperatures in the commercial purity aluminum.

c) Dislocation debris is observed as small loops, possibly prismatic, at all levels of deformation. As Stage IV becomes more obvious with decreasing temperature and increasing alloy content, so the observed density of loops increases. Where loops are visible inside subgrains, the number density of loops can be estimated approximately as being of order $10^{21} \text{ m}^{-3}$

d) Loops were observable within subgrains at large strains with sizes on the order of 10 nm. They may also be present at subgrain boundaries but high dislocation densities rendered observation difficult.

e) In Al-1Mg microbands or shear bands were not evident at any of the strains examined. Commercial purity aluminum deformed at 77K also did not exhibit strain inhomogeneities of this type. The conclusion is
that Stage IV is not associated in any significant way with
inhomogeneous deformation.

V.9 References

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Chapter VI. Models for Stage IV Work Hardening

VI.1 Introduction

This chapter describes three models for Stage IV work hardening in metals. The placement of this chapter in the thesis reflects the fact that the models were developed after the majority of the experimental data had been obtained. As so often happens in metallurgy, the need for empirical information means that the essential facts are often known before a theory is produced. An aim of this chapter is to attempt to provide models for Stage IV that are adequate constitutive relations while being based on microscopic mechanisms that are in keeping with the known experimental facts. The discussion, chapter VII, will then compare the experimental results presented in previous chapters in the light of these models. The first model provides an alternative to the standard Hall-Petch analysis for two-phase materials. This first model applies to two-phase materials in which the second-phase entities are hard enough to enforce geometrical storage of dislocation line length. Two models are discussed for single-phase materials. The first takes Stage I behavior as its starting point since the hardening rates in Stage I and Stage IV are so similar. The model considers debris accumulation to occur by glide and was found to be less satisfactory than the next model to be described.

The second model for single-phase materials examines the role of debris in affecting the saturation stress at what would be the end of Stage III if Stage IV did not intervene. In this model the accumulation of debris is considered to occur as a result of dynamic recovery events.
This model is proposed as the best model for Stage IV in single-phase materials.

VI.2 Single-Phase versus Two-Phase Materials

The behavior of two-phase materials is quantitatively different from that of single-phase materials if the second-phase particles have a high aspect ratio. The difference is that, whereas single-phase materials exhibit a hardening rate in Stage IV that is either constant or slowly falling, two-phase materials can show a hardening rate that increases during Stage IV. The experimental evidence for this will be discussed further in the Discussion. The following section outlines an alternative for the existing model of high strain behavior in such materials as pearlitic steel.

VI.2a Model for Two-Phase Materials

The large strain work hardening behavior of materials in tension that contain high aspect ratio second phases has been modeled previously on the basis of the Hall-Petch relation, for example Embury and Fisher [6.1]. The basis of their model is that the effective "grain size" at large strains is set by the spacing of the second-phase particles. This spacing varies as the exponential of half the tensile strain leading to an additive contribution to the flow stress that varies as the exponential of one quarter of the strain. Therefore the hardening rate is proportional to one quarter of the flow stress.

It is possible, however, to provide an alternative model, with the same results, that is based on the mean free path of the mobile dislocations being limited by the mean spacing between second-phase entities. For axisymmetric tensile deformation of, say, a fiber
reinforced composite, it is expected that the mean spacing between the fibers, $d$, will decrease as

$$d = d_0 \exp \left( -\frac{\varepsilon}{2} \right),$$

(6.1)

where $d_0$ is the initial spacing. Other deformation modes such as plane strain compression and torsion lead to a contraction the goes as $\exp(-\varepsilon)$ rather than $\exp(-\varepsilon/2)$. This consideration would appear to lead to a deformation path dependence for the model developed below. Such a dependence of hardening rate in Stage IV in two-phase materials is not apparent in the literature. Therefore the example of tension will be worked through as an example.

If the mean free path of dislocations is limited by this spacing then a geometrically enforced storage of dislocation line length takes place such that

$$\frac{d\rho}{d\varepsilon} = k'' d^{-1}$$

(6.2)

where $k''$ contains the details of the geometry of the storage process.

The use of this equation is justified by analogy with the description of dislocation storage in Stage II; see the discussion in section II.2b.3.

In this case the obstacles causing dislocation storage are the stored dislocations. Then with the standard relation between the flow stress and the dislocation density,

$$2\sigma \frac{d\sigma}{d\varepsilon} = (M \alpha G b)^2 k'' d_0^{-1} \exp \left( \frac{\varepsilon}{2} \right)$$

(6.3)

This can be re-arranged and integrated to give

$$\sigma = \sqrt{(M \alpha G b)^2 k'' d_0^{-1} 2 \exp \left( \frac{\varepsilon}{2} \right) + A}$$

(6.4)

where $A$ is a constant of integration. Setting $B = (M \alpha G b)^2 k'' d_0^{-1}$ and differentiating,

$$\frac{d\sigma}{d\varepsilon} = B \exp \left( \frac{\varepsilon}{2} \right) / \sqrt{(A + B \exp \left( \frac{\varepsilon}{2} \right))}$$

(6.5)

In the limit of large strains, $B \exp(\varepsilon/2) >> A$ and
\[ \theta = \sigma/4 \quad (6.6) \]

If the constant \( A \) were zero, Eq. 6.6 would hold at all strains instead of being a high strain limit. This result is the same result as obtained with the Hall-Petch model by Embury and Fisher [6.1] and suggests that Eq. 6.6 describes both the transition from Stage III to Stage IV and the work hardening rate in Stage IV itself.

Note also that if Eq. 6.6 is expressed in terms of stresses resolved onto a single slip plane (by dividing the LHS by \( M^2 \) and the RHS by \( M, M=3.2 \)), the constant changes from 1/4 to 0.08, approximately. That is, Stage IV can be described by \( \theta=0.08\tau \) which is the criterion proposed for Stage IV by Embury and Mecking (see the discussion in the literature review, Chapter II).

VI.3 Debris Accumulation by Glide model (DAG)

The literature review described the accumulation of dipoles that controls the small but finite work hardening rate observed in Stage I. It is possible that this process occurs even during multiple slip in polycrystals: the microstructures would not show the debris in fashion of single slip because the dipoles would be cut by primary and secondary slip on intersecting planes. The model to be described in this section therefore asks the question of what would happen to the strain hardening properties of a material in which dislocation debris accumulated at a constant rate. The underlying assumption is that at small strains, the monopolar dislocation forest develops at such a high rate that any contribution from dipoles or loops is masked. At large strains, however, the development of the monopolar forest saturates at which point only the accumulation of debris makes any contribution to the observable hardening rate. The model is that it makes no detailed assumptions about
the microstructure, e.g. a cellular dislocation structure is not assumed.

If debris accumulates steadily as straining proceeds, there may be a second component to the flow stress that is small but constantly increasing. In easy glide, Stage I, the type of debris that accumulates is bundles of dipoles, termed multipoles. At large strains, however, such dipole arrays are not observed and instead the debris appears to take the form of prismatic loops. Therefore in this model and the "Debris Accumulation by Dynamic Recovery" model that follows, the discussion will be based on the properties of loops. Similar considerations would apply to dipole debris.

VI.3a Stress Superposition

The last section of the review chapter, II.4, found that the strength of obstacles such as prismatic loops was comparable with that of the dislocation forest. However low the density of dislocation debris and weak their contribution to the flow stress, it is necessary to add the two (effective) dislocation densities, Kocks et al. [page 226 of ref. 6.2],

\[ \tau = \sqrt{\tau_1^2 + \tau_2^2}. \] (6.7)

\( \tau_1 \) is assumed to be described by the Kocks model for athermal storage of dislocation line length that generates a constant hardening rate until dynamic recovery sets in and reduces the observed hardening rate. At a large enough flow stress the rate of dynamic recovery equals the rate of dislocation storage and \( \tau_1 \) saturates. \( \tau_2 \) is the stress contribution from the dislocation debris such as dipoles. \( \tau_1 \) is assumed to be the major contribution to the flow stress, due to the monopolar dislocation forest. \( \tau_2 \) is the minor one due to the accumulation of debris.
For prismatic loops, Kroupa [6.3] has shown that a reasonable relation between stress contribution and mean loop size, \( <r> \), and loop number density, \( n \), is

\[
\tau_2 = \alpha' G b \sqrt{<r> n} \tag{6.8}
\]

Kroupa has estimated the interaction coefficient, \( \alpha' \), as 0.18. A commonly observed loop size in work on quenched and aged aluminum is about 10 nm, Dollar and Thompson [6.4]. The experimental evidence presented above suggested that Stage IV is essentially athermal when the deformation temperature is a small fraction of the melting point (say \( T < 0.2T_m \)), suggesting an accumulation rate that scales with the current mean dislocation spacing, as in Stage II only with a much smaller proportionality constant. This argument is not new as it is precisely that used by Prinz and Argon [6.5] in the derivation of their model of large-strain work hardening. The physical picture is that of mobile dislocations passing near enough to each other that they trap each other to form a dipole. The model presented in this section differs from that of Prinz and Argon in that no assumptions are made about the location of the debris. Prinz and Argon [6.5], assumed that the debris accumulated in cell walls and therefore included separate strength contributions from the cell walls and cell interiors.

VI.3b Hardening Rates

The increment of dipole density per strain increment is proportional to the mobile dislocation density and to the slip plane spacing. The latter is, however, inversely proportional to the current flow stress and hence to the root of the current dislocation density. So starting with Eq. 18 of Ref. [6.5], if \( \rho_{\text{debris}} \) is the line density of debris,
\[
\frac{d\rho_{\text{debris}}}{d\gamma} = k' \sqrt{\rho} \tag{6.9}
\]

where \(\rho\) is the dislocation density of primary monopolar dislocations. At the end of Stage III, the density of monopolar dislocations is presumed to saturate at a value corresponding to \(r_{s1}\), so the appropriate value of \(\rho\) in Eq. 6.9 is given by
\[
\rho_{IV} = (r_{s1})^2 / (aGb)^2 \tag{6.10}
\]

This leads to a hardening rate for the second stress contribution of
\[
\frac{d\tau_2}{d\gamma} = k' \sqrt{\rho_{IV}} \alpha' G b / (2r_2). \tag{6.11}
\]

This expression is awkward to deal with because the quantity \(r_2\) depends on the prior history during Stage III. This is because the debris that constitutes \(\rho_{\text{debris}}\) accumulates throughout the straining history. It does, however, indicate that the hardening rate is expected to decrease slowly in Stage IV with increasing stress.

VI.3c Numerical Solution of Debris Accumulation by Glide (DAG) model

Obtaining an analytical expression for the total work hardening rate with Eqs. 6.7-6.8 (assuming that the monopolar dislocation density is given by Eq. 2c.4 from Chapter II) is difficult to do. Therefore a numerical solution was used to characterize the transition from Stage III to Stage IV behavior. Selecting reasonable values for the various parameters shows that \(k'\) must be of the order \(5.10^{-5}\) in order to obtain work hardening rates in Stage IV of the order of \(2.10^{-4}\) as found experimentally. By reasonable values is meant that \(\alpha'\) is taken to be equal to \(\alpha\), i.e. 0.5, implying that the interactions between mobile dislocations and debris such as loops is the same as for the dislocation forest.

This value of \(k' (5.10^{-5})\) is small when compared to the value of approximately 0.1 used by Prinz and Argon to fit work hardening data for
an aluminum alloy at room temperature. Note, however, that the PA model was intended to account for the curvature of the plot of hardening rate versus stress of unalloyed aluminum at room temperature. Therefore the PA model was dealing only with Stage III whereas the model discussed here focuses on much lower hardening rates. A further comment on Stage III that should be made is that Kocks’s quantitative model for Stage III, section II.2c.3, contains a linear stress dependence for the rate of dynamic recovery. There is nothing sacred about the linear stress dependence and the curvature characteristically observed in $\theta$--$\sigma$ plots at high homologous temperatures can be accommodated by simply varying this dependence (Eq.II.2c.2).

VI.3d Results of Numerical Model

Figure 6.1 shows the results of a computer code listed in Appendix C, for five different saturation stresses for the monopole dislocation density. The values of the saturation stress (of $\tau^*_1$, the monopole dislocation density) were chosen to be in the same range as those found experimentally, i.e. $\tau^*_1/G = 0.001 - 0.010$. The other parameters were taken as $k' = 0.0005$, $<r> = 10$ nm and $\alpha' = 0.5$. The plots of work hardening rate versus stress show that the work hardening rate at the transition from Stage III to Stage IV is approximately constant, as observed experimentally. As expected from Eq. 6.11, the work hardening rate in Stage IV decreases slowly as the stress increases. The transition from Stage III to Stage IV, however, is smoother than that observed experimentally (c.f. Fig. 3.13B in Ch. III), particularly for higher flow stresses.
Fig. 6.1 Plot of Hardening Rate versus Stress for the Debris Accumulation by Glide model. All quantities have been normalized by the shear modulus and are in terms of resolved shear stresses on a slip plane.
VI.3e Loop Densities

Looking in detail at the densities of prismatic loops that the model produces, Table 6.1 lists the shear strain, reduced stress, dislocation debris line density and loop number density, assuming a mean loop size of 10 nm: only the results for the lowest and highest values of \( \tau_{s1} \) plotted in Fig. 6.1 (\( \tau_{s1} = 25 \) MPa, part A, and \( \tau_{s1} = 125 \) MPa, part B) are given.
Table 6.1A

Results of Numerical Solution for the DAG model

Part A: Low Stresses in Stage III

<table>
<thead>
<tr>
<th>$\gamma$</th>
<th>$10^3 r/G$</th>
<th>$\rho_{\text{debris}}$</th>
<th>loop # density</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.50</td>
<td>1.0</td>
<td>0.875E+13</td>
<td>0.875E+21</td>
</tr>
<tr>
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<td>1.1</td>
<td>0.204E+14</td>
<td>0.204E+22</td>
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<td>1.2</td>
<td>0.322E+14</td>
<td>0.322E+22</td>
</tr>
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<td>1.3</td>
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</tr>
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<td>1.4</td>
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<td>0.559E+22</td>
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<td>0.795E+22</td>
</tr>
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<td>0.913E+22</td>
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<td>2.8</td>
<td>0.345E+15</td>
<td>0.345E+23</td>
</tr>
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</table>

Part A; $r_{s1} = 25$ MPa = 0.00094G, equivalent to a monopolar dislocation density of $2.10^{14}$ m$^{-2}$. Calculation of loop number density based on a mean loop size of 10 nm.
Table 6.1B

Part B: High Stresses in Stage III

<table>
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<tr>
<th>$\gamma$</th>
<th>$10^3 \sigma/G$</th>
<th>$\rho_{\text{debris}}$</th>
<th>loop # density</th>
</tr>
</thead>
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</tr>
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<td>0.198E+23</td>
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<td>0.664E+23</td>
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<td>0.164E+24</td>
</tr>
</tbody>
</table>

part B: $\tau_{\text{sl}} = 125$ MPa $= 0.0047G$, equivalent to a monopolar dislocation density of $1.1 \times 10^{15}$ m$^{-2}$. Calculation of loop number density based on a mean loop size of 10 nm.

The tabulated results indicate that even for the lowest stress levels at the end of Stage III, large densities of debris are required in order to achieve a Stage IV behavior. In fact, the debris line density exceeds that of the monopolar dislocation density at shear strains less than 3. Dollar and Thompson [6.4] observed loop densities as high as $10^{22}$ m$^{-3}$ in quenched aluminum so the results of the model are approximately an order of magnitude higher at large strains.
VI.3f Stored Energy Considerations

If energy is stored in a deforming metal only as dislocation line length that contributes to the flow stress, then Eq. 2b.1 from chapter II can be rewritten as

\[
\text{Energy} = c \rho - c r^2 / (\alpha G b)^2
- c' \sigma^2
\]  

(6.13)

Ronngel and Schwink [6.6] found, however, that the stored energy increased as the cube of the flow stress at high strains, suggesting the storage of defects that do not contribute to the flow stress as strongly as monopolar dislocations. One aspect of the DAG model for Stage IV is that the debris contributes to the flow stress as a simple addition of the total dislocation density. Therefore the stored energy should scale as the square of the stress and the accumulation of debris cannot be used to explain the results of Ronngel and Schwink [6.6].

Incidentally, the interaction energy of dislocations decreases slowly (logarithmically) with the mean spacing between dislocations. This would tend to make the stored energy rise with stress raised to an exponent slightly less than two, not the higher than two as found experimentally.

VI.4. Debris Accumulation from Dynamic Recovery model (DADR)

This next section develops a model for Stage IV based on the production of debris inside the tangles that are essential to the work hardening observed in Stage II. The reason for focusing on these tangles is that, in spite of the evidence for debris in cell interiors, it is in the tangles that the saturation stress at the end of Stage III is presumed to be determined. The basis for the model is that debris production in the tangles increases the saturation stress: this upward drift in saturation stress generates the small Stage IV work hardening.
The term "saturation stress" is taken to mean not a fixed parameter towards which the flow stress tends asymptotically but stress level that a material would reach if only Stage III behavior occurred.

VI.4a Description of Recovery Events

Dynamic recovery events inside the tangles produced in Stage II may not always lead to perfect elimination of the dislocation segments involved in the recovery event, as suggested by Kocks [6.7]. As an example, consider the fate of two edge segments from the same slip system in a tangle that have arrived from opposite sides of the tangle and therefore have opposite sign, Fig. 6.2. A recovery event occurs, for example, when a mobile dislocation sweeps a nearby plane and assists these segments to move through the tangle until they meet and mutually annihilate. The difficulty with this simple picture is what happens when the segments are not on exactly the same slip plane. For opposing screw segments, the answer is simple, at least in a high stacking fault energy material, and that is that the segments cross slip out of their original planes to get to the same slip plane where they can mutually annihilate. For edge segments, however, this is not possible and if the edge segment slip planes are less than h apart,

$$ h = \frac{G b}{8 \pi (1-\nu) \tau}, \quad (6.14) $$

they will align themselves into a dipole. Although dislocation line length is apparently preserved in this recovery event, the resulting edge dipole has less effect on subsequent slip on nearby slip planes than would the original monopoles. Therefore recovery has occurred.
Fig. 6.2 Diagram of dipole formation by glide recovery event within a tangle, (a) before and (b) after the recovery event.
VI.4b. Fraction of Recovery Events Expected to Form Dipoles

It is important to the later discussion to estimate, however crudely, the fraction of such recovery events that might be expected to deposit a dipole inside the tangle. To begin with, only edge segments can form dipoles so that introduces a factor of one half. Then, at whatever the current flow stress in the tangle happens to be, the mean spacing of dislocations in the generally three dimensional network will be 1,

\[ 1 = (\rho)^{-0.5} - \alpha \frac{G b}{\tau}. \]  \hspace{1cm} (6.15)

The ratio of h to l is \((8 \pi (1-\nu) \alpha)^{-1}\) or about 0.13. Therefore if a single edge segment is moving through a tangle, its probability of being near enough to a suitable dipole pair segment is 0.13. Taking the factor of 0.5 into account, only about 1 in 15 of the type of recovery events described would be expected to generate dipoles.

VI.4c Conversion of Dipoles to Prismatic Loops

A dipole can break up into a string of prismatic loops without loss of line length of dislocation. This process requires pipe diffusion which can operate at low homologous temperatures. The clearest experimental evidence for this was found by Price [6.8] in TEM examination of Cd and Zn foils. A diagram of the process is shown in Fig. 6.3. Evidence for the presence of prismatic loops was found in TEM examination of the aluminum alloys investigated in this work. It is clear therefore that both dipoles and loops can contribute to debris density in a worked metal. This mechanism of loop production suggests that the size and spacing of loops produced would depend on the dipole spacing from which they originated. The maximum dipole spacing is set by the current flow stress, Eq. 6.12, which decreases with increasing flow
stress. This suggests that the loop size observed should decrease as the flow stress increases. The 10 nm loop size used below corresponds to a flow stress that is only $1.2 \times 10^{-3} \text{G}$ whereas the flow stresses were as high as $5 \times 10^{-3} \text{G}$ in some of the aluminum alloys tested, Chapter III. The dipole generation mechanism described above implied, however, that dipoles are produced inside the tangles where the local flow stress is higher than the mean. Not enough data on size distributions of loops were available in the TEM examination, Chapter V, to determine mean loop size as a function of stress. A single loop size is used to illustrate the results of the models as a convenience rather than the more physically accurate but less tractable detail discussed here.
Fig. 6.3 Diagram of the break-up of dipoles into loops.
VI.4d. Production Rate of Debris

If some fixed fraction of the dislocation line length lost in dynamic recovery is not in fact lost but converted to debris density, an equation can be written for the production rate of debris.

Quantitatively, the work hardening rate in Stage III can be written (in terms of a resolved quantity) as, \[6.9\],

\[\theta = \theta_0 \left(1 - \frac{\tau}{\tau_s}\right)\] \hspace{1cm} (6.16)

Given that the flow stress, \(\tau\), is related to the dislocation density, \(\rho\), by the well established relation [6.9],

\[\tau = \alpha \, G \, b \sqrt{\rho}\] \hspace{1cm} (6.17)

Then the rate of change of dislocation density with strain, \(d\rho/d\gamma\), is given by Eq. 6.16 and the differentiated form of 6.17

\[
\frac{d\rho}{d\gamma} = \frac{d\rho}{d\tau} \times \frac{d\tau}{d\gamma}
\]

\[
= 2 \, \theta \, \tau / (\alpha \, G \, b)^2
\] \hspace{1cm} (6.18)

If the rate of accumulation of debris, \(d(\rho_{\text{debris}})/d\gamma\), is some fraction, \(f\), of the dynamic recovery rate of dislocation density loss, then

\[
\frac{d\rho_{\text{debris}}}{d\gamma} = f \, \theta_0 \, \tau / \tau_s \times \frac{d\tau}{d\gamma}
\] \hspace{1cm} (6.19)

Near the saturation of Stage III, the dynamic recovery rate will be approximately equal to the storage rate, \(\theta_0\), and therefore constant. Therefore the debris accumulation rate can be written as

\[
\frac{d\rho_{\text{debris}}}{d\gamma} = f \, \theta_0 \times \frac{d\tau}{d\gamma}
\]

\[
= f \, \theta_0 \, \tau_s / (\alpha \, G \, b)^2
\] \hspace{1cm} (6.20)

VI.4e. Rate of Increase of the Saturation Stress

At this point we suppose that the saturation flow stress in the tangles is due to the two contributions of ordinary dislocation segments and debris. For the purpose of this model, the tangles are supposed to
be composed of a fixed dislocation structure to which debris is added so the flow stress in the tangles can be written

\[ \tau_s = \alpha G b \sqrt{(\rho_{\text{disl}} + \rho_{\text{debris}})} \]  

(6.21)

which can be differentiated in the same way as Eq. 6.17 to give

\[ \frac{d\tau_s}{d\rho_{\text{debris}}} = 0.5 \alpha G b (\rho_{\text{disl}} + \rho_{\text{debris}})^{-0.5} \]  

(6.22)

Implicit in this treatment is the assumption that the debris has similar strength to the rest of the tangle structure, permitting the addition of line length densities, Kocks et al. [6.2]. Note that this is a different assumption about the superposition of obstacles to dislocation glide than was made in the Continuous Debris Accumulation Model. To obtain the hardening rate in the tangles, combine Eqs. 6.22 and 6.19 above,

\[ \frac{d\tau_s}{d\gamma} = \frac{d\tau_s}{d\rho_{\text{debris}}} \times \frac{d\rho_{\text{debris}}}{d\gamma} \]  

(6.23)

or

\[ \frac{d\tau_s}{d\gamma} = f \theta_0 / \tau_s \times \frac{\tau^2}{\tau_s} \]  

(6.24)

In Stage IV, the flow stress is very close to the current saturation stress and so Eq. 6.24 can be approximated as

\[ \frac{d\tau_s}{d\gamma} = f \theta_0 \]  

(6.25)

This predicts that the transition to Stage IV will occur at almost the same work hardening rate, of order \( f\theta_0 \), for any Stage III behavior.

Further, the work hardening rate in Stage IV remains constant at this level indefinitely. These conclusions are made more apparent in the numerical analysis that follows.

VI.4.f Numerical results of DADR model

This model was incorporated into a FORTRAN program, listed in Appendix D, for convenience in plotting the results. The key parameter is the fraction of dynamic recovery events that store debris, \( f \), which was chosen to be 1/25 because the value of 1/15 discussed above gives
hardening rates in Stage IV that are higher than those observed experimentally. The results of running the calculations are shown in Fig. 6.4 for various values of the saturation stress that would apply to the end of Stage III. The plots indicate that Stage IV intervenes at the same hardening rate, regardless of the prior small strain history and continues without apparent limit.
Fig. 6.4 Plot of Hardening Rate versus Stress for the DADR model. All quantities have been normalized by the shear modulus and are in terms of resolved shear stresses on a slip plane.
Table 6.2 lists some of the numerical results which show that the debris density required to achieve Stage IV behavior is essentially the same in the Debris Accumulation by Dynamic Recovery model (DADR) and the Debris Accumulation by Glide model (DAG) described in Section VI.3. The number density of loops, mean size 10 nm, is plotted as a function of resolved shear strain in Fig. 6.5 for the five different Stage III behaviors, using the DADR model. The DADR model implies that the debris accumulates inside the dislocation tangles which at large strains are the cell walls. Dislocation loops in cell walls are harder to observe than when they are distributed throughout the material. This may explain the fact that the predictions of debris density are higher than the observed densities.

The effect of debris accumulation on flow stress is quantitatively similar in the DADR model as in the DAG model. Therefore the stored energy would still be expected to scale with the square of the stress. Therefore it would appear that stored energy considerations cannot be used to distinguish the two models.
Fig. 6.5 Plot of the number density of dislocation loops, mean size 10 nm, as a function of shear strain for the DADR model. The number density increases with increasing stress in Stage III. The five curves correspond to the five curves in Fig. 6.4.
Table 6.2A
Results of Numerical Solution for the DADR model

Part A; Small Stresses in Stage III

<table>
<thead>
<tr>
<th>$\gamma$</th>
<th>$10^3 \tau/G$</th>
<th>$\rho_{\text{debris}}$</th>
<th>loop # density</th>
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<td>0.92</td>
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<td>0.575E+21</td>
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<td>1.04</td>
<td>0.155E+14</td>
<td>0.155E+22</td>
</tr>
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<td>0.263E+22</td>
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<td>0.380E+22</td>
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<td>0.505E+22</td>
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Part A. saturation stress at the end of Stage III = 25 MPa = 0.00094G, equivalent to a monopolar dislocation density of $2.10^{14}$ m$^{-2}$. Loop number density calculated using a mean loop size of 10 nm.
Table 6.2B

Part B: Large Stresses in Stage III

<table>
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<tr>
<th>$\gamma$</th>
<th>$10^3 \tau/G$</th>
<th>$\rho_{\text{debris}}$ mm$^{-3}$</th>
<th>loop # density m$^{-3}$</th>
</tr>
</thead>
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<td>0.384E+21</td>
</tr>
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<td>0.813E+22</td>
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<td>0.164E+23</td>
</tr>
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<td>0.257E+23</td>
</tr>
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<td>0.847E+23</td>
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<td>10.00</td>
<td>6.04</td>
<td>0.906E+15</td>
<td>0.906E+23</td>
</tr>
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</table>

Part B: Saturation stress at the end of Stage III $= 125$ MPa $= 0.0047G$, equivalent to a monopolar dislocation density of $10^{15}$ m$^{-2}$. Loop number density calculated using a mean loop size of 10 nm.
VI.4g. Recovery of Debris

It is obvious from the experimental data reviewed above that at intermediate temperatures Stage IV occurs but is limited in extent. If Stage IV is indeed caused by the accumulation of dislocation debris then there must also be recovery mechanisms for the debris. The possibilities for diffusion controlled recovery have been explored by Prinz & Argon and by Nix et al. but with the drawbacks already discussed, see section II.3d. The work on irradiated metals, see for example the review by Wechsler [6.10], has shown that dynamic recovery of prismatic loops leads to "dislocation channeling" or localization of slip in metals with high loop concentrations. The experimental stress-strain tests show that the materials have a raised yield stress, as compared with well-annealed material, but much lower work hardening rates. Saada and Washburn discussed specific mechanisms [6.11] of interaction between mobile dislocations and prismatic loops that would lead to loss of the loop without long range diffusion. There are several examples in the literature of dynamic recovery of loops in both irradiated materials, Makin [6.12], and from quenching and aging experiments, Vandervoort and Washburn [6.13]. The latter authors pointed out the equivalence between a thermal recovery treatment, ten minutes at 473K, and a plastic deformation, 5% by rolling, for removing a loop density of $10^{21} \text{ m}^{-3}$ from quenched aluminum. This is a further reason to consider recovery at large strains, in Stage IV, to be dominated by dynamic recovery rather than necessarily involving diffusion as the rate limiting step.

This observation can be incorporated into the quantitative model outlined above as follows. If dynamic recovery of loops occurs by
intersections between mobile dislocations and loops then the rate of loss of loops is proportional to the number density of loops,

\[ \frac{dn}{d\gamma} = k'' n \quad (6.26) \]

where \( n \) is the number density of loops and the constant of proportionality, \( k'' \), will be left as an adjustable parameter for now. The simplifying assumption is now made that the mean loop size is constant so that Eq. 6.26 can be written in terms of the line density of loops,

\[ \frac{d\rho_{\text{debris}}}{d\gamma} = k'' \rho_{\text{debris}} \quad (6.27) \]

There is an obvious analogy here to the model for dynamic recovery in Stage III that was discussed in the review chapter; that is, the rate of loss of line density is proportional to the density itself. The major difference between the models, however, is that the Stage III model includes all strain rate and temperature effects into the proportionality constant; this discussion of Stage IV assumes that the intersection rate is determined by geometrical considerations such that a single value of \( k'' \) can be used.

The results of incorporating dynamic recovery of loops, Eq. 6.27 above, into the quantitative model for Stage IV given in section VI.4.e is displayed in Fig. 6.6, where \( k'' \) was set at 0.1. This value is reasonable if it is recalled that the mean distance that each mobile dislocation moves in a strain increment is \( d\gamma/b\rho_m \); if each mobile dislocation sweeps approximately one Burgers vector thickness then the volume fraction swept is \( d\gamma/b\rho_m \times b\rho_m = d\gamma \). The constant of proportionality, \( k'' \), in Eq. 6.26 is less than unity because not all interactions between mobile dislocations and stationary loops lead to elimination (recovery) of the loop; Saada and Washburn [6.11].
These plots show that, rather than remaining constant, the hardening rate in Stage IV slowly decreases in a manner that produces a nearly linear plot on the graph of $\theta$ against $r$. The hardening rate at which the transition to Stage IV occurs is essentially unaffected, and so is the sharpness of the transition.
Fig. 6.6 Plot of Hardening Rate versus Stress for the DADR model, including the effect of dynamic recovery of the debris. All quantities have been normalized by the shear modulus and are in terms of resolved shear stresses on a slip plane.
VI.4g Temperature and the Effect of Static Recovery on Stage IV

At a high enough temperature it is reasonable to expect that climb of the components of a dipole would lead to annihilation of the dipole in times short enough to nullify any effect on work hardening. For the dislocation dipole spacings expected in tangles, it can be calculated that bulk diffusion would not be effective until approximately 0.5 of the melting point. For prismatic loops, one half the melting point is also the temperature above which the recovery rate, controlled by bulk diffusion, becomes rapid, Smallman and Westmacott [6.14]. Both these considerations suggest that if Stage IV is controlled by debris accumulation, Stage IV will not be observed above 0.5 Tm. This prediction is not well tested by this work but some of the experimental data are believed to support it, as discussed below.

These qualitative observations can be made quantitative to a limited extent. An estimate of the static recovery rate of prismatic loops by bulk diffusion at the highest temperature used in this work, 473K, however, indicates that Stage IV should still be effective. The estimate is made as follows. The shrinkage rate, dr/dt, of a loop is given by an equation from Hirth and Lothe, Eq. 15-108 of ref. [6.15],

\[
\frac{dr}{dt} = D_s \frac{V G}{2(1-\nu)} R k T
\]  

(6.28)

where \(D_s\) is the bulk diffusion coefficient, \(V\) is the atomic volume, \(G\) the shear modulus, \(R\) the size of the loop and the other symbols have their usual meanings. The rate of loss of line length of loop is simply \(2 \pi \frac{dr}{dt}\), so the rate of loss of total debris line density due to loop shrinkage is

\[
\frac{d\rho_{\text{debris}}}{dt} = \frac{n 2 \pi D_s V G}{2(1-\nu) R k T}
\]  

(6.29)
Peterson et al. [6.16] recently discussed the experimental data for the diffusion coefficient of aluminum and concluded that the best value to take for activation energy is 1.3 eV or 121000 J.mole\(^{-1}\), so that,
\[
D_s = 1.7 \times 10^{-4} \cdot \exp(-121000/RT) \text{ m}^2\text{s}^{-1}
\] (6.30)
A representative value of the loop number density, \(n\), for a mean loop size of 10 nm, is \(10^{21} \text{ m}^{-3}\), as calculated from the model of section VI.4.e. At a temperature of 473K, with
\[
\begin{align*}
D_s &= 1.2 \times 10^{-19} \text{ m}^2\text{s}^{-1} \\
V &= 1.65 \times 10^{-29} \text{ m}^3 \\
G &= 2.7 \times 10^{10} \text{ N.m}^{-2} \\
k &= 1.38 \times 10^{-23} \text{ J.K}^{-1},
\end{align*}
\]
the static recovery rate of loops is calculated as \(5.10^{-9} \text{ m.s}^{-1}\), multiplied by the loop density, giving \(d\rho_{\text{debris}}/dt = -5 \times 10^{12} \text{ m}^{-2}\text{s}^{-1}\). For this loop size and density, the loop line density, \(\rho_{\text{debris}}\), is \(10^{13} \text{ m}^{-2}\) so the estimated recovery rate is more than adequate to explain the observation of Vandervoort and Washburn [6.13] that ten minutes at 473K sufficed to anneal out a loop number density of \(10^{21} \text{ m}^{-3}\).

Turning now to consideration of Stage IV, an estimate of the accumulation rate of loops from the model of section VI.4.f gives
\[
\frac{d\rho_{\text{debris}}}{d\gamma} = 4.2 \times 10^{13}
\] (6.31)
To obtain the accumulation rate per unit time, multiply by the strain rate, where the "standard" strain rate used in this investigation is \(6.9 \times 10^{-3} \text{ s}^{-1}\) in shear strain terms,
\[
\frac{d\rho_{\text{debris}}}{dt} = \frac{d\rho_{\text{debris}}}{d\gamma} \times \frac{d\gamma}{dt} = 1.8 \times 10^{11} \text{ m}^{-2}\text{s}^{-1}
\] (6.32)
This result is clearly less than the estimate for the static recovery rate obtained above, suggesting that in aluminum at \(T/T_m > 0.5\), prismatic
loops would not be expected to accumulate and lead to a Stage IV behavior. Note that at 100°C or 373K, the self-diffusion coefficient of aluminum is more than three orders of magnitude smaller than the value given above for 473K; this suggests that static recovery of prismatic loops by bulk diffusion should be negligible at T/Tm<0.5 in aluminum.

VI.5. Summary

Two models have been developed for Stage IV in single-phase metals. More emphasis has been placed on the "Debris Accumulation from Dynamic Recovery" model based on debris generation by recovery events for reasons that will become apparent in the Discussion. For debris in the form of prismatic loops, it was shown that significant accumulations of loops are unlikely above T/Tm>0.5 in aluminum. A model has been presented for Stage IV in two-phase materials where the second is nearly enough continuous to limit the mean free path of dislocations. Whereas the single-phase models predict an approximately constant work hardening rate in Stage IV, the two-phase model predicts a rising work hardening rate.

VI.6 References


Chapter VII. Discussion

VII.1 Introduction

This discussion of the results presented above will be divided into two parts, a longer part on single-phase materials and a shorter part on two-phase materials. The over-aged Al-Cu alloy is the only alloy discussed in the two-phase section, VII.4, and all the other alloys are discussed in section VII.5. The objective of the discussion is to compare the experimental results obtained here with the models. Additional support for the models is sought in the data from the literature. Before the models of large-strain strain hardening are dealt with, however, the topics of misorientation and strain reversal are discussed.

VII.2 Misorientation between Subgrains

The results of the TEM study suggested that large misorientations develop between cells or subgrains at large strains. The misorientations are large enough, >20°, as to constitute high angle grain boundaries. The creation of high angle grain boundaries has been reported by Chandra et al. [7.1] and Heilmann et al. [7.2].

One of the proposed causes for Stage IV hardening that was reviewed in Chapter II was the misorientation increase at cell or subgrain boundaries. Since many metals exhibit an increase of strength with decreasing grain size it has seemed plausible that the creation of high angle boundaries might lead to a strengthening at high strains. Even the limited data on misorientation presented above support the idea of grain boundary creation. The same data, however, show that equally large misorientations develop in the Al-1Mg alloy as in the commercial
purity aluminum whereas the Stage IV behavior at room temperature is very different. That is, the alloy shows a marked Stage IV with a large cumulative increase in flow stress but the unalloyed material does not and yet both show the development of high angle boundaries.

It might be argued that Al-Mg alloys show a grain size effect, Rossig [7.3], whereas unalloyed Al shows very little, Hansen [7.4]; therefore it might be thought reasonable that the alloy would show a Stage IV but not the unalloyed material. Unalloyed aluminum does show a classical Stage IV behavior at low temperatures, however. Unless aluminum were to exhibit a Hall-Petch effect at 77K but not at room temperature, this appearance of Stage IV at low homologous temperatures is not predicted. Thompson [7.5] has suggested that the Hall-Petch effect is only effective for grain sizes larger than 1 μm. The subgrains in Al-1Mg during Stage IV are appreciably smaller than 1 μm. The conclusion is that grain size strengthening is unlikely to be the cause of Stage IV.

The Hall-Petch strengthening effect rises with magnesium concentration in Al-Mg alloys, Rossig [7.3]. If subgrain strengthening occurs in the same way as the Hall-Petch effect for grain size strengthening at yield, an increase in Stage IV hardening rates might be expected. The evidence presented before does not support this however. Beyond this qualitative argument there is the more fundamental problem of what controls the refinement of the structure. That is to say, the Hall-Petch relation is a statement about the current flow stress of a material. For hardening to occur, the controlling unit size, be it grain size at yield or subgrain size, must decrease and the hardening rate is controlled by the rate of decrease of unit size. Decreasing the unit
size requires the accumulation of dislocation density which means that the hardening rate, $\theta_{IV}$, is still controlled by rates of accumulation of dislocation density.

The conclusion drawn from this brief discussion of misorientation angle at subgrain boundaries is that the evidence does not support the concept that Stage IV work hardening is due to an increase of misorientation at the cell or sub-grain boundaries with strain.

VII.3 Texture Development after Strain Reversal

The retention of a deformation texture after returning to zero net strain from a large strain in torsion was verified (Ch. IV). It was also shown that the current texture simulation codes do not predict this result but tend to return the texture to the initial texture. Texture development under monotonic straining is currently simulated by models that only take account of the geometry of limited slip of dislocations. This accounts for the reversibility of texture development in the present models. Work hardening is an example of an irreversible property of plastic deformation. It was demonstrated in Chapter III that reversing the sense of twist does not "undo" the work hardening; on the contrary, work hardening continues after a transient as if no reversal had occurred. Another irreversible change that occurs is the refinement of the structure into subgrains with surprisingly large misorientations between the subgrains. Taken together, the evidence suggests the need to modify the existing texture simulation models in order to take account of irreversible microstructural changes.

VII.4 Two-Phase Materials

The data for the overaged Al-Cu alloy showed an apparent increase in the work hardening rate during Stage IV. The microstructure showed
that the precipitates were elongated by the straining into plate like forms so that a semi-continuous second phase was formed. The model for two-phase materials suggested that the hardening rate should be proportional to the flow stress at large strains and that the slope would be 1/4 for the specific case of axisymmetric tensile deformation. The data from fiber-reinforced metal-metal composites obtained by Bevk [7.6] and by Embury and Fisher [7.7] on pearlitic steel appear to support this model. Figure 7.1 plots (in measured quantities) the hardening rate versus stress for these data and shows the trend predicted by the model of increasing hardening rate. Some caveats are necessary at this point, however. For the case of pearlitic steel, Embury and Fisher [7.7] discussed a model for the hardening behavior based on a Hall-Petch analysis. This model is satisfactory apart from the usual difficulty of calculating from first principles what the constant of proportionality should be between the strength and the characteristic dimension (grain size). Also for pearlitic steel, Langford [7.8] showed that each grain tends to deform in plane strain so that he had to include the effect of fragmentation of the cementite plates into his model in order to account for the observed hardening rates.

The Al-0.8Mn alloy investigated here is also a two-phase material. Its behavior is more like that of a single-phase material than a two-phase material. This is perhaps because the second phase is in the form of discrete, approximately spherical particles that are not sheared during deformation. Also the Mn that remains in solution has little effect on dynamic recovery, as evidenced by the TEM micrographs of strained Al-0.8Mn which have a similar appearance to those of the
commercial purity aluminum. For the purposes of this discussion then, Al-0.8Mn is regarded as a single-phase material because its second phase does not deform with the matrix.
Fig. 7.1 Hardening rate ($d\sigma/d\varepsilon$) plotted versus stress for wiredrawing of three different fiber-reinforced copper alloys, from Bevk [7.6] and data for overaged Al-4.6Cu (this work). Note that the Stage IV in Al-Cu is compressed into a short segment at the bottom of the curve. Bevk’s data lacks the descending branch because only high strain data was reported.
For the limited data available on large strain hardening in two-phase materials with continuous or semi-continuous second phases, the available models exhibit a hardening rate that rises one fourth as rapidly as the flow stress.

VII.5 Single-Phase Materials

VII.5a DAG model versus DADR model

Two alternative models for Stage IV work hardening in single-phase metals were presented in the previous chapter, the Debris Accumulation by Glide model (DAG) and the Debris Accumulation from Dynamic Recovery model (DADR). Two tests of the models favor the DADR model. The experimental data plotted as $\theta$ versus $r$ show that, when an extended Stage IV exists, the transition from Stage III to Stage IV is quite sharp. This sharp transition is documented both in the literature and the work on aluminum alloys reported in this thesis. Also, the hardening rate in Stage IV remains nearly constant during an appreciable rise in flow stress. The DADR model is compatible with these phenomenological aspects of Stage IV whereas the DAG model is not. Therefore the discussion that follows takes the DADR model as a basis for comparison with the experimental data.

VII.5b Variation with Material at Low Homologous Temperature

The model presented in Section VI.4e gives the result that the transition to Stage IV occurs at the same work hardening rate for any Stage III behavior. Figure 7.2 shows the work hardening behavior of all the single-phase alloys tested at 77K, 0.08Tm. The horizontal dashed line indicates the work hardening rate at which the transition appears to occur. The magnitude of the level suggests that the fraction $f$ in Eq. 6.19 has the value 1/25. This is a reasonable value when compared with
the crude estimate of $1/15$ obtained in section VI.4b and suggests that the debris generation process is somewhat less efficient than modeled above.
Fig. 7.2 Reduced hardening rate plotted versus reduced flow stress for various aluminum alloys tested in torsion at 77K, some tested at more than one strain rate.
Comparing now with data from other researchers and materials, Fig. 7.3 plots work hardening data, reduced in the same way as before, for:

a) Cu at 198K, 0.15Tm, Alberdi [7.9],

b) Commercial purity Al at 77K, 0.08Tm, Alberdi [7.9]

c) Ni at room temperature (RT), 0.17Tm, Hughes [7.10]

d) Ni-30Co at RT, 0.17Tm, Hughes [7.10]

e) Fe-0.049Ti, wire drawn at RT, 0.16Tm, Rack & Cohen [7.11].

The data plotted have been chosen to represent f.c.c. and b.c.c. alloys deformed at low homologous temperatures where diffusional recovery cannot operate. Once again, the transition to Stage IV occurs at approximately the same reduced work hardening rate in all these materials over a range of (reduced) stresses. The conclusion from these comparisons is that the model is at least a reasonable phenomenological model.
Fig. 7.3 Reduced hardening rate plotted versus reduced flow stress for various materials: wire drawn Fe-0.049Ti, Rack and Cohen [7.11]; Ni and Ni-30Co, torsion at room temperature, Hughes [7.10]; Al, torsion at 77K, Alberdi [7.9]; Al-1Mg, torsion at 77K, this work.
In some cases, the hardening rate decreases slowly in Stage IV, e.g. the Al-1Mg investigated in this work. The variation of flow stress at the same hardening rate in Stage IV with variations of strain rate (constant strain rate for each test, section III.4e) suggested that the recovery mechanism is dynamic rather than static. The inclusion of dynamic recovery in the DADR model of Stage IV work hardening, section VI.4g, reproduces the effect of a slow drop in hardening rate while retaining the sharp transition from Stage III to Stage IV that is such a characteristic of the phenomenon.

VII.5b Variation with Temperature, Constant Material

VII.5b.1 Commercial Purity Aluminum

Figure 7.4 plots the work hardening data for the large strain behavior of the commercial purity aluminum at four different temperatures. For all temperatures except the lowest, Stage IV makes a minor contribution to the flow stress. For the purposes of this discussion, Stage IV is defined as deviations from linear decrease of work hardening rate to zero at the end of Stage III. Using this definition, the dashed line in the figure represents an estimate for the transition to Stage IV as a function of temperature. This line shows a gradual decrease in the hardening rate at the onset of Stage IV with increasing temperature. On the basis of the DADR Model, this suggests a gradual drop in the fraction of recovery events that generate debris. This could be rationalized on the basis of the increasing sharpness of cell walls with increasing temperature in aluminum. A perfectly sharp cell wall is a low or high angle boundary and cannot contain any other (redundant, as opposed to geometrically necessary) dislocation segments. Any loops or dipoles arriving at a high angle boundary would be expected
to be absorbed. Alberdi’s data for commercial purity aluminum, with somewhat more solute present, Fig. 7.5, shows a similar pattern of behavior. The conclusion is that although the DADR model may be a good phenomenological description of Stage IV in the low temperature limit, further development will be necessary to account for \( f(T) \) in Eq. 6.19 (Ch. VI).
Fig. 7.4 Reduced hardening rate plotted versus stress for commercial purity aluminum (Al-0.17Fe-0.07Si) deformed at 473K, 373K, 293K and 77K in order of ascending flow stress.
Fig. 7.5 Reduced hardening rate plotted versus reduced flow stress for commercial purity aluminum, data taken from Alberdi [7.9], deformed at 373K, 293K, 198K and 77K in order of ascending flow stress.
VII.5b.2 Al-1Mg

The same plot of work hardening versus stress for the Al-1Mg alloy at four different temperatures and a single strain rate, $4 \times 10^{-3}$ s$^{-1}$, is shown in Fig. 7.6. In this case the stresses do not vary as smoothly with temperature as they did for the unalloyed aluminum because of the dynamic strain aging of the magnesium. The variation in onset of Stage IV, however, appears to show the same slow variation as before, suggesting a similar variation of the factor $f$ in Eq. 6.19.

This data has an interesting feature at the beginning of Stage IV which is a transient increase in the hardening rate before the hardening rate slowly declines. This "blip" in the work hardening curves says that for a small interval of stress, the work hardening is increasing in Stage IV. The data of Alberdi [7.9] on Cu, Fig. 7.7, shows the same type of transient increases in work hardening rate at the beginning of Stage IV. Neither one of the models of single-phase Stage IV work hardening developed in Chapter VI predicts an increasing work hardening rate in Stage IV, however, so this phenomenon remains unexplained.
Fig. 7.6 Reduced hardening rate plotted versus reduced flow stress for Al-1Mg deformed at 473K, 373K, 293K and 77K at a shear strain rate of 6.9 \times 10^{-3} \text{s}^{-1}. 
Fig. 7.7 Reduced hardening rate plotted versus reduced flow stress for copper deformed at 473K, 373K, 293K, 198K and 77K in torsion, data from Alberdi [7.9].
VII.6 References


Chapter VIII. CONCLUSIONS

The major conclusions of this work are as follows.

Stage IV work hardening has been confirmed to exist in aluminum and its alloys. Taken together with previous work on f.c.c. metals by Alberdi [7.9] and Hughes [7.10] and on b.c.c. iron by Rack and Cohen [7.11] for example, it is concluded that Stage IV occurs in all cubic metals for all deformation paths, provided that the deformation temperature is low enough.

A practical consequence of this conclusion is that analyses of the kinetics of plastic deformation based on the existence of a fixed saturation stress can be applied at low temperatures only in the range of small to intermediate strains. Even at one third of the melting point in aluminum (room temperature) a small amount of soluble alloy addition is enough to produce a prolonged Stage IV.

The rate of work hardening observed at low homologous temperatures (at which Stage IV is sustained over a large range of stress) is approximately $2.10^{-4}G$ in many cubic metals. This suggests that Stage IV is not sensitive to the stacking fault energy which contrasts with Stage III behavior.

Taking the results of varying temperature and solute content together yields a picture of Stage IV where the hardening rate in Stage IV is a constant in the low temperature limit. Raising the temperature of deformation has little effect on the alloys that contain solute. In pure materials, however, higher temperatures result in an onset of Stage IV at lower hardening rates. At the same time the hardening rate in
Stage IV decreases, with the result that the extent of Stage IV decreases.

Variation in the applied strain rate has little effect on the large-strain behavior. The lack of high strain-rate sensitivity at large strains indicates that dynamic recovery is more important than static recovery even at temperatures where pipe diffusion could play a role. Only for Al-1Mg deformed at 473K is there a marked variation in behavior: at a shear strain rate of $6.9 \times 10^{-1} \text{ s}^{-1}$ a Stage IV behavior is observed; at $6.9 \times 10^{-3} \text{ s}^{-1}$ however, the flow stress saturates.

Stage IV differs between single-phase and two-phase materials. The existing theory for two-phase materials appears to be adequate. For single-phase materials, however, the previously proposed models for Stage IV have been rejected on the basis of the current experiments and data from the literature.

The development of large misorientations to the point of creation of high-angle boundaries appears to be a general phenomenon at large strains and independent of the work hardening behavior. That is, large misorientations developed whether or not a Stage IV was observed. This was the basis for rejecting misorientation development as a cause of Stage IV.

A new model of Stage IV work hardening has been developed that is based on the accumulation of dislocation debris, e.g. prismatic loops. The debris is considered to be a byproduct of dynamic recovery events in dislocation tangles which lead to an increase of the strength of the tangles. The resulting model exhibits a sharp transition from Stage III to Stage IV and a constant work hardening rate in Stage IV at a reasonable level of work hardening. This is the behavior exhibited by
single-phase materials at low temperatures. When the effect of dynamic recovery of the debris is included in the model, the transition is smoother and the work hardening rate during Stage IV decreases, as is observed at higher temperatures.

If debris accumulation is the cause of Stage IV, as modeled in this thesis, then the above conclusions from the experiments can be rationalized as follows. The solute containing alloys are little affected by temperature because the solute stabilizes both the monopole dislocation networks (restraining the tendency to collapse into a cellular structure) and the dislocation loops. In the pure metals, however, raising the deformation temperature permits the formation of cellular dislocation structures in which recovery of debris becomes easier. Once dislocation debris can no longer accumulate, Stage IV does not occur.

Reversing the sense of torsion after a large prestrain only reduces the hardening rate transiently. After the transient, which may last for 50% strain, Stage IV work hardening is re-established at the same level as before the transient. The "constant structure" strain rate sensitivity appears unaffected by the reversal of torsion. Examination at zero net displacement has confirmed Backofen's result that the initial grain shape is restored.

Backofen's remarkable result was also confirmed that the texture is not restored to the initial texture upon complete strain reversal. This means that there is an irreversible component of texture development, as there is for work hardening, for example.
Chapter IX. FUTURE WORK

Five possibilities for future work are noted here:

1. The conclusion has been reached that Stage IV is a universal phenomenon at large strains in cubic metals. To the knowledge of the author, no large-strain tests have been made of Pb or its alloys at a low enough temperature that Stage IV should be observable. Pb is an interesting material because, like aluminum, it has a high ratio of stacking fault energy to shear modulus. This means that pure Pb should only exhibit a Stage IV at temperatures well below room temperature but that the addition of solute should raise the maximum temperature at which Stage IV is observable. Another f.c.c. metal for which large-strain tests are lacking is silver which has a small stacking fault energy. If it is true that stacking fault energy does not affect Stage IV behavior then silver should exhibit similar behavior to the other f.c.c. metals at large strains.

2. In the area of two-phase materials, it is possible to make directionally grown Al-Ni eutectics where the second phase is present as extended fibers. Large-strain tests of such a material would provide a confirmation of Bevk's work on filamentary composites based on copper. The suggestion was made in chapter VI (modeling) that there might be a deformation path dependence of Stage IV in two-phase materials. Therefore it would be worthwhile to compare wire-drawing with torsion.

3. The model of Stage IV in single-phase metals as developed here does not account for the variation in Stage III to Stage IV transition with temperature. Further development of the model is required in order to obtain a complete constitutive model for large strain behavior especially in view of the fact that large strains magnify small
differences in Stage IV hardening rate into large differences in flow stress.

4. Recovery experiments should provide a test of the presence of debris in heavily strained materials. Recovery of material with high debris content should show a noticeable decrease in stored energy and flow stress with little change in the observable dislocation structure.

5. The measurements of misorientation between neighboring subgrains should be confirmed and extended to ascertain whether this is a general phenomenon. Correlations should be possible to the macroscopically observed texture which suggests that the individual orientations should be not random.
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BIBLIOGRAPHY

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APPENDIX A

LIST OF SYMBOLS

\( \alpha \) geometric factor that characterizes an average of the various interaction strengths between dislocations

\( G \) shear modulus, decreases slowly with temperature

\( \tau \) flow stress: a critical resolved shear stress on a slip system

\( \sigma \) applied stress (a scalar measure)

\( b \) magnitude of the Burgers vector

\( \theta \) work hardening rate, \( d\sigma/d\epsilon \) or \( d\tau/d\gamma \)

\( \nu \) Poisson's ratio

\( D \) slip plane spacing

\( d_n \) number of dislocations extending through a slip plane in a given strain increment

\( d\gamma \) increment of shear strain

\( \rho \) dislocation density

\( M \) Taylor Factor, ratio of the applied stress for plastic flow to the resolved shear stress on a slip system

\( T \) temperature

\( T_m \) melting point

\( \theta_0 \) an initial \( \theta \), value of \( \theta \) in Stage II

\( \sigma_s \) "saturation stress" for Stage III, generally
measured by extrapolation to zero hardening rate of a
plot of hardening rate versus stress.

$\sigma_{s0}$ saturation stress at 0K
$
\tau_{III}$ resolved shear stress at which Stage III commences.

$\dot{\varepsilon}$ applied strain rate (s$^{-1}$)

$\dot{\varepsilon}_0$ a reference strain rate, of order $10^8$ s$^{-1}$

$A$ A parameter for thermal activation (the activation work) (J)

$k$ Boltzmann's constant

$D_s$ bulk diffusion coefficient (m$^2$.s$^{-1}$)

$V$ atomic volume

$R, <R>$ loop radius, mean loop radius.

$\varepsilon_{VM}$ von Mises equivalent strain
Listing of FORTRAN program used to calculate misorientations between a pair of orientations.

program cryst2
.
c from Erdogan Koken, McMaster University, 1986
c who obtained it from Faivre's thesis (Sussex University)
c modified again by Anthony Rollett, Los Alamos, 1987
.
c integer h,qqh
dimension title(20),phimin(50,50)
dimension a(25),qqa(50)
1,tet(25),qqtet(50)
2,b(25),qqb(50)
3,eta(25),qqeta(50)
4,psi(25)
5,n(25),iqqn(50)
6,m(25,3,2)
7,an(25,3,3)
8,alpha1(25,3)
9,beta1(25,3)

c tet(1)= angle betw. norm of 1st kik-line & ref axis,deg
dimension phi(24)
dimension alpha(24)
1,beta(24)
2,z1(24,3)
3,sign(25)
4,alpl(3)
5,h(25,3,2),qqh(50,3,2)
6,u(3)
7,v(3)
8,un(3)
9,vn(3)

c h(1,i,1)=indices of one kikuchi-pair of 1st subgr.
dimension w(3)
1,p(3)
2,q(3)
3,r(3)
4,pl(3)
5,q1(3)
6,x(3)
7,y(3)
8,z(3)
9,s(3)
dimension t(3)
1, d(3)
2, a1(3)
c normal distance between transm.
c spot and trace of one kik-pair
3, a2(3)
4, b1(3)
c normal dist as above for second kikuchi line
5, b2(3)
do 1, i=1, 50
do 1, k=1, 50
phimin(i, k)=0.0
1 continue
type*, 'Do you want output on the printer? (0=NO, 1=YES)'
accept*, iprint
type*, 'starting: maximum of 50 subgrains will be read'
open(unit=2, name='KIKIN.DAT', type='OLD', access='SEQUENTIAL')
read(2,33) title
read(2,*) c

c read in title and camera length
do 133, k=1, 50
read (2,*, err=2134) iqn(k), qqh(k,1,1), qqh(k,2,1), qqh(k,3,1)
1, qqa(k), qqtet(k), qqh(k,1,2), qqh(k,2,2), qqh(k,3,2)
2, qqb(k), qqeta(k)
if(qqtet(k).lt.0.0) qqtet(k)=qqtet(k)+360.0
if(qqeta(k).lt.0.0) qqeta(k)=qqeta(k)+360.0
type*, iqn(k), qqh(k,1,1), qqh(k,2,1), qqh(k,3,1)
1, qqa(k), qqtet(k), qqh(k,1,2), qqh(k,2,2), qqh(k,3,2)
2, qqb(k), qqeta(k)
133 continue

2134 type*, 'found EOF'
pi=3.14159
numsub=k-1
do 1301, ijk1=1, numsub-1
n(1)=iqn(ijk1)
a(1)=qqa(ijk1)
b(1)=qqb(ijk1)
tet(1)=qqtet(ijk1)
et(1)=qqeta(ijk1)
do 135, k=1, 3
h(1,k,1)=qqh(ijk1,k,1)
h(1,k,2)=qqh(ijk1,k,2)
135 continue
do 1301, lkji=ijk1+1, numsub
n(2)=iqn(lkji)
a(2)=qqa(lkji)
b(2)=qqb(lkji)
tet(2)=qqtet(lkji)
et(2)=qqeta(lkji)
do 136, k=1, 3
h(2,k,1)=qqh(lkji,k,1)
h(2,k,2)=qqh(lkji,k,2)
136 continue
c c=camera length, mm
c n= number of "only" two-subgrains
c k=1 = 1st subgrain
c k=2 = 2nd subgrain

do 60, k=3,25
a(k)=a(2)
tet(k)=tet(2)
b(k)=b(2)
etal(k)=etal(2)
n(k)=n(2)
60 continue

60 continue

loop 63: determination of all 24 possible orients. of cryst2

do 63, j=1,2

d type*, 'starting loop 63'
do 61, k=2,5
h(k,1,j)=h(2,1,j)
h(k+1,1,j)=h(2,2,j)
h(k+8,1,j)=h(2,3,j)
h(k+12,1,j)=-1*h(2,1,j)
h(k+16,1,j)=-1*h(2,2,j)
h(k+20,1,j)=-1*h(2,3,j)
61 continue

61 continue

h(3,2,j)=-1*h(2,2,j)
h(4,2,j)=-1*h(2,3,j)
h(5,2,j)=-1*h(2,3,j)
h(6,2,j)=h(2,3,j)
h(7,2,j)=h(2,3,j)
h(8,2,j)=h(2,1,j)
h(9,2,j)=h(2,1,j)
h(10,2,j)=h(2,1,j)
h(11,2,j)=h(2,2,j)
h(12,2,j)=h(2,3,j)
h(13,2,j)=-1*h(2,2,j)
h(3,3,j)=-1*h(2,3,j)
h(4,3,j)=-1*h(2,3,j)
h(5,3,j)=h(2,3,j)
h(6,3,j)=h(2,1,j)
h(7,3,j)=-1*h(2,1,j)
h(8,3,j)=-1*h(2,3,j)
h(9,3,j)=h(2,3,j)
h(10,3,j)=h(2,2,j)
h(11,3,j)=-1*h(2,2,j)
h(12,3,j)=-1*h(2,1,j)
h(13,3,j)=h(2,1,j)
do 62, k=2,13
h(k+12,2,j)=h(k,2,j)
h(k+12,3,j)=-1*h(k,3,j)
62 continue

63 continue

do 6, k=1,25
do 2, i=1,3
p(i)=float(h(k,i,1))/sqrt(float(h(k,1,1))**2+
1*float(h(k,2,1))**2+float(h(k,3,1))**2)
q(i)=float(h(k,i,2))/sqrt(float(h(k,1,2))**2+
1*float(h(k,2,2))**2+float(h(k,3,2))**2)
continue
tet1=tet(k)*pi/180.
etal=tet1+acos((prosca(p,q)*sqrt((a(k)**2+c**2)*(b(k)**2+
1e**2)))-a(k)*b(k))/c**2)
if (cos(eta(k)*pi/180.0-etal)-0.99) 3,4,43 
etal=2.0*tet1-etal
u(1)=cos(tet1)
u(2)=sin(tet1)
u(3)=a(k)/c
v(1)=cos(eta1)
v(2)=sin(eta1)
v(3)=b(k)/c
call univec(un,u)
call univec(vn,v)
call provec(un,vn,w)
call provec(p,q,r)
call provec(q,r,pl)
call provec(r,p,ql)
do 5, i=1,3 
do 8, j=1,3
an(k,1,1)=(pl(j)*un(1)+ql(j)*vn(1)+r(j)*w(1))/prosca(r,r)
continue
\psi(k)=acos(an(k,1,1)*an(k,1,2)+an(k,2,1)*an(k,2,2)+
1an(k,3,1)*an(k,3,2))*180.0/pi
sign(k)=-(an(k,2,1)*an(k,3,2)-an(k,3,1)*an(k,2,2))*an(k,1,3)+
1(an(k,3,1)*an(k,1,2)-an(k,1,1)*an(k,3,2))*an(k,2,3)+(an(k,1,1)
2*an(k,2,2)-an(k,2,1)*an(k,1,2))*an(k,3,3)
do 9, j=1,3
d type*, 'starting loop 9'
betal(k,j)=acos(an(k,3,j))*180.0/pi
if (1.0-an(k,3,j)**2) 22,21,22
21 alp1(j)=0.0
goto 20
22 alp1(j)=asin(an(k,2,j)/sqrt(1.0-an(k,3,j)**2))*180.0/pi
20 ALPHAL(K,J)=ALP1(J)
if (an(k,1,j).ge.0.0) goto 9
alphal(k,j)=180.0-alphal(k,j)
continue
6 continue
do 32, k=1,24 
do 7, i=1,3
a1(i)=an(1,i,1)
b1(i)=an(1,i,2)
a2(i)=an(k+1,i,1)
b2(i)=an(k+1,i,2)
7 continue
do 24, i=1,3
x(i)=a1(i)-a2(i)
y(i)=b1(i)-b2(i)
24 continue
call provec(x,y,z)
call provec(z,al,s)
call provec(z,a2,t)
call provec(s,t,d)
beta(k) = acos(z(3)/sqrt(prosca(z,z)))*180.0/pi
if (z(1)**2+z(2)**2) 11,10,11
10  alp=0.0
   goto 12
11  alp=asin(z(2)/sqrt(z(1)**2+z(2)**2))*180.0/pi
c
goto 12
12  if (z(1)) 13,14,14
13  alpha(k)=180.0-alp
   goto 15
14  alpha(k)=alp
c
goto 15
15  ph=acos(prosca(s,t)/sqrt(prosca(s,s)*prosca(t,t)))*180.0/pi
if (prosca(z,d)) 16,17,17
16  phi(k)=-1.0*ph
   goto 18
17  phi(k)=ph
c
goto 18
c Miller indices of the rotation axis
18  zl(k,1)=10.0*prosca(al,z)/sqrt(prosca(z,z))
zl(k,2)=10.0*prosca(bl,z)/sqrt(prosca(z,z))
zl(k,3)=sqrt(100.0-zl(k,1)**2-zl(k,2)**2)
   continue
c
write(7,33) title
if(iprint.eq.0) goto 1201
write(7,34)
write(7,35)
write(7,36) n(1), (h(1,j,1),j=1,3), a(1), tet(1), (h(1,j,2),j=1,3)
   1,b(1),eta(1),n(1)
write(7,40) n(2), (h(2,j,1),j=1,3), a(2), tet(2), (h(2,j,2),j=1,3)
   1,b(2),eta(2), (zl(1,j),j=1,3), alpha(1), beta(1), phi(1),n(2)
write(7,40) k, ((h(k+1,j,1),j=1,3),a(k+1),tet(k+1), (h(k+1,j,2),
   1j=1,3),b(k+1),eta(k+1), (zl(k,j),j=1,3), alpha(k), beta(k),
   2phi(k),k,k=2,24)
write(7,41)
write(7,42) (n(k), (alphal(k,j), betal(k,j),j=1,3), psi(k), k=1,2)
write(7,42) (k, (alphal(k+1,j), betal(k+1,j),j=1,3),
   lpsi(k+1), k=2,24)
write(7,43)
write(7,44) (n(k), ((an(k,i,j),i=1,3),j=1,3), sign(k), k=1,2)
write(7,44) (k, ((an(k+1,i,j),i=1,3),j=1,3), sign(k+1), k=2,24)
write(6,33) title
write(6,34)
write(6,36) n(1), (h(1,j,1),j=1,3), a(1), tet(1), (h(1,j,2),j=1,3)
   1,b(1),eta(1),n(1)
write(6,40) n(2), (h(2,j,1),j=1,3), a(2), tet(2), (h(2,j,2),j=1,3)
   1,b(2),eta(2), (zl(1,j),j=1,3), alpha(1), beta(1), phi(1),n(2)
write(6,40) k, ((h(k+1,j,1),j=1,3),a(k+1),tet(k+1), (h(k+1,j,2),
   1j=1,3),b(k+1),eta(k+1), (zl(k,j),j=1,3), alpha(k), beta(k),
   2phi(k),k,k=2,24)
write(6,41)
write(6,42) (n(k), (alphal(k,j), betal(k,j),j=1,3), psi(k), k=1,2)
write(6,42) (k, (alphal(k+1,j), betal(k+1,j),j=1,3),
   lpsi(k+1), k=2,24)
write(6,43)
write(6,44) (n(k),(an(k,i,j),i=1,3),j=1,3),sign(k),k=1,2
write(6,44) (k,(an(k+1,i,j),i=1,3),j=1,3),sign(k+1),k=2,24

336
337 format(1h1,20a4)
338 format(1h0,16hcamera length = ,f4.0)
339 format(1h0,43x,30hrotation between two subgrains/1h ,43x,30
1 (lh*)/1h0,6hsubgr.,6x,15hkikuchi lines a,12x,15hkikuchi lines b,
2 22x,13rotation axis,14x,5hangle,4x,6hsubgr./1h ,6(h1-),6x,
3 15(h1-),12x,15('-'),22x,13(h1-),14x,5(h1-),4x,6(1h-)/1h0,2x,2hno
4,5x,1hh,2x,1hh,1x,1hh,5x,1ha,5x,3htet,6x,1hh,2x,1hh,2x,1hh,5x,
5 1hb,5x,3heta,12x,1hp,4x,1hq,4x,1hr,4x,5halpha,4x,4hbeta,7x,
6 3hphi,7x,2hno/1h0)
343 format(1h ,2x,i2,3x,3i3,3x,f5.1,2x,f5.1,3x,3i3,3x,f5.1,2x,
1 f5.1,56x,i2)
346 format(1h0,2x,i2,3x,3i3,3x,f5.1,2x,f5.1,3x,3i3,3x,f5.1,2x,
1 f5.1,8x,3f5.1,3x,f5.1,3x,f5.1,4x,f7.2,6x,12)
349 format(1h1,26x,5h(100),26x,5h(010),26x,5h(001),23x,3hpsi/1h0
1 ,5x,2hno,13x,5halpha,7x,4hbeta,15x,5halpha,7x,4hbeta,15x,
2 5halpha,7x,4hbeta/1h )
352 format(1h0,5x,i2,12x,f6.1,6x,f5.1,14x,f6.1,6x,f5.1,14x,
1 f6.1,6x,f5.1,17x,f4.1)
356 format(1h1,26x,5h(100),30x,5h(010),30x,5h(001),15x,4hsign/
1 1h0,5x,2hno,12x,1hx,8x,1hy,8x,1hz,16x,1hx,8x,1hy,8x,1hz,16x,
2 1hx,8x,1hy,8x,1hz,/1h )
360 format(1h0,5x,i2,6x,3f9.3,8x,3f9.3,8x,3f9.3,5x,f4.1)

1201 phimin(ijk1,1kji)=360.0
1202 do 1250, kl=1,24 test=abs(phi(kl))
1203 if(test.1t.phimin(ijk1,1kji)) phimin(ijk1,1kji)=test
1204 continue
1301 continue
1302 type 33, title
1303 type 1350, ((phimin(i,j),j=1,numsub),i=1,numsub)
1350 format(50f7.3)
1351 print 33, title
1352 print 1380, (iqqn(i),i=1,numsub)
1380 format(x,16(x,17))
1381 do 1401, i=1,numsub
1382 print 1390, (phimin(i,j),j=1,numsub)
1390 format(x,16(x,f7.3))
1401 continue
1402 call exit
end

c subroutine prove(a,b,c)
dimension a(3),b(3),c(3)
c(1)=a(2)*b(3)-a(3)*b(2)
c(2)=a(3)*b(1)-a(1)*b(3)
c(3)=a(1)*b(2)-a(2)*b(1)
return
end
c subroutine univec(e,f)
dimension e(3),f(3)
do l, i=1,3
  e(1)=f(1)/sqrt(prosca(f,f))
  return
end

c
  function prosca(u,v)
    dimension u(3),v(3)
    prosca=u(1)*v(1)+u(2)*v(2)+u(3)*v(3)
    return
  end

c
  function acos(z)
    double precision s,t
    s=1.0
    t=z
    acos=datan2(dsqt(s-t**2),t)
    return
  end

c
  function asin(z)
    double precision s,t,u
    s=1.0
    t=z
    asin=datan2(t,dsqt(s-t**2))
    return
  end

end of Appendix B
APPENDIX C

FORTRAN program for Stage IV Model based on Continuous Debris Accumulation

program ml
  c calculates hardening rate and stress for a model of Stage IV
  c in which debris accumulates continuously during deformation
  dimension stresl(1000)
  open (unit=2, name='MODEL1.HIS',access='SEQUENTIAL',type='NEW')
  write (2,*) 'nosort'
  type *, 'Do you want a hard copy? [1=YES]'
  accept *, iqhd
  alpha=0.5
  g=2.7e10
  b=2.8e-10
  type*, 'value of k-prime? [e.g. le-3]'
  accept*, rkl
  rkl=rkl/b
  type*, 'value of tausl? MPa'
  accept*, tausl
  tausl=tausl*le6
  dtau=tausl
dg=0.05
  type*, 'What maximum theta [50 or 10]?'
  accept*,scale
  call initt
  xfac=4000.
yfac=2900.
call movabs(int(0.1*xfac),2900)
call drwabs(int(.1*xfac),int(.1*yfac))
call drwabs(int(1.*xfac),int(1*yfac))
do 200, j=1,5
  write (2,*) 'strain stress/G rho.debris loop # density'
s1=0.0e6
  s0=s1
  rho2=0.0
  gamma=0.0
  call movabs(int(0.1*xfac),2900)
do 100, i=1,500
  gamma=gamma+dg
  if (gamma.gt.15.0) goto 101
  s20ld=alphl*gb*b*sqrt(rho2)
s0old=s0
  dd=0.005*(s1/tausl)
  if(dd.gt.0.005) dd=0.005
  dsl=g*dd*(0.005-dd)
s1=s1+dsl
  200 coutinue
  101 coutinue
rhol=(s1/alpha/g/b)**2
drho2=rk1*sqrt(rhol)
rho2=rho2+drho2
s0=alpha*g*b*sqrt(rhol+rho2)
theta=(s0-s0old)/dg
if(mod(1,5).eq.0)
  1 write (2,51) gamma, s0/g*1e3, rho2, rho2/1e-8
      format(x,f7.2,x,f7.1,x,g13.3,x,g13.3)
      iy=int(theta/g*1e4/scale*xfac*0.9)+(0.1*xfac)
      if(iy.gt.int(yfac)) iy=int(yfac)
      ix=int(s0/g*1e3/10.0*xfac*0.9)+(0.1*xfac)
      if(ix.gt.int(xfac)) ix=int(xfac)
call drwabs(ix,iy)
      format(6g10.3)
100  continue
tausl=tausl+dtau
200  continue
call movabs(2000,2800)
if (iqhd.eq.1) call hdcopy
pause 'hit <CR> when ready!'
call finitt
call exit
end
APPENDIX D

FORTRAN program for Stage IV model based on Debris Accumulation by Dynamic Recovery

```
program ml
  dimension stresl(1000)
  alpha=0.5
  alphl=0.18
  g=2.7e10
  b=2.8e-10
  f=1/25.0
  type*,'Do you want a hard copy? [1=yes]'
  accept*,iq
  type*,'value of tausl? MPa'
  accept*, tausl
  tausl=tausl*1e6
  dtau=tausl
dg=0.05

  type *, 'sl, rrhol, thetal, s2, theta2, theta-total: /G*le4'
call initt
  xfac=4000.
yfac=2900.
call movabs(int(0.1*xfac),2900)
call drwabs(int(.1*xfac),int(.1*yfac))
call drwabs(int(1.*xfac),int(.1*yfac))
do 200, j=1,5
  sl=1.0e6

c  rho2=0.0
call movabs(int(0.1*xfac),2900)
taus=tausl
do 100, i=1,500

c  s2old=alphl*g*b*sqrt(rho2)
  dd=0.005*(sl/taus)
  if(dd.gt.0.005) dd=0.005
dsl=g*dg*(0.005-dd)
s1=sl+dsl
dts=f*0.005*g/taus1/taus*s1**2*dg
taus=taus+dts
  iy=int((sl)/dg/g*le4/50.*yfac*0.9)+(0.1*yfac)
  if(iy.gt.int(yfac)) iy=int(yfac)
  ix=int((sl)/g*le3/10.*xfac*0.9)+(0.1*xfac)
  if(ix.gt.int(xfac)) ix=int(xfac)
call drwabs(ix,iy)

  format(6g10.3)
  continue
  tausl=tausl+dtau
  continue
```
call movabs(2000,2800)
if(iq.eq.1) call hdcopy
pause 'hit <CR> when ready!'
call finitt
call exit
dend
VITA

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Professional Experience: Experience at Los Alamos has included work on Molecular Beam Chemistry, especially data acquisition; Metallography using Optical, Scanning Electron and Transmission Electron Microscopes; Design and Manufacture of Graphite Fuel Elements for Nuclear Reactors; programming of a Robot used for welding; operation of a Foundry; Computer Simulation of Metal Casting; Theoretical Prediction of Crystallographic Texture in Metals; Computer Simulation of Recrystallization and Grain Growth.


