# CREEP OF SINGLE PHASE METALS

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THE INFLUENCE OF SUBSTRUCTURE AND DISLOCATION MOBILITY ON THE CREEP PROPERTIES OF SOME SINGLE PHASE METALS

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by

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# Abstract

Single phase pure metals and alloys deformed at high températures show large variations in the form of their straintime curves and in the stress dependence of the strain rate. At present, it is not possible to predict theoretically the mechanical responses of a given alloy.

This thesis is concerned with the deformation of pure copper, copper-5 wt.% tin and iron-3½ wt.% silicon at temperatures above half the melting point. Mechanical experiments involving both constant load and constant strain rate tests have established that these three materials exhibit a spectrum of behaviour as wide as any previously reported. Detailed microstructural examination has revealed corresponding variations in dislocation arrangements and in the extent of grain boundary distortion in crept specimens.

It is argued that the form of the primary creep curve and strain transients following a change in stress, obtained in a given metal, is closely related to the mobility of mobile dislocations, rather than to an internal resistance arising from the substructure. To explain the observed mechanical responses, a model has been proposed which provides some semi-quantitative predictions of material behaviour. Both the mechanical and microstructural results suggest that the recovery theories of creep, in which hardening and recovery are considered as separately definable parameters, do not deal realistically with the creep

process.

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#### CHAPTER 1

#### INTRODUCTION

The mechanical behaviour of solids at elevated temperatures is controlled primarily by thermally activated processes. As a result the strength of metals decreases with increasing temperature. An important · ^ characteristic of high-temperature strength is that it must always be considered with respect to some time scale. Under constant stress conditions, deformation at low temperatures relative to the melting point is essentially independent of time, involving only a very limited transient strain. At elevated temperatures, a metal subjected to a constant tensile stress will undergo a time dependent change in length. This progressive deformation at constant stress is called creep. The differences in magnitude of creep at high and low temperatures are a reflection of the facility with which thermally activated processes can proceed.

Over the years a vast array of mechanical data has been collected describing the strain-time behaviour of many materials at elevated temperatures. This has led to considerable advances in the understanding of creep mechanisms. Sufficient progress has been made that several of the deformation mechanisms have been identified, and equa-

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tions developed, either theoretically or from an empirical evaluation of the data, to describe the strain rate under . steady-state conditions, where the rate of extension is independent of time.

This information can be used in the construction of a deformation mechanism map in which the normalised stress  $\sigma_{app}/G$ , is plotted as a function of the homologous temperature T/T<sub>m</sub>, at constant grain size, where  $\sigma_{app}$  is the applied stress, G is the shear modulus, T is the absolute temperature, and T<sub>m</sub> is the melting point of the material. Such maps are constructed by using the best available constitutive equation to describe each of the known mechanisms, so that the maps are then divided into fields of stress/temperature space within which a single mechanism dominates the deformation behaviour. An example of such a map is shown in Figure 1-1, indicating six independent ways in which a polycrystal can be deformed, three of which are creep mechanisms.

These maps have a considerable potential as an aid in the design and interpretation of experiments and for ease in handling data used in the design of engineering components such as turbine blades. The deformation map concept can be extended by the use of contour displays to show the effect of changing variables such as grain size or the spacing of obstacles to dislocation motion, but frequently this would demand a much better understanding of



Figure 1-1. A deformation mechanism map for pure aluminium (after Ashby, 1972).

creep than is presently available in order to generate more precise constitutive equations.

In the regime of interest in this thesis, that of dislocation creep, the models describing the mechanisms tend to be somewhat idealised derivatives of a basic matter transport process, such as dislocation climb. In their simplest forms, they bear little relation to the microstructural features actually encountered in crept specimens. In addition, the models are frequently unrealistically constructed so as to provide a theoretical agreement with the experimental value of the stress dependence of the steady state strain rate. Being derived from a description of the steady state, the simple models usually lack the more detailed description needed to obtain a strain-time relationship and thus cannot be used to predict primary or transient creep responses, that is, those immediately following a sudden change in stress. Yet it is these features which provide the most valuable information on material behaviour. Discrimination between models thus becomes very difficult, as is evidenced by the still continuing controversy over the importance of jog dragging screw dislocations relative to climbing edge dislocations.

In recent years, increasing attention has been paid to the problem of deciding the extent to which creep is dependent on the dislocation substructure. Improving tech-

niques of microstructural observation have permitted extensive examination of the internal structure resulting from creep deformation. The detailed dislocation structure becomes important because the dislocations are not uniformly distributed after deformation, but are arranged in roughly planar groups or cell walls. Unfortunately, the structural observations are frequently not capable of indicating the operating dislocation mechanism, particularly in the steady state, where there are no changes in macroscopic variables to which the substructure can be related.

There is thus a need for models relating the creep process to structural hardening, analogous to the work hardening theories applicable to low temperature deformation, which were developed over a decade ago. Such models would seek to provide predictions of the sub-grain size, at least in the steady state, and also descriptions of the structural changes accompanying a creep transient. The extent to which the mechanical response arises from structural tural effects should also be capable of assessment.

Commonly metallic solid colutions are divided into two categories in regard to creep behaviour. Those designated as class I materials exhibit a power law dependence of creep rate where the stress dependence is about 3, and short transients. Class II alloys exhibit a 5 power dependence and extensive transients, both of which are also

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shown by pure metals. It has been argued that sub-grain formation is a feature of class II material only. However, the increasing store of experimental data has been accompanied by revelations of more variable behaviour, and there are hybrid alloys which show a mixed class I-class II character.

This thesis is concerned with an assessment of the relationship between substructure and mechanical behaviour during high temperature deformation. In line with the foregoing comments, this study has been performed on several materials having basically dissimilar creep properties. It includes the mechanical measurement of both primary and transient creep responses as well as of those characteristic of steady state deformation. A theoretical description of these transient responses has been included to provide insight into the role of the substructure.

Each of the materials studied has also been subjected to microstructural examination. Apart from the measurement of substructural parameters, an attempt is made to assess the degree of homogeneity within the substructure. This is an important feature since the grain boundary deformation, slip bands and deformation bands, which are often found in crept specimens, might lead to inhomogeneities in the substructure. Thus, it is of interest to know the extent to which the substructure reflects the instantaneous

deformation rather than the strain history. An attempt has been made to model the behaviour of sub-boundaries during steady state creep so as to provide a prediction of the sub-grain size. The models have of necessity been restricted to views of uniform microstructural activity, that is without reference to grain boundary movements or deformation bands. Since the creep rates of metals are believed to be sensitive to the substructural changes that take place, the formation of a realistic creep theory must at least consider, and if possible predict, the significant substructural features in order to arrive at an appropriate description of high-temperature creep.

#### CHAPTER 2

# GENERAL DESCRIPTION OF HIGH TEMPERATURE CREEP BEHAVIOUR

#### 2-1. Introduction

The following chapter briefly reviews the current experimental findings concerning the basic aspects of high temperature deformation. The regime of interest deals with relatively coarse grained single phase materials at temperatures between 0.4 and 0.6  $T_m$  where  $T_m$  is the melting point, and strain rates below  $10^{-1}/min$ .

A certain emphasis has been placed on the description of dislocation substructure and creep transients as well as on those features which provide indications of i differences in behaviour between pure metals and certain of their alloys. These aspects will be of primary interest in the experimental work described later on.

Garofalo (1965), Sherby and Burke (1967), Mukherjee et al (1969) and Lagneborg (1972) have all published detailed reviews, but with differing emphasis on substructural effects.

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Any description of high temperature deformation must necessarily touch upon the concepts of thermally activated flow and internal stress. Appendix A provides a brief summary of their underlying theory.

### 2-2. Creep Curves

The strain-time plots obtained from isothermal constant stress uniaxial tensile tests on annealed single phase materials can be described in terms of a three-stage curve. Stages II and III are regions of constant and accelerating strain rate, respectively, though the duration of each stage varies considerably from one material to another. Two fundamentally different types of stage I behaviour are encountered and these are depicted schematically in Figures 2-1(a) and 2-1(b). Pure metals and a large number of solid solutions have primary regions over relatively large strains and of decreasing strain rate (Figure 2-1(a)). This is designated "normal" primary creep. Another group of solid solutions shows a relatively small primary strain within which the creep rate may be increasing or decreasing (Figure 2-1(b)).

Increasing amounts of prior cold work may cause a gradual change in the shape of the stage I from one of to one of increasing creep rate, al-



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Figure 2-1. Types of Creep Curve. (After Bird, Mukherjee, and Dorn, 1959).

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generally the same as in the annealed metal. Recrystallization or phase changes occurring during deformation may also cause distortions in the shape of the strain-time curve.

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The third stage of creep is associated with the onset of localised necking and the formation of cavities, particularly at grain boundaries, leading to tensile fracture. It is apparent that the extent of stage III will depend critically on the microstructural stability of the material. In addition, if testing is performed under constant load conditions rather than constant stress, the increasing stress during straining tends to promote longer stage III strains at the expense of stage II. The terms secondary creep, steady state creep and stationary creep all refer to stage II.

## 2-3. Empirical Strain-Time Creep Laws

Various relationships have been used to describe creep behaviour during stages I and II. At intermediate and high temperatures (above 0.4  $T_m$ ), the most satisfactory one is of the form:

$$\varepsilon = \varepsilon_0 + \varepsilon_t (1 - \varepsilon_1^{-at}) + \dot{\varepsilon}_s t$$
 (2-1)

where  $\varepsilon_0$  is the instantaneous strain on loading,  $\varepsilon_t$  is the limiting transient creep strain, and  $\varepsilon_s$  is the secondary creep rate, a is a constant (see Figure 1(a)).

This type of relation appears to give good fits to experimental data for a variety of pure metals and alloys showing "normal" primary behaviour (Garofalo, 1965). The instantaneous strain,  $\varepsilon_0$ , increases approximately parabolically with applied stress, implying a preponderance of work hardening during this plastic extension similar to that found in low temperature deformation.

There appear to have been no attempts to fit a relation such as (2-1) to the types of curve shown in Figure 1(b) other than by considering only that part of primary creep during which the strain rate is decreasing.

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### 2-4. Stress and Temperature Dependence of Creep Rate

Steady state creep rates that are measurable in a laboratory experiment (that is, greater than about  $10^{-6}$ /min) are found at stress levels greater than about  $10^{-5}$  G, where G is the average shear modulus, and at temperatures greater than about  $T_m/3$  where  $T_m$  is the melting temperature. This stress and temperature dependence arises predominantly

is a very vated process.

Thus local thermal activation provides added energy, beyond that provided mechanically, to overcome barriers to the transport processes giving rise to creep deformation. The stress both helps to overcome these barriers and also gives direction to the resultant flow.

Fairly coarse grained pure metals and solid solutions stressed between  $10^{-3}$  G and  $10^{-5}$  G at intermediate temperatures usually obey a power law relation:

$$\dot{\varepsilon}_{g} = B \sigma_{app}^{n}$$
 (2-2)

where  $\dot{\epsilon}_s$  is the secondary creep rate, and  $\sigma_{app}$  is the applied stress, B is a temperature dependent constant related to the self diffusion coefficient. On the basis of the value of the constant n, it is found that such materials can be roughly classified into two groups. Pure metals and the so-called class II solid solutions have n values ranging from 4 to 7, while another group of solidsolutions (class I) have n values ranging from 3 to 4 (Sherby and Burke, 1967). For the purpose of this thesis, class II materials are taken to include pure metals.

At stresses greater than about  $10^{-3}$  G the creep rate increases faster with increasing stress than predicted by equation (2-2). An expression of the form: 13

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$$\dot{\epsilon}_{s} = A (sinh (\alpha \sigma_{app}))^{n}$$
 (2-3)

has been advanced (Garofalo, 1965) to provide a unified expression of the power law at lower stresses and of the exponential law at high stresses, such that a change in mechanism does not have to be invoked.

Cannon and Sherby (1970) have proposed a criterion for class I and II behaviour in terms of the size difference between solute and solvent, and the solvent elastic modulus. Class I alloys have high size differences and relatively low moduli. The correlation appears to be good (see Figure 2-2) but at present is largely empirical.

Thermally activated processes show a rate dependence on temperature predominantly through the Arrhenius factor, and the general expression for the creep rate is of the form (Garofalo, 1965):

$$\dot{\varepsilon} = \Sigma_{i} Z_{i} (v, T, S) \sigma_{i} (T, S) \exp \frac{-\Delta F_{i} (T, S)}{kT}$$
(2-4)

Here  $\Delta F_i$  is the activation energy for the i<sup>th</sup> mechanism. If several mechanisms operate simultaneously but independently, the fastest one will require the lowest activation energy and control. If they operate dependently, the slowest one, requiring the largest activation energy, will control. The function  $Z_i$  may depend on temperature T, the frequency of the flow unit v, and on the structure term S which itself includes the number, distribution and length of dislocations, the grain size, and other geometrical details. The stress function is also temperature and structure dependent.



Figure 2-2. Grouping of alloys according to volume size factor and elastic modulus. (After Cannon and Sherby, 1970).

2<sub>i</sub> may also include an entropy term but in practice this is usually neglected.

Since the precise forms of the functions  $Z_i$  and  $\sigma_i$ are not known, experimental procedures are generally devised to maintain them constant (i.e., by abrupt changes in temperature to give before and after creep rates at constant structure) so as to determine the apparent activation energy. A linear plot of ln  $\dot{\epsilon}$  against  $\frac{1}{T}$  implies a single rate controlling process and the slope gives the activation energy.

The apparent activation energy for creep in single phase material has generally been shown to be in good agreement with that for self diffusion, and this correlation is improved if the temperature dependence of the elastic modulus contained in the pre-exponential term is allowed for. Lack of detailed diffusion data for most alloys has generally restricted these comparisons to pure metals, but those alloy systems for which it has been done have also yielded good agreement.

Dorn and his co-workers (Mukherjee, Bird and Dorn, 1968) have combined the stress and temperature dependencies of creep rate into a semi-empirical expression for the steady state creep rate, viz.:

 $\frac{\varepsilon_{B} kT}{T} = A \left(\frac{\sigma_{ADD}}{G}\right)^{n}$ 

(2-5)

where D is the self diffusion coefficient, b the burgers vector, G the shear modulus,  $\sigma_{\rm app}$  the applied stress, T the temperature, and k Boltzmann's constant. This is a form of equation (2-4) at constant structure. It is argued that using this expression, materials can be grouped into such crystallographic categories as FCC pure metals and FCC class II solid solutions, BCC pure metals, class I alloys, etc., for which a common A and n value will predict creep rate values within at worst an order of magnitude of experimental rates, and frequently much closer.

### 2-5. Grain Boundary Contributions to Creep

For grain sizes above a critical value (typically about 0.05 mm) there appears to be no appreciable grain size effect on the secondary creep rate. In particular, the creep rates for single crystals are reasonably close to those for polycrystals of the same material over the same stress range. The experimental evidence is, however, inconclusive and there is little information regarding the grain size effect in alloys.

Grain boundary sliding and migration are prominent features of polycrystalline materials deforming in creep. Both reflect the details of plastic flow in the grains

themselves and the continuity requirements of the aggregate, and through the associated development of grain boundary serrations and voids, have an important effect on creep ductility.

Except at small grain sizes, grain boundary flow appears to contribute an approximately constant fraction of the total strain during both primary and secondary creep. This implies that the boundaries do not have a controlling influence on the deformation and that the sliding and migration processes are controlled by the deformation within the grains. The ratio  $\varepsilon_{\rm gb}/\varepsilon_{\rm total}$  at constant stress is found to be largely independent of temperature, but it increases as the stress is decreased. The activation energy obtained from grain boundary sliding experiments agrees with those for self diffusion, which again suggests a close relationship between the deformation of the grain boundaries and of the interior of the grains (Stevens, 1966).

Frequently, the boundary flow is found to be initially localised close to the boundary but subsequently to take place over a boundary zone in the neighbourhood of which finer subgrains may be found than elsewhere. The displacements are generally non-uniform along the boundary and extensive sliding and migration may result in the formation of serrations in the boundary due to the interaction of migrating boundaries with subgrain walls. In copper above a

grain size of 0.1 mm, grain boundary processes contribute about 15% of the total strain above 0.5  $T_m$  (Sherby and Burke, 1967). Similar values have been reported for aluminium and aluminium-magnesium alloys (which generally show class I behaviour).

### 2-6. Substructural Changes During Creep

The energy of a crystal containing randomly distributed dislocations is lowered when they are removed or when they rearrange themselves into planar groupings of more or less regularly spaced individual dislocations, particularly when these are free of long range internal stresses. The formation of tilt and twist boundaries during annealing of a crystal deformed at low temperature is a manifestation of this fact. The annealed crystal consists of blocks of relatively stress free material separated from each other by sub-boundaries and which are slightly misoriented from one another.

Deformation at very low temperatures (i.e., at  $77^{\circ}X$ in iron (Keh, et al, 1963)) produces a random distribution of dislocations. As the deformation temperature is increased, dislocation mobility increases through the increasing ease of such thermally activated processes as dislocation inter-

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section, cross-slip, interactions between dislocations and point defects, and climb. As a result, those dislocations which survive the annihilation processes occurring within the crystal, tend increasingly to arrange themselves in subboundaries, and the overall dislocation distribution becomes increasingly inhomogeneous. Correspondingly the dislocations within the sub-boundaries themselves tend to become more regular as the testing temperature increases. Such an arrangement is known as a cell structure or sub-grain structure.

It has long been recognised that the change from primary to secondary (creep implies the development of a steady state among those substructural parameters which influence the macroscopic flow. As a result, a considerable effort has been made in recent years to determine precisely which microstructural features do reach an equilibrium, and how these features relate to the externally measured macroscopic variables.

Among the important substructural parameters are considered to be the tendency to subgrain formation and the nature and distribution of dislocations within the subgrain walls and in the cell interior. In addition, the relationships between stress, strain and temperature on the one hand, and the dislocation density, subgrain diameter and misorientation across the sub-boundaries on the other, are frequently

determined.

The tendency to form subgrains has frequently been related to the existence of "normal" (i.e., that typical of pure metals) primary creep, involving large strains, and materials showing primary behaviour involving only small strains, or showing "inverse" primary creep, particularly class I alloys, were thought not to be subgrain formers. Pure metal behaviour seems to confirm this criterion, but there seems to be a spectrum of alloy behaviour in which some alloys showing inverted primary creep strains and low stress exponents do in fact form subgrains (Fuchs and Ilschner, 1969) while a few cases also exist where a fairly large primary creep and/or high stress exponents do not coincide with early subgrain formation (Evans and Wilshire, 1970). This diversity of behaviour may be linked to variations in stacking fault energy (which may also influence the subgrain size) but it could also be taken to imply that the characterisations of class I and class II materials do not necessarily relate to differences in the rate controlling dislocation mechanism.

The current view of subgrain formation (Bird, Mukherjee and Dorn, 1969) is that during the initial period of rapid straining a rapid increase in dislocation density occurs to give a rough cellular distribution similar to that obtained in stage III deformation at lower temperatures. This

structure then undergoes rapid but gradual rearrangement (either by disintegration of the cell walls or by localised movements) so that a subgrain structure is evolving quite early on in the primary stage. The sub-boundaries become increasingly well defined and the subgrain diameter smaller (being typically between 2 and 25 microns) as deformation proceeds. After its initial increase, the dislocation density not associated with sub-boundaries (often considered to be a measure of the mobile dislocation density) decreases gradually so that the overall dislocation distribution becomes increasingly inhomogeneous as straining proceeds. Experimental evidence also indicates that in the steady state (secondary creep) the subgrain size sub-boundary structure and dislocation density within subgrains all reach constant values, though the structure is quite possibly dynamic in the sense that continuous rearrangement is occurring. Evidence concerning the misorientations across the subboundaries seems increasingly to indicate that they are usually less than 1° in magnitude, and slowly increase\_during stage II creep (Orlova et al, 1972). However, misorientation has frequently been considered to be another unchanging variable during secondary creep (Mukherjee et al, 1969). There appear to be no systematic variations of misorientation with either temperature or stress.
The subgrain size and dislocation density within subgrains are strong functions of stress, but at constant • stress, are insensitive to temperature. There is evidence to suggest that the same relationships hold whether the stress variation results from a variation in strain rate or from a wide variation in temperature, which implies that these substructural parameters are not directly related to the mode of dislocation motion, at least in pure metals (Staker and Holt, 1972).

The average subgrain diameter reached during secondary creep is found to be inversely proportional to the applied stress, and the empirical relation:

$$\frac{L}{b} = 20 \left( \frac{G}{\sigma_{app}} \right)$$
 (2-6)

where L is the subgrain size, b the burgers vector, and G the shear modulus, has been found to provide predictions of subgrain size within a factor of four of all experimental data. This equation probably overestimates the subgrain size since much of the data on which it is based was obtained by etch-pitting which generally gives higher values than does transmission electron microscopy.

Dislocation density within subgrains is given by:

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where n is determined to be between 1.5 and 2. Both equations (2-6) and (2-7) are described in detail by Bird et al (1969). Equation (2-7) corresponds quite well to the relation:

$$\sigma = \alpha G b \rho^{\frac{1}{2}}$$
 (2-8)

which is frequently made use of at lower temperatures and which arises principally from dimensional considerations. Equation (2-7) is also obeyed in non-subgrain forming materials where experimental studies indicate both increases and decreases in density may be possible during primary creep (Lagneborg, 1969a; Evans and Williams, 1972). Dislocation densities generally fall in the range  $10^6$  to  $10^{10}$  per.cm<sup>2</sup>.

In regard to the actual structure of the dislocation sub-boundaries, the most detailed work has been carried out in single crystals of molybdenum (Clauer et al, 1970) and copper (Gupta and Strutt, 1967; Hasegawa et al, 1970; Orlova and Cadek, 1970). Inevitably polycrystal studies are less detailed because of the more complex deformation and the uncertainty in crystallographic orientations in thin foils.

A common feature of the FCC single crystal observations is the formation of coarse slip bands parallel to the (111) primary slip plane in crystals oriented for glide on a single slip system. After extensive straining deforma-

tion bands of fine subgrains lying in the  $(\overline{1}01)$  plane (perpendicular to the primary slip direction) are frequently revealed by etch pitting (Gupta and Strutt, 1967) or thermal etching (Orlova and Cadek, 1970). Clauer et al reported fine subgrains within pronounced slip bands, and estimated that about half the total tensile creep strain in molybdenum resulted from slip on a highly stressed system within these coarse bands, a comparable amount resulting from fine slip on an alternative system. Both coarse and fine slip bands and deformation bands have been found after creep of both single and polycrystalline aluminium (Garofalo, 1965). There is no quantitative data on the partitioning of strain between coarse and fine slip, the extent to which the initial deformation modes persist during primary and secondary creep, or on the shape changes accompanying creep in single crystals. The formation of subgrains with characteristically "tidy" appearance at high temperature is often considered to result from easy non-planar slip (Challenger and Moteff, 1973). Thus, the process of subgrain construction is generally described without reference to slip lines and deformation bands, concentrating on homogeneous flow and fine slip.

Within the bands of fine subgrains, Gupta and Strutt (1967) found rough cellular arrangements of etch pits which coarsened with strain while developing into well-defined subgrains. These were equiaxed in some areas and elsewhere

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elongated along the primary slip direction. The subgrains within deformation bands remain finer than elsewhere. Hasegawa et al (1970) also found colonies of so-called "low angle" sub-boundaries of a few microns diameter, within "high angle" boundaries of several hundred microns separation, but the corresponding misorientations were not given. Etch pit studies in copper also indicate isolated long sub-boundaries tens of picrons apart encompassing smalf subgrains, and a similar duplex structure has been observed <sup>6</sup> in LiF single crystals (Reppich, 1971).

Studies of crept polycrystals have reported more © uniform equiaxed subgrains. The material used in creep studies is fairly coarse grained and will generally have a pronounced recrystallisation texture, so that similar slip modes to those in single crystals should operate in many grains. Non-uniformity of substructure is found from grain to grain or between grain interiors and regions near the boundaries. Barrett et al (1966) found bands of subgrains in some grains in etch pitted silicon steel and equiaxed subgrains in others which were finew adjacent to the grain boundary. In many cases such variations in substructure are not detected because insufficient foil area is surveyed. However, whilst Orlova et al (1971) detected elongated as well as equiaxed subgrains in iron, Karashima et al (1971), t a higher test temperature,

did not report such variations in their HVEM study.

There are relatively few detailed analyses of the types of dislocations making up the sub-boundaries or of the misorientations across them. Burgers vector determinations on dislocations within sub-boundaries and/or trace analyses have been carried out on FCC material by Orlova and Cadek (1970) and Hasegawa et al (1970) on copper, by Myshlyaev et al (1973) on aluminium, and by Challenger and Moteff (1973) on stainless steel. The most detailed work, however, is that of Clauer et al (1970) on molybdenum.

In the FCC systems, the sub-boundaries form by the intersection of dislocations belonging to the highly stressed slip systems on intersecting (111) planes, primary dislocation activity predominating. The most commonly observed boundaries are pure tilt walls lying in the (101) plane (normal to both the primary slip direction and the primary slip plane) which is also parallel to the deformation bands. In addition, predominantly twist boundaries are found lying in the (111) primary plane (Orlova<sup>2</sup> and Cadek, 1970).

Little information has been obtained on dislocations within subgrains. Those observers advocating a jogged-screw model for creep (Hasegawa et al, 1970) have reported evidence for jogs on dislocations within subgrains, but their visual identification is beyond the resolution of the electron

An added complication to the assessment of the role of substructure in creep is the possibility of sub-boundary migration and coalescence. Li (1966) has outlined the basic kinetics of coalescence, and there are isolated bits of evidence arising from studies of creep in pure aluminium which suggest that these processes may make a significant contribution to the total strain (Exell and Warrington (1972) suggested up to 25%). It is particularly difficult to establish experimental methods of separating out such contributions because of the essentially static nature of the observations (usually measurements of surface tilts resulting from subboundary movement) of what is clearly a dynamic process. However, the fact that subgrains are in many cases thought to remain more or less equiaxed during large strain increments suggests that the motion of dislocations associated with subboundaries, either individually or in groups, must be occurring, and correspondingly contribute to the strain.

#### 2-7. Transient Behaviour

The use of the word transient in this thesis is restricted to the history dependent effects observed after a specimen deforming in a steady state regime is subjected to a sudden change in stress or strain rate. Several such observations have been reported on crept specimens, usually involving a stress reduction, but few systematic studies have been made involving both increases and decreases in stress at high temperature (Sherby et al, 1956; Raymond and Dorn, 1964). The initial response to a change in stress or strain rate may reflect a complex superimposition of elastic and anelastic effects, as well as continued forward straining. After a short time, however, the strain rate or stress response becomes characteristic of the deformation process.

Class I and class II materials are found to give different strain transients following a reduction in stress during creep. Figure 2-3 shows the responses of pure aluminium (class II) and aluminium - 2.1 at. \* magnesium (class I). In class II materials there is an abrupt drop in creep rate to a value below that typical of the steady state at the lower stress followed by a region of increasing strain rate. In class I material the strain rate decreases during the transient towards the new steady state value.

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The conventional explanation of these observations is that the subgrains in the class II material provide a source of strength so that at the lower stress the creep rate is retarded until the substructure has coarsened to the scale appropriate to the new stress. Apparently the mobile density within subgrains either does not control the strain rate, or )1 the supply of such dislocations.





Metallographic observations of subgrain coarsening during the transient appears to support this kind of interpretation (Barrett et al, 1970).

Raymond and Dorn (1964) made a detailed study of partial unloading and reloading transients in pure aluminium. By measuring the initial creep rate after various values of load drops from a given steady state condition (that is from an ostensibly constant structure) they were able to show that the creep rate is a stronger function of stress at constant structure than under conditions where the substructure was allowed to equilibrate to the applied stress (Figure 2-4).

In a class I material, the transient observations imply that subgrains either do not form, or do not provide the resistance to flow found in class II materials. The creep rate is thus proportional to the dislocation density, which, after a stress drop, decreases during the strain transient, as does the strain rate.

In addition to stress change tests, strain rate change tests have occasionally been used to study high temperature deformation processes. Transient stress and strain rate responses have also been used to determine the level of internal stress during creep. Owing to the more detailed theoretical approach required in discussing these techniques, they will be considered further in Appendix A.

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Figure 2-4. Schematic diagram of the initial creep rates (points b-e) resulting from stress drops  $\Delta\sigma$  during secondary creep. The full line shows the stress dependence of the steady state creep fate. (After Bird, Mukherjee and Dorn, 1969).

# CHAPTER 3

#### CREEP THEORIES

#### 3-1. Introduction

There are two fundamentally different ways of looking at creep deformation caused by dislocation motion. The first view is that the strain rate is controlled by the mobility of gliding dislocations which in turn is sensitive to the distribution of local obstacles to flow. Thus, dislocations glide in a viscous manner and there is little or no resultant substructural hardening. The other considers the initial glide event to lead to some structural change, such as a pile-up or the refinement of a dislocation network. This change provokes a recovery response to off-set the structural hardening, which usually involves dislocation climb and annihilation. Generally, in this type of approach the creep rate is controlled by the rate of recovery.

Such is the diversity of possible mechanisms that, given these two views, it is possible to devise numerous models to describe creep deformation. However, because of the lack of clearly critical features, that is, those indigenous to the process rather than indirect consequences, discrimination between theories becomes very difficult. is compounded by the fact that many of the models were derived to describe only the steady state regime through a balance between either glide and climb or hardening and recovery.

As a.result, the basic mathematical relationship used for the purpose of comparison with experiment has been the stress dependence of the secondary creep rate. The strain rate is commonly written:

$$\dot{\varepsilon} = \rho b v$$
 (3-1)

where  $\rho$  is the mobile dislocation density, b is the burgers vector and v is the mean dislocation velocity. Since the dislocation density inevitably obeys a quadratic stress dependence, then any applied stress dependence greater than 2 must come from the dislocation velocity. In practice, it is theoretically very difficult to justify a velocitystress dependence much greater than unity. In addition, the range of exponents measured experimentally even for a single class of material suggests that this parameter has little fundamental significance.

There are three major models for the rate controlling mechanism governing dislocation activity during high temperature creep. Two of them, the dislocation climb model (Weertman, 1957a, 1968) and the jogged screw dislocation model

the our of class II

type materials. The other, the viscous drag model (Weertman, 1957b) is concerned with materials in which dislocations experience a strong solute drag and which is typically a class I alloy.

All three models describe a steady state dislocation motion, the former two predicting a value of 4.5, the latter a value of 3, for the stress exponent of the strain rate. While the viscous drag theory provides a plausible explanation of class I solid solution behaviour, the climb and jogged-screw models employ somewhat artificial descriptions of dislocation velocity and density respectively, to arrive at a sufficiently high stress exponent. In accordance with experimental results, all the models predict a temperature dependence of the creep rate primarily through the relevant diffusion coefficient.

None of the models make any assertions about either primary creep or sub-grain formation, or about the form of creep transients resulting from stress changes. The only substructural features predicted by the models, namely dislocation pile-ups (for the climb model) and jogged screw dislocations, have either not been observed or are at the limit of resolution of the electron microscope. On the other hand, the viscous glide model cannot be used to predict which alloys will show class I behaviour.

In the following sections, various more macroscopic approaches to the creep problem are outlined. Those models most pertinent to the data derived during this investigation are described in more detail in Chapter 6.

#### 3-2. The Basic Recovery Creep Model

Steady state creep is regarded as a situation in which strain hardening and thermal softening (recovery) are in balance. Orowan (1946) originally defined a coefficient of work hardening:

$$h = \left(\frac{\partial\sigma}{\partial\varepsilon}\right)_{r=0}$$
(3-2)

from the slope of a stress-strain curve in the absence of recovery, and a rate of recovery:

$$\mathbf{r} = -\left(\frac{\partial\sigma}{\partial t}\right)_{h=0} \tag{3-3}$$

As from an already established

from the decrease in yield stress with time when a work hardened specimen is annealed. More recently, h and r have been determined from the strain  $\Delta \varepsilon$  and the time  $\Delta t$ required to establish a new steady state after a small

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secondary creep (Figure 3-1). "h" can also be found from the same specimen deformed at room temperature to the same temperature-adjusted stress level (Mitra and McLean, 1967).

During the steady state the flow stress remains constant, hence:

$$d\sigma = (\frac{\partial\sigma}{\partial t}) dt + (\frac{\partial\sigma}{\partial\epsilon}) d\epsilon = 0$$
 (3-4)

i.e.,

The states

$$\dot{\epsilon}_{g} = \frac{r}{h}$$
(3-5)

(the Bailey-Orowan equation)

Experimental tests of equation (3-5) have confirmed this relation, but the values for h and r tend to be high. For example, Mitra and McLean obtained values for h up to 1.5 G for nickel and aluminium. The hardening measurement must have been performed close to the elastic-plastic region, where the machine effects are largest and the potential error greatest. Furthermore, the recovery rate is measured in the complicated transient region where the substructural changes cannot be expected to be the same as those resulting from simple recovery. The balance between hardening and recovery implies a dynamic balance between the accumulation and attrition of dislocations and an independent measure of r may be



Figure 3-1. Schematic illustration of the stress reduction and stress increment tests used to determine r and h.

unrelated to this interaction.

The Orowan theory makes no assertions about the stress and temperature dependencies of the strain rate, or of the substructural aspects of steady state creep. Numerous more detailed models have been proposed and these are discussed below, being crudely classified as Network Growth and Glide-Recovery models. Gittus (1971) has also formulated a non-structural recovery model which is described in Chapter 6. It should be noted that Weertman's climb model falls within the recovery framework, and that the viscous glide, and jogged-screw dislocation mechanisms do not preclude a balance between hardening and recovery.

# 3-3. Network Growth Models (McLean, 1966, 1968)

The substructure is assumed to be a three-dimensional dislocation network located within subgrains from which segments can be freed by thermal activation, move a certain distance and then be held up again, thereby hardening the material by refining the network. Recovery tends to reduce the total density by coarsening the network in a manner analogous to grain growth (the dislocations climbing or dragging jogs) so as to permit further release of gliding

in

balance.

The recovery rate can be calculated from the rate of increase of mesh size, using an analysis originally due to Friedel (1964). Based on the change in line energy of the network, the recovery rate is found to be proportional to  $\rho^{3/2}$  (and thus to  $\sigma^3_{app}$ ). McLean (1968) took the hardening yrate to be proportional to  $1/\sigma^2_{app}$ , a higher dependence than is commonly found at room temperature, and the model thus gives a power of 4-5 for the stress dependence of strain rate according to equation (3-5).

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Since the model is not mechanistic enough to permit the creep rate to be evaluated by equation (3-1), an indirect approach is made, by viewing the hardening and recovery processes as causing a net change in internal stress. This is reflected in the height of the energy barrier to be overcome by the dislocation, that is in the activation energy. The creep rate can be expressed as:

$$\dot{\varepsilon} = \dot{\varepsilon}_{0} \exp{-\frac{bA(h\varepsilon-rt)}{kT}}$$
 (3-6)

where h and r are now partial differentials of the internal stress and are functions of time during primary creep.  $\varepsilon_0$ , is the creep rate at t = 0, and A is the activation area. In principal, h and r can be expressed in terms of some

structural parameter (usually the dislocation density) the time dependence of which can also be estimated, and the equation solved to yield a strain-time relationship. An approach of this type, due to Lagneborg (1969b) is discussed in Chapter 6.

A major weakness of the network growth model, as with most other theories, is that it does not deal realistically with the mobile dislocation density, the entire density being used to derive the recovery and hardening terms. In practice only a small fraction of the links in the network will be potentially mobile at any given instant, and this fraction will change with time during primary creep, as the network changes scale.

Lagneborg (1973) has recently attempted to define this mobile density in terms of a distribution of link lengths in the network. The elastic interactions between dislocations at junctions in the network is considered to give rise to an athermal barrier expressed in terms of the stress on the dislocation required to overcome it  $\sigma_i$ , as:

$$\sigma_{i} = \frac{\alpha G b}{e}$$
 (3-7)

where G is the shear modulus, and L is the link length between junctions. Thus, only those link lengths over a

certain size can break free and become mobile. For example, if  $\sigma_{app}/G$  is  $10^{-3}$ , and taking  $\alpha = 1$ ,  $b = 2.5 \times 10^{-8}$  cm,  $l \simeq 0.2$  microns. If the network were imagined to be in the form of a three-dimensional square array, this would give a dislocation density of about  $10^{10}/\text{cm}^2$  which would be a lower bound, since most of the network would be finer than this. Equation (2-8) would predict a density of about the same magnitude at this stress, since it was derived from consideration of similar elastic interactions.

The problem is now one of describing the distribution of link lengths in the network at any time t, by considering the net effect of hardening (the release of links and their gliding to other parts of the network) and recovery (the coarsening of the network by both the shrinkage and expansion of the mesh). The creep rate can then be defined directly in terms of the rate of change of the distribution, from the rate of release of links in the network (the gliding of free links being assumed to be rapid and to provide the creep strain).

This approach is attractive since it obviously is a much more realistic view of mobile density and of the dynamics of the network. At present, it suffers from an inability to predict a true steady state, and from a degree of imprecision in the distribution function arising from " vais to describe the link

of

release rate. The mobile dislocation density obtained from this analysis is still large, being about 40% of the total in the steady state, but the model does provide a reasonable strain-time fit to experimental data.

A major weakness of the network growth theory is that the type of substructure envisaged does not resemble that observed in practice and in particular that the theory ignores subgrain formation. Generally, those dislocations located within subgrains lack the regularity of distribution implied by the model. To sustain the network view of the substructure, increasingly reference is made to materials which are non-subgrain forming (primarily ironchromium-nickel alloys) and consequently maintain a much more homogeneous dislocation distribution throughout creep, thus approximating to the network structure.

Not only are these materials not typical, but in regard to the key experimental substructural observation, that of the dependence of dislocation density on strain, contradictory results have been reported. Lagneborg (1969a) observed an increasing dislocation density during primary creep while Evans and Williams (1971) found a decreasing density (as is also found in subgrain forming material). Consequently, two recovery views of primary creep have been developed. Lagneborg (1969b) considered primary creep to reflect the initial dominance of the hardening process

causing a progressive refinement of the network and thus a gradual increase in recovery rate as the dislocation density increases. Evans and Williams (1971) on the other hand, asserted that the hardening rate, after an initial rapid increase, remains essentially constant while the recovery rate decreases as the network coarsens during the rest of the primary strain.

It could be argued that these obviously conflicting views are a genuine expression of divergent material behaviour (even though they relate to contradictory experimental observations of similar material). However, both approaches have enabled strain-time predictions of the same form of primary creep to be made and both have suggested that the generalised empirical creep equation (2-1) is an expression of their recovery model. Until the experimental situation is clarified, the idea of network growth does not provide a clear indication of either primary creep or the role of substructure.

#### 3-4. Glide Recovery Models

Cottrell and Aytekin (1950) approached the creep process from a cooperative view of glide and recovery

intimately connected, and subsequently many phenomenological models along these lines have been developed. It is assumed that the driving force for dislocation glide is the mean effective stress,  $\sigma_{eff}$ , defined as the difference between the applied stress and the mean internal stress,  $\sigma_{int}$ . The effective stress component is assumed to result from short range obstacles to dislocation motion, e.g., Peierls stress, impurity atoms and forest dislocations. Thermal agitation assists in moving dislocations past these obstacles. The internal stress component is assumed to result from the targles and cell walls, which cannot be surmounted by means of thermal fluctuations.

During primary creep the internal stress continually increases due to dislocation interactions and strain hardening. This diminishes the rate of dislocation glide since the mean effective stress is decreasing. On the other hand, the increase in internal stress will favour recovery, since the elastic interactions between dislocations is the, driving force for dislocation annihilation and rearrangement: According to this description, the steady state is achieved when the mean internal stress equilibrates at a value which allows the glide process to exactly compensate by strain hardening for the reduction of internal stress due

to recovery.

Gibbs (1971) has proposed a unified glide-recovery model to explain the breakdown of the strain rate-stress, power law, and the observation of a decline in the average internal stress/applied stress ratio with increasing strain rate. He considers the important glide event to be the motion of screw dislocations, jogs on which make a local obstacle type resistance to flow. At the same time, the long range internal stress develops as a consequence of glide and a climb controlled recovery counteracts this, as in conventional recovery models. Since the jog density increases as the strain rate increases, the local (thermally activatable) contribution to the flow stress increases, so that  $\sigma_{int}/\sigma_{app}$ declines.

The strain rate for this process resembles that for the jogged-screw model except that the mobile dislocation density is made subject to a mobilisation-annihilation condition, viz.:

$$\frac{d\rho}{dt} = A\rho v - B\rho^2 v \qquad (3-8)$$

with an increasing mobile dislocation density in primary creep. Analytical solution of this equation requires an assumption that v and the jog spacing are constant during nary creep (that is that the jog spacing equilibrates

very early on). A corresponding solution for the internal stress can be obtained from the knowledge of the immobilised dislocation density (using an appropriate recovery expression for the rate of annihilation of immobilised dislocations). These solutions yield a steady state creep rate expression similar to equation (2-5), provided that  $\sigma_{int} \simeq \sigma_{app}$ , and a decreasing  $\sigma_{int}/\sigma_{app}$  ratio with increasing strain rate.

There are two serious objections to this model. Firstly, it lacks a clear description of the changes in mobile density and/or velocity during primary creep, since the concept of an increasing  $\rho$  with a constant v cannot be rationalised with a decreasing creep rate. Secondly, the divergence of the strain rate from the power law stress relation at high stress is derived from an exponential effective stress dependence of the jogged-screw velocity. Nichols (1971) maintains that such an expression is incorrect, and that the jog velocity reaches a limiting value at high stresses, so that the deviation from the power law would be in the opposite direction. The model does, however, provide at least a qualitative explanation of why the exfective stress increases faster with increasing strain rate than does the internal stress. The departure from the power law towards an exponential stress relationship has usually been associated with an excess of vacancies dislocation intersection processes resulting

in enhanced climb rates (Sherby and Burke, 1967).

3-5. Mobility Models

Li (1968), Balasubramanian and Li (1970) have interpreted the stress dependence of high temperature creep in terms of the dislocation mobility implied by the stress dependence of the activation area. He has argued that the "structure theories" of creep, i.e., those which require variations in microstructure with stress to explain the observed stress dependence of creep rate, describe a fundamental rate mechanism which is considered linear with stress, the non-linearity arising from microstructural variations. However, the non-linearity is observed under conditions in which the stress or strain rate is suddenly changed, i.e., at constant microstructure. He has further argued that this non-linear stress dependence arises from the dislocation velocity, which is known to be non-linear at low temperatures.

Hence, for a metal deforming in steady state, Li's model predicts that an instantaneous increase in strain rate results in an acceleration of the mobile dislocations? Without changing the mobile dislocation density. The

is an

ase in effective stress

to a new value corresponding to the new steady state dislocation velocity.

In its simplest form, the theory thus assumes no structural changes, and an instantaneous flow stress response to a strain rate change. The internal stress thus remains constant as a proportionately greater fraction of the applied stress at the lower strain rate. The "mobility" theory is not mechanistic except in that it requires dislocation motion and a jogged-screw mechanism could provide the required activation area-stress relationship.

Although the activation rate analysis supports a non-linear velocity-strain rate, experimental evidence from measurements of the internal stress and transmission electron microscopy indicates that, at least for pure metals, a simple mobility argument is no more valid than a purely structural one. Transient responses to stress changes, the increase in internal stress with increasing strain rate, and the corresponding decrease in subgrain size and apparent mobile dislocation density all indicate some structural influence. Anything which increases (say) the mobile dislocation density will increase the rate of dislocation interaction, causing a gradual increase in internal stress through refinement of the substructure.

> Unfortunately, there is little concrete evidence on of either mobile dislocation density or dis-

location velocity on stress. The direct methods of measuring velocities, by observing individual dislocation movements, are extremely difficult to use at elevated temperatures due to the high dislocation mobility and densities operative and their consequently complex interaction. Likewise, mobile densities are not easily determined from total densities and are based on very subjective techniques, though there is some agreement that mobile density is a function of effective stress (Solomon and Nix, 1970).

A phenomenological view of creep which attempts to bring together the mobility and structural concepts has been proposed by Mecking and Lucke (1970). They assumed that dislocations move very fast, so that if in a small time,  $\delta t$ , a density  $\delta \rho$  becomes mobile and travels by a spurt-like motion a mean free path L, then in the same  $\delta t$  interval the dislocations are both originated and the strain  $\delta \epsilon$  is achieved. Thus:

$$\delta \varepsilon = b L \, \delta \rho \qquad (3-9)$$

and

$$\dot{\epsilon} = bL \dot{\rho}$$
 (3-10)

The velocity does not enter equation (3-9) and provided it is large enough, does not affect the strain rate, which

depends solely on  $\dot{\rho}$  (assuming L is independent of stress). If the mobilisation and immobilisation of dislocations in a small time interval is a function of the total mobile dislocation content, then equation (3-10) can also be used in situations of lower dislocation velocity such as steady state creep.

This model leads to some interesting qualitative predictions of the form of transients which result from a sudden change in strain rate. Assuming that immediately after the change the mobile dislocation density is unchanged, the initial response is a larger velocity and correspondingly a higher effective stress. Then, however, a higher production rate  $\dot{\rho}$  is established and consequently a higher mobile dislocation density. The Mecking and Lucke material thus shows transients like those in Figure (3-2), where the velocity and effective stress begin to decline from their instantaneous values, until after a time  $t_L$  the stationary values corresponding to the new strain rate are reached.

Mecking and Lucke did not point out that the precise shape of the applied stress-time curve (the one obtained from a strain rate change test) will, however, depend on the velocity-effective stress dependence. If the stress dependence is high, there will be a large change in effective stress for a given velocity (i.e., strain rate) change.

new ' - ' of effective stress may be greater than that



Figure 3-2. Schematic representation of the responses to a strain rate change.

corresponding to the steady state at that strain rate, the transients resembling those of Figure (3-2) with quite possibly a peak in the applied stress level similar to that in the effective stress curve.

If the effective stress dependence of the velocity is low, the jump in effective stress will be small after a strain rate change. Thus, the applied stress transient, due to a build up in internal stress, may follow the shape of the mobile density curve in Figure (3-2), rather than the effective stress curve.

### 3-6. Summary and Statement of Objectives

It is apparent from the foregoing sections that creep deformation is a complex process which cannot be adequately described solely in terms of the macroscopic variables. Apart from the problem of identifying a rate controlling mechanism and the relative importance of glide and recovery, the deformation has been shown to be subject to variations in the mobile dislocation density, dislocation mobility, and substructural arrangements.

The chief problem with current creep theories is that they were invariably derived from the consideration of a icular dislocation mechanism with a view to ex-

plaining the known dependence of the creep rate on the macroscopic variables, stress, activation energy, and temperature. In view of the complexity of the problem, it is not surprising that these theories were either assembled without reference to substructure and/or assumed that the creep rate was controlled either by dislocation mobility or mobile dislocation density. None of the theories can conform to or describe in sufficient detail the creep and substructural behaviours.

The precise role of sub-boundaries in creep is still not clearly understood. An extreme view might be that in the steady state they simply reflect the result of an equilibrium number of mobile dislocations of given burgers vectors moving at certain velocities and possessing a certain capability for local rearrangement by climb and cross slip. In this view, the rate controlling process would involve dislocation motion within sub-grains and the substructure would not be intimately connected with this process. An alternative view would be that the rate limiting process involves dislocations within or adjacent to sub-boundaries.

The development of sub-grains is undoubtedly closely related to the relative importance of dislocation mobility and mobile dislocation density, and the work

in

chapters is primarily an attempt

to relate variations in creep behaviour to differences in these parameters.

Since direct measurement of velocity and mobile density are impossible to make at high temperatures, it is recognized that information must be obtained indirectly from the study of both the basic creep behaviour and transient responses on the one hand, and the details of the dislocation arrangements on the other.

The experimental work has been carried out on a pure metal and on an alloy of the same metal which shows class I behaviour. It was anticipated that these materials would display the largest possible differences in high temperature deformation characteristics. An additional alloy has been studied which possesses characteristics of both class I and class II materials to provide indications of intermediate behaviour.

In each case, the shape of the strain-time creep curves and the dependence of strain rate on applied stress have been used to provide a basis for classification. In addition, high temperature tensile tests have been performed to compare the constant strain rate response with that at constant stress. To investigate the relative importance of dislocation mobility and structural changes during creep in the different materials, a study of stress rate change transients was made. Where possible,

a numerical comparison with an appropriate model has been used to provide additional support for the conclusions drawn.

A detailed study of the dislocation arrangements in each alloy resulting from stage II creep has also been attempted, using transmission electron microscopy and etch pitting. These observations have been related to the inferences drawn from the mechanical tests concerning the role of substructure. Particular attention has been paid to the presence of strain inhomogeneities and their effect (if any) on the substructure. The kinetics of sub-grain boundary motion have been modelled in an attempt to describe the creep process in a "structural" material.

If the transients and dislocation substructures are consistent in relation to, for example, mobility, then a more complete view of creep becomes possible. The differences in behaviour will still require models within which the functional dependencies of the critical parameters are either different or have to be artificially constrained in order to make the problem tractable, but the choice of such models would then be based on a clearer view of the creep process.

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## CHAPTER 4

### EXPERIMENTAL PROCEDURES

#### 4-1. Choice of Materials

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Pure copper was selected for study on the grounds that it is an f.c.c. metal with a moderate melting point, and that its creep behaviour, both mechanical and microstructural, is well documented as being typical of pure metals (Orlova and Cadek, 1970; Pahutova et al, 1971).

Several alloys of copper have been found to exhibit class II behaviour, notably copper-aluminum (Evans and Wilshire, 1970) and copper-nickel (Jones and Sellars, 1970). However, no copper alloy showing all the features of class I behaviour has been reported. Jones and Sellars (1970) found that copper-gold shows a low n value and inverted transients, but a normal primary creep.

Nickel-tin was found to have a low stress exponent with a correspondingly strong solute strengthening effect, but no information was given on the other features (Davies and Dennison, 1961). The atomic size difference in this system puts it well into the class I region according to the classification of Cannon and Sherby (1970) (see Figure 2-2). Since the size difference in copper-tin is similar to nickel-tin and the phase diagrams are similar at<sub>i</sub> corresponding fractions of the melting point of the solvent, it was decided to examine the alloy Cu-5 wt% Sn (which was available in this laboratory). At this concentration the solubility limit is exceeded at about 275°C (compared with about 625°C for Ni-5 wt% Sn). In both systems the second phase is reportedly extremely difficult to precipitate out (Hansen, 1958). Preliminary mechanical tests tended to confirm class I behaviour in the copper-tin alloy.

Included in the study was silicon steel (also commonly called silicon iron). The commercial grade used has a nominal silicon content of 3 1/4 wt%. This alloy has been the subject of a number of creep studies which, in general, indicated class II properties in regard to subgrain formation and stress exponents (Solomon and Nix, 1970; Barrett et al, 1966). The primary and transient responses appear to be variable, perhaps due to differences in material preparation (Barrett et al, 1970; Pahutova et al, 1972). In particular, Lytton et al (1965) found that heavily textured silicon steel showed almost no primary creep and no subgrain formation. According to the Cannon and Sherby classification, this alloy lies well within the class II region. It thus appears that silicon steel can be prepared so as to show characteristics of

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#### 4-2. Analysis and Specimen Preparation

#### 4-2-1. Pure Copper Polycrystals

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The OFHC copper (nominal purity 99.995 wt%) was donated by M and T Metal Co. Ltd. of Hamilton, in the form of 50 mm diameter rod. Table 4-1 shows the detailed analysis of this material. The rod was sectioned longitudinally, cold rolled 30% and machined into round threaded tensile specimens of dimensions shown in Figure 4-1(a). After chemically polishing to remove machine damage, the specimens were annealed in vacuum for 2 hours at 800°C and furnace cooled. The final grain size was about 0.2 mm.

#### 4-2-2. Copper Single Crystals

Three crystals were used for electron microscopy examination from a batch originally grown by Watt (1967), and the details concerning their preparation are given in his thesis. The specimens were cylindrical, with a gauge length 6 mm in diameter and 18 mm long, the head diameter being 12.5 mm. Prior to testing they were annealed in vacuum at 800°C, and chemically polished in a solution of equal parts of nitric, acetic and phosphoric acids. Laue back reflection X-ray photographs were then taken to deter-

## TABLE 4-1

## CHEMICAL ANALYSIS (WT%)

## a) Copper

Cu	99.95
Ni	0.006
Sn ·	0.01
Fe	0.015
Zn	0.001

# b) Copper-Tin

Cu	95.22
Ni	0.044
Sn	4.18
Fe	0.021
Zn	0.028

c) Silicon Steel

Fe	96.32
C	0.038
S	0.022
Si	3.33
Mn	0.041
MO	0.008
Cr	0.010
Ni	0.022



Figure 4-1. Test-piece dimensions.

mine their orientation, which proved to be that of Watt's series D10 as shown in Figure 4-2. The Schmid factors for the prominent slip systems in these crystals are shown in Table 4-2 using the notation of Steeds (1966),

#### 4-2-3. Copper-Tin

The nominally copper - 5 wt% tin was supplied in the form of cold rolled sheet 1.6 mm thick. The analysis is shown in Table 4-1. Flat tensile specimens of dimensions shown in Figure 4-1(b) were machined longitudinally from the sheet, polished mechanically to a 5 micron finish and annealed in vacuum for 2 hours at  $800^{\circ}$ C, and furnace cooled. The final grain size was about 0.1 mm and metallographic examination after etching in 50% nitric acid solution did not reveal any second phase or grain boundary segregation. The density of annealing twins was significantly greater than in pure copper, indicating a lowering of stacking fault energy as a result of alloying.

## 4-2-4. Silicon Steel

This was supplied by Dofasco in Hamilton in the form of "electrical steel" sheet 2 mm thick in the "hot band" hot ' is material is used in AISI-M grades





## TABLE 4-2

## NOTATION OF PROMINENT SLIP SYSTEMS

## IN THE COPPER SINGLE CRYSTALS

System

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Schmid Factor

Primary Slip Plane

(111) [Ī01]	0.402
(111) [0]1]	0.101
(111) [110]	0.299

## Conjugate Plane

(11) [011]	0.351
(11)[101]	0.111
(11)[10]	0.241

Critical Plane

(111)[101]	0.270
(Ī11) [110]	0.066

(111)[011] 0.198

## Cross Plane

(111)[101]	0.043
(111) [110]	0.004
(111) [011]	0.047

<u>A</u> 1

for transformer cores, and has a nominal chemistry of 3 1/4 wt% Si with low carbon manganese and sulphur. The actual analysis is given in Table 4-1. The steel is normally cold rolled in two stages to about 0.33 mm with intermediate annealing and then given a ginal anneal above  $1100^{\circ}$ C. The distribution of inclusions is controlled such that grain growth is retarded at lower temperatures, and the final anneal results in secondary recrystallization to produce a coarse grained product with a "cube on edge" ( (110) [001] orientation ) texture.

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Flat tensile specimens of the same dimensions as for copper-tin were cut longitudinally from the as-supplied sheet. An initial anneal confirmed that this material will not be recrystallised at 900°C. After an anneal at 1100°C in vacuum the initially "pancake" shaped grains were replaced by disc shaped grains, more or less equiaxed in the plane of the original sheet and with an aspect ratio of about 2:1. Metallographic examination after etching in a mixture of 2 parts hydrofluoric acid, 1 part nitric acid, 3 parts methanol and 4 parts glycol showed an average grain size in the plane of the sheet of 0.25 mm. Qualitatively, this structure is similar to that found by Lytton et al (1966) but they used a higher annealing temperature and got much coarser grains. Curved grain boundaries with located in them were observed in

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etched specimens indicating secondary recrystallization.

#### 4-3. Creep Testing Procedure

The creep tests were performed using a Satec CE model constant load creep tester, a lever operated dead weight machine equipped with a 2 kilowatt kanthal wound furnace. Furnace control was achieved by means of a zone control variac, a d.c. rheostat and a capacitrol controller and included an automatic preheat system, using a platinumrhodium thermocouple located in the furnace wall. The vertically mounted furnace was capable of adjustment so as to accurately locate the specimen in the hot zone.

Specimen temperature was monitored independently of the furnace controller by attaching three chromel-alumel thermocouples at the ends and middle of the specimen gauge length. The temperatures were read on a portable potentiometer or multi-channel chart recorder, depending on the length of the test, to an accuracy of  $1^{\circ}$ C. Under steady state conditions of temperature, the maximum tolerated temperature gradient was  $2^{\circ}$ C along the specimen gauge length, and from test to test the temperature was allowed to stabilise within  $\pm 2^{\circ}$ C of that desired. The rig could be lead thermally within a few hours of reaching test temperature.

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The strain was measured using a creep extensometer equipped with an LVDT capable of a total travel of either 2.5 mm or 6.3 mm, and a micrometer head permitting zero adjustment or suppression as desired. The extensometer head was capable of modification to allow attachment to either flat or round specimens. The LVDT output was monitored by a demodulator incorporating built-in range and zero adjustments and was fed to a single pen chart recorder displaying an extension-time curve. Several such recorders were used so that wide ranges of strain magnification and chart speed were obtainable. Full scale deflections were used corresponding to elongations between 0.125 and 1.5 mm, with a maximum measurable displacement of about 2.5 microns. Chart speed was varied between 1.50 mm per hour and 100 mm per minute.

The machine lever arm setting was factory precalibrated. A rough check of the load ratio was made by substituting an 18 kgm spring balance for the specimen. The specified load lever arm ratio was found to be true within the accuracy of the balance reading (that is, within 2% of the smallest load, 19 kgm, applied to any specimen).

The specimens were connected to the load train by means of rectangular stainless steel grips, those for round specimens bearing a threaded hole, those for flat

specimens having parallel plates. The grips were loosely threaded into the hot grip assembly of the machine.

The tests were done at  $550^{\circ}$ C (copper and copper-tin) and  $600^{\circ}$ C (silicon steel) corresponding to about 0.6 T<sub>m</sub> and 0.5 T<sub>m</sub> respectively. Specimen cross-sections were measured with a micrometer. The gauge lengths, before and after each test, were estimated from the parallel length between the specimen shoulders using a Bausch and Lomb optical comparator, all measurements being to within  $\pm 25$  microns. The extensometer head was attached to the specimen shoulders and the strains calculated from the parallel length.

The first series of experiments was performed to characterise each material in terms of the overall creep behaviour, i.e., the shape of the creep curve, and the stress dependence of the stationary creep rate. The range of applied stresses was chosen to give minimum creep rates ranging from roughly  $10^{-5}$ /min to  $10^{-1}$ /min for each material. The strain magnifications and chart speeds were also selected so as to provide comparable sensitivity in the measurement of the strain rates. For these tests, the load was applied using the motor driven weight car provided with the machine.

In order to establish the nature of the transient strain response of specimens subjected to a rapid change in stress, an additional series of experiments was carried

out in which a specimen was crept to stage II and then subjected to a rapid increase or decrease in load so as to increase or decrease the strain rate during the steady state by one or two orders of magnitude between  $10^{-2}$ /min and  $10^{-4}$ /min. In this case, the load adjustment was made by using the appropriate weight suspended above the weight pan from a moveable arm by means of a thick piece of rubber which extended about 15 centimeters elastically under the weight. The arm could be raised or lowered hydraulically allowing the extra load to be added or removed within a few seconds whilst minimising the impact, which can give rise to erroneous transient responses. Earlier attempts to perform rapid load changes using liquid additions were unsuccessful because the load change could not be made rapidly enough.

Both up and down load changes were done so that the effect of both a sudden increase and decrease in the effective stress on the specimen could be studied. The tests were designed to complement a series of strain-rate change tests performed on the Instron machine. Large load changes were employed in ofder to remove any ambiguity in the initial transient response to a load drop arising from the magnitude of the applied stress with respect to the initial internal stress. In these tests, the applied stress on partially unloading should be well below the internal

stress which would be found during a "dip" test (Appendix A).

#### 4-4. Tensile Testing Procedure

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The high temperature tensile tests were carried out using a table model Instron machine at constant crosshead speed. The range of speeds used was chosen to give the same range of strain rates in the steady state as in the constant load tests (assuming that all the crosshead movement is translated into plastic strain in the specimen). The chart drive system produced a load-time curve with a minimum load span of 4.5 kgms full scale and zero suppression as desired, and a 1 second span response of the pen with a range of chart speeds from 125 mm/min to 5 mm/ min.

The specimen (inserted in the same gripping unit used in the creep tests) was located inside a 3 zone kanthal wound split furnace. When desired, rapid cooling could be achieved by opening the furnace about a vertical hinge at the rear. The power supplied to each zone was independently adjustable by means of variable transformers, so as to maintain a constant temperature hot zone of sufficient length, and the neight of the furnace was adjustable to accommodate the different types of specimens. Three chromelalumel thermocouples were attached to the specimen at the middle and ends of the gauge length and monitored using a switch box and portable potentiometer. The power supply to the central zone of the furnace was regulated by means of an on-off controller using the centrally located thermocouple.

Inside the furnace the specimen was surrounded by a cylindrical Inconel tube which was open at the top end (so as to admit the thermocouples) and which protruded about five centimeters above the top of the furnace, and which was sealed to the bottom crosshead. This allowed tests to be carried out in a stream of dry deoxidised argon when desired.

The entire straining rig was surrounded by a wooden container to minimise temperature fluctuations and the load cell and lower crosshead were water-cooled. In addition, the main voltage supply was routed through a constant voltage transformer to minimise fluctuations and the water was supplied from a constant head device. During a test, once the system was stabilised after the preheat period, no measurable temperature variation was tolerated, though the temperature was allowed to stabilise within  $\pm 2^{\circ}$ C of the desired value, with a maximum gradient along the specimen

The thermal stability of the rig was monitored prior to starting the test by using the chart recorder to follow fluctuations in load about a small applied value. The system was considered stable enough to allow the test to begin when the maximum load fluctuation was + 200 grams or less, this representing less than 0.3 percent of the lowest yield load observed and is of the order of the accuracy of the load cell. Frequently, the fluctuation was below + 100 grams, although up to 24 hours might be needed to achieve stabilisation. At this stage, a typical load-time curve showed an approximately 50 gram fluctuation with a period of about 30 seconds superimposed on a more irregular variation of about 100 grams within 10 minutes. Neither of these fluctuations could be directly related to the on-off action of the controller which changed approximately every ten seconds.

The bulk of the experiments were carried out using a CT type load cell which has a maximum load capacity of 90 kgm. Some high strain rate tests were also done using a D type cell, maximum load 450 kgm. The CT cell, being used over a longer segment of its range, exhibited better stability characteristics than did the D cell, and this was particularly important in these relatively long time tests.

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same deflection for an equivalent fraction of their load ranges) so that in a region of rapidly changing load, a considerable fraction of the crosshead movement may be taken up in the machine. This must be taken into account when considering the actual strain rate being imposed on the specimen.

To illustrate these effects load-crosshead extension curves were calculated for the elastic straining of one of the round copper specimens at room temperature. In Figure 4-3 the results are shown for an infinitely hard machine and for the cases where the D and CT cells are used. The specimen modulus was taken to be  $1.32 \times 10^6$  kgm/cm<sup>2</sup>, and the deflections for the load cells and their couplings are those quoted by the manufacturer (which turned out to be slightly larger than the actual values).

The effect of the load cell on the plastic strain rate in the specimen is illustrated in Figure 4-4.

Initially, the basic load-time curve was obtained from each material at the same temperatures and over the same range of strain rates as were used in the creep tests. Subsequently, strain rate change tests were performed on specimens undergoing steady state deformation to provide a comparison with upward stress change data from creep

· · · · · · · strain rates.



Figure 4-3. Effect of machine stiffness on the elastic load extension curve for copper at room temperature.



The Instron is equipped with two gear boxes with a basic 10:1 ratio, and modifications were made to permit up to four decade reducers to be incorporated in both boxes, so that several orders of magnitude variations in crosshead speed could be accommodated in each gear system. Strain rate change tests could then be carried out at low strain rates simply by depressing a button to activate the second gear box. The response was very rapid, taking at most two seconds to establish the new crosshead speed (which is indicated by a new region of linear loading in a strain rate increase test). Due to the mechanical nature of the gear , system a small pip in load of up to  $\pm 100$  grams was occasionally observed at the moment of change, which was of one or two orders of magnitude in speed.

# 4-5. Procedures for Microstructural Examination

# 4-5-1. Copper Single Crystals

The three single crystals described in Section 4-2-2 were deformed at 550°C in the Instron under conditions identical to those for the polycrystals except that in this case split Hounsfield type grips were used to hold the specimens. They were extended 1, 11 and 39 percent total

strain at 2 x  $10^{-3}$ /min and rapidly cooled to retain the creep structures for examination. After surface examination, 1 mm slices were spark cut parallel to the (111) primary slip plane and either the  $(\overline{1}01)$  plane which is perpendicular to both the primary slip plane and primary slip direction, or the  $(1\overline{2}1)$  plane which is perpendicular to the primary slip plane and contains the primary slip direction. Spark machining damage was removed by chemical polishing and the orientation was checked by the back reflection Laue method. The Laue method was reproducible to about  $\pm 3^{\circ}$  and the slices were found to be within  $\pm 6^{\circ}$  of the required section. The slices were then chemically polished to about 70 microns using the nitric-acetic-phosphoric solution, and finally electropolished in a methanol-nitric acid solution at -25°C, using the "window" technique. The foils were examined in either the Siemens Elmiskop 1 or the Phillips EM 300 at 100 kV.

## 4-5-2. Copper Polycrystals

Specimens were strained in the creep machine to stage II at stresses giving strain rates of 2 x  $10^{-4}$ /min and 2 x  $10^{-2}$ /min and then rapidly cooled by raising the furnace above the specimen. After checking the elongation

then spark machined perpendicular to the tensile axis. The surfaces of the discs were ground flat on a 600 grade emery paper and one or two slices mechanically polished to a 1 micron finish and etched for a few seconds in a 50% nitric acid solution and examined under the optical microscope. Those destined for electron microscope examination were chemically polished to about 200 microns thickness, located in a standard PTFE holder and thinned to perforation in a double-jet gravity fed electro-polishing riq. The polishing solution was 25% phosphoric acid in distilled water, at a potential of about 20 yolts with a stainless steel cathode (in the form of hypodermic needles used to direct the jets). After washing in methanol the perforated discs were placed in the single tilt holder used in the Phillips microscope and examined without the use of supporting grids. The phosphoric acid solution did not produce as good a polish as did methyl-nitric, the foil frequently being slightly etched. It did, however, provide specimens which had much larger thin areas. For particularly detailed work, especially in dark field, the double tilt holder was used.

The information sought from these foils included the subgrain size, the dislocation density and subgrain misorientations, and the distribution of those features. From the optical microscopy, observations were made on

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the appearance of slip bands, the effect of deformation on the grain size and shape, and on the tendency to void formation.

A series of experiments were also performed to assess the stability of the substructure, and in particular its resistance to thermal rearrangement. Specimens were crept well into the steady state in the creep machine and then the load removed and the furnace left on for increasing time periods up to 200 hours. The resulting substructures were then examined in the above manner.

Where appropriate, dislocation densities were made using the method due to Keh (1963) in which a rectangular grid of lines with sets of regular but different spacings perpendicular to each other was made on a transparency. This was then superimposed on a print of the electron micrograph and the number of intersections along the two sets of grid lines  $N_1$  and  $N_2$  was measured. If the total length of lines in one direction is  $L_1$  and that in the normal direction is  $L_2$ , then the dislocation density is given . by:

$$\rho = \left(\frac{N_1}{L_1} + \frac{N_2}{L_2}\right) \frac{1}{\epsilon}$$
 (4-1)

where  $\varepsilon$  is the foil thickness.

In these experiments, foil thicknesses were not measured experimentally because in any given foil it was difficult to judge at the time of examination whether a dislocation density measurement would be meaningful, and foil thickness estimation on all the numerous areas looked at would have been extremely laborious. For reasons discussed in Appendix B, dislocation density measurement is at best an imprecise procedure, so that in the results quoted here, the relative measurements are the important feature. Thus, throughout the foil thickness has been taken as 2250 Å. This arbitrary value was chosen being mid-way between the likely range of foil thicknesses (1500 - 3000 Å) and representing for copper about 5 extinction distances in the (220) reflection and 8 extinction distances in the (200) and (111) reflections, the most frequently used. True foil thickness was unlikely to vary by more than one extinction distance either way in these foils.

The misorientation across a dislocation subboundary was measured from the shift in the Kikuchi lines of the selected area diffraction pattern. This is detected by comparing micrographs taken under SAD conditions from either side of the boundary.

The method used was to identify certain prominent Kikuchi lines on the diffraction patterns and locate an

arbitrary but convenient point adjacent to them. The procedure was repeated on the second pattern and the shift and direction measured between the two. A rotation  $\alpha$ about an axis in the micrograph causes a shift L $\alpha$  in the pattern where L is the effective diffraction camera length of the microscope (Hirsch et al, 1965). In the Phillips EM 300 at the diffraction settings used L is 71.7 cm so that the shift on the plate is 12.5 mm per degree, with an accuracy of about 0.1 degrees.

The subgrain size was measured by counting the number of subgrains per unit area  $n_A$ , determined from a montage of micrographs at a magnification of 11,000. According to Fullman (1953) the subgrain diameter is then given by:

$$d = \frac{1.38}{\sqrt{n_A}}$$
 (4-2)

Other authors quote slightly different values for the proportionality constant. Implicit in this approach is the assumption that the subgrains are of uniform size and are equiaxed.

#### 4-5-3. Copper-Tin

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Copper-tin specimens were deformed 3% (early stage II) in the creep machine at stresses corresponding to the same strain rates as for the copper specimens. The deformed material was reduced in thickness to 1 mm on 600 grade emery paper and then polished to a 1 micron diamond finish before being etched in 50% nitric acid solution for a few seconds and examined under the optical microscope.

An attempt was also made to prepare thin foils for transmission electron microscopy. This involved thinning mechanically to 0.3 mm on a 600 emery paper, care being taken to minimise damage (the chemical polishing solution used for copper was not effective on copper-tin). 3 mm discs were then punched out of the gauge length and jet polished in the same rig as for copper. A reasonable polish was obtained using the phosphoric acid solution, but the foils tended to perforate locally into tiny holes (perhaps originally present as voids formed during deformation), and only very small thin areas were obtained. An alternative approach was attempted, involving dishing the specimens in the jet polishing rig, and then transferring to a methanol-nitric acid bath and polishing to perforation at a very low temperature ( $\sim$  -50°C), using a copper cathode at 10 v. This gave a very good polish, but again the thin

areas were very small.

The small strains used for these specimens was made necessary because a preliminary check at 10% strain showed severe grain distortion and void formation in the optical microscope. A comparison will be made of optical examinations of the 3% and 10% strained specimens.

#### 4-5-4. Silicon Steel

It is well established that the substructure of silicon steel can be readily revealed by the etch pit technique, and that the correspondence between pits and the points of dislocation emergence is one to one (Picker-The substructural examination of this material ing, 1965). was therefore carried out by etch-pitting using a procedure originally due to Morris (1949). Silicon steel specimens were deformed well into the steady state at stresses giving stationary creep rates of 2 x  $10^{-4}$ /min and 2 x  $10^{-2}$ /min. They were then cooled rapidly to about 200°C and then allowed to cool more slowly from then on by lowering the furnace back down to just above the grips. This procedure is considered necessary to permit decoration of the dislocations by solute atoms (notably carbon) so that the material readily pits at the dislocation sites.

The deformed specimens were then reduced in thick-

ness to 1.25 mm on 600 grade emery paper and then diamond polished to 1 micron. They were then electropolished in a chromium trioxide-acetic acid-water solution 20 volts and at  $19^{\circ}$ C, using a stainless steel cathode. When a good polished surface was produced the voltage was reduced to 1 or 2 volts for one or two minutes during which time etch pitting occurred. The exact time and voltage for each new batch of solution had to be redetermined using a control specimen which had been pitted previously so that the substructural arrangements were known. Dislocation density measurements were made by counting the etch pits within a 25 cm<sup>2</sup> area at 5000 X. At this magnification the etch pits are about 2 mm in size and a density of  $10^{10}/cm^2$ would occupy 12% of the area.



## EXPERIMENTAL RESULTS - MECHANICAL TESTS

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#### 5-1. Introduction

In this Chapter the results of the mechanical experiments described in the previous chapter are presented. The results are given in the form of nominal stress and strain. Where comparisons are made, care has been taken to ensure that the data were obtained at the same nominal strains (and thus the same cross-sectional change). The maximum strain used in the determination of transient responses was of the order of 8%, so as to avoid any problems associated with either the onset of tertiary creep, or reaching the ultimate stress. At this strain level, the error between true and nominal strain is less than 4%. Some preliminary analysis of the mechanical test results are given in this chapter, while quantitative comparisons with a suitable model are given in Chapter 6.

#### 5-2. Creep Curves from Constant Load Tests

Figures 5-1, 5-2, and 5-3 show strain-time curves for



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regions of primary, secondary and tertiary creep leading eventually to fracture. There are, however, the desired major differences in the extent and shape of the primary curve and also in the extent of stage II.

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Copper shows the large primary strain and continually decreasing strain rate typical of pure metals and many class II alloys. The shape of the creep curve is qualitatively similar at all the stress levels examined, the primary strain increasing with stress from about 6 to 10%, between 1.5 and 4.5 kg/mm<sup>2</sup>, but remains essentially constant thereafter.

Copper-tin, on the other hand, shows an increasing strain rate throughout stage I at low stresses, but at high stress levels it shows a "normal" region of decreasing strain rate as is shown in Figure 5-2(b).

The silicon steel stage I behaviour is more complicated. Initially, there is a very short inverse strain rate region followed by a longer period of decreasing strain rate. The latter was difficult to monitor at low stresses involving very small rate changes, and was more apparent at high stresses (Figure 5-3(b)). Both copper-tin and silicon steel reach the steady state at much lower strains than does pure copper (generally less than 3%) and neither shows a large sudden elongation on applying the load.

The extent of stage II is of the order of 5% for copper, 8% in silicon steel, and 10% or more in copper-tin. In copper-tin, however, there is a very gradual increase in strain rate throughout, and a shorter stage III, fracture often occurring abruptly (i.e., within 1% strain of an apparent steady state). Stage III in the other two materials was longer in duration and usually involved larger strains.

The use of constant load rather than constant stress produces differences in the shape of the strain-time curve shown qualitatively in Figure 5-4. At low strains the increased hardening compensates for the loss of crosssectional area. At higher strains a mechanical instability develops and causes an inflexion in the strain-time curve, and eventually fracture, at shorter times than in a constant stress test.

It is apparent that in copper and copper-tin the modes of instability are different, and that the behaviour of silicon steel approximates to that of copper rather than copper-tin in this regard, resulting in noticeably different stage III creep.

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Figure 5-4. Schematic description of creep tests performed under constant load and stress conditions with identical initial conditions. (After Andrade, 1910).

#### 5-3. Stress Dependence of the Steady State Creep Rate

The steady state creep rates at various nominal applied stresses are shown in Table 5-1 and are plotted logarithmically in Figure 5-5.

In the case of copper-tin and silicon steel two measurements were made on each specimen at two different stress levels, apart from those specimens used to generate full creep curves. In these alloys the strain increment needed to reach the second steady state was small. For copper each measurement corresponds to a different specimen since the short stage II and long primary creep make it impossible to establish a new steady state unless the load change was made very early in stage II.

A linear relationship in Figure 5-5 implies a relationship of the form:

$$\hat{\epsilon}_{g} = \kappa \sigma_{app}^{h}$$
 (5-1)

The values for n for each material, together with 95% confidence intervals determined by a least squares linear regression based on the logarithms is shown in Table 5-2. Unfortunately, most investigators, when reporting exponents, do not indicate the statistical procedure followed, and such a linear

## TABLE 5-1

# STRESS DEPENDENCE OF THE STATIONARY CREEP RATE

a) Copper at 550°C	
Stress kg/mm <sup>2</sup>	Strain Rate/min
1.50	$2.25 \times 10^{-5}$
2.01	$5.24 \times 10^{-5}$
2.51	$1.29 \times 10^{-4}$
2.76	$2.45 \times 10^{-4}$
3.02	$4.84 \times 10^{-4}$
3.52	$1.53 \times 10^{-3}$
4.01	$3.33 \times 10^{-3}$
4.56	9.40 x $10^{-3}$
5.02	$1.37 \times 10^{-2}$
5.77	$3.94 \times 10^{-2}$

b) Cop	per-Tin at 550°C	
	Stress kg/mm <sup>2</sup>	Strain Rate/min
	1.78	$2.7 \times 10^{-5}$
	2.22	$.7.62 \times 10^{-5}$
	3.11	$3.7 \times 10^{-4}$
*	3.56	$6.75 \times 10^{-4}$
	4.00	$1.04 \times 10^{-3}$
	4.45	$1.5 \times 10^{-3}$
4	5.34	$2.85 \times 10^{-3}$
	6.23	$4.86 \times 10^{-3}$
•	7.12	7.83 x $10^{-3}$
•,	8.01	$1.29 \times 10^{-2}$
	8.90	$2.14 \times 10^{-2}$
	9.79	$2.60 \times 10^{-2}$

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## Table 5-1 (contd)

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<u>c)</u>	Silicon Steel at 600 <sup>0</sup> C	
	Stress kg/mm <sup>2</sup>	Strain Rate/min
	3.49	$2.63 \times 10^{-5}$
	3.99	$5.50 \times 10^{-5}$
	4.13	$8.00 \times 10^{-5}$
	4.49	$1.55 \times 10^{-4}$
	5.10	$2.97 \times 10^{-4}$
	5.27	$3.80 \times 10^{-4}$
	6.02	$6.50 \times 10^{-4}$
	6.95	$1.3 \times 10^{-3}$
	8.15	$3.15 \times 10^{-3}$
	9.07	$9.77 \times 10^{-3}$
	9.49	$1.37 \times 10^{-2}$
	10.11	$1.74 \times 10^{-2}$
	11.70	$8.2 \times 10^{-2}$

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Figure 5-5. Log-Log plot of secondary creep rate against applied stress.
# TABLE 5-2

LEAST SQUARES FIT OF THE DATA IN TABLE 5-1

TO THE POWER LAW  $\varepsilon_{s} = \kappa \sigma^{n}$  app

AND 95% CONFIDENCE LIMITS

 $n = 6.23 \pm 0.42$ 

(a) Copper at  $550^{\circ}C$   $n = 5.88 \pm 0.72$  n' = 6.82(b) Copper-Tin at  $550^{\circ}C$   $n = 3.97 \pm 0.18$  n' = 3.62(c) Silicon Steel at  $600^{\circ}C$  technique. A better result is obtained using a curve fitting procedure and the solutions may be very different. This is illustrated in the table where the n' values were determined from the same data using the modified Gauss-Newton least squares method (Hartley, 1961). A unique solution was not obtained from the silicon steel data, but the differences for copper-tin and copper are such that comparisons of these exponents with, those from other investigations can only be approximate.

To estimate the reproducibility of the strain rate measurement a series of the copper specimens was tested at a stress level of 2.9 kg/mm<sup>2</sup>. The average secondary creep rate was  $3.22 \times 10^{-4}$ /min with a 90% confidence interval for the mean of  $\pm 0.68 \times 10^{-4}$ /min.

## 5-4. Load-Time Curves From the Instron Tests

These were obtained at crosshead speeds of  $5 \times 10^{-3}$ ,  $5 \times 10^{-2}$  and  $5 \times 10^{-1}$  mm/min, and are displayed as stressstrain curves in Figure 5-6. In order to convert the loadtime charts to stress-strain, it is necessary to subtract the deflections of the load cell from the nominal strain values indicated by the crosshead speed. Since the load was relatively soft, then as mentioned in



Section 4-4, during the period of increasing load, the true strain rate of the specimen varied from a very low value up to the nominal rate. The latter is approached at a rate dependent on the rate of work hardening of the specimen and thus the nominal strains do not provide useful comparisons, particularly between different materials.

Initially, the values for cell deflection supplied by the manufacturers were used. However, the cell and grip systems combined was in fact slightly stiffer than those indicated, so that this procedure would have given rise to negative strains at low stresses. Therefore, the true cell deflections were measured by elastically straining a specimen of each material at room temperature under conditions otherwise identical to those employed in the high temperature tests, and subtracting out the elastic strain on the specimen. For this purpose modulus values for pure copper and pure iron were used.

The deflections found averaged 2.53 microns per kgm with a variation of  $\pm$  0.10, compared to the manufacturers' value of 2.80 for the cell and its coupling only, and of the order of 0.05 extra due to the gripping system. The resultant fit gives linear curves in Figure 5.6 over a reasonable load range, though there is a small non-linearity at very low stresses (too small to be apparent in the

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The slope of the linear region compares well with the modulus values at high temperatures, taken from Koster (1939).

Only the first few percent strain are shown in Figure 5-6 to clearly indicate the distinguishing features. As in the creep curves there are marked differences in tensile behaviour between the three materials. In copper, the load corresponding to a departure from linearity was insensitive to crosshead speed. At low speeds, a constant load condition was reached after about 3% strain following a\_region of gradual hardening. At high speeds, however, (that is above about  $5 \times 10^{-2}$  mm/min) this plateau was never reached and the specimen continued to harden at a diminishing rate until the onset of failure.

In copper-tin and silicon steel the onset of nonlinearity was markedly strain-rate sensitive and constant load was reached after 1-2% strain at all strain rates. In copper-tin at low and moderate speeds there was a peak in load soon after the onset of plastic deformation. This peak is not sudden as in yield points at lower temperatures and the extent of the load drop decreases with increasing strain rate, disappearing at the highest value. Correspondingly, the strain to the onset of constant load increases with strain rate.

In silicon steel, on the other hand, an apparent load instability occurs during the first percent or so of elongation, characterised by a short region of little or no work hardening preceding the increase in load up to the plateau. Stress drops were never observed in silicon steel but in contrast to copper-tin the instability increased with strain rate, being unobserved at low crosshead speeds. It is possible that this material is undergoing a yielding process which is partly masked by the softness of the machine. A considerable sudden extension of the specimen is needed to produce a rapid load drop in such a system because of the large deflections per unit load and the rapidly increasing strain rate in this region.

In copper-tin the plateau in load was in fact one of slowly decreasing stress due to loss of cross-sectional area. A comparison with the other two materials in this regard is not possible because in those the constant stress region is much shorter.

Pure copper and silicon steel had limited ductility with elongations to fracture as low as 10% in copper and 14% in silicon steel. Copper-tin always gave more than 18%. In nickel-tin Davies and Dennison (1961) reported that additions of tin did not affect the elongation to fracture in nickel, but the solvent metal in this case is relatively irable strain rates copper and silicon steel both give considerably greater strain to fracture in the creep machine than in the Instron, primarily because of the greater stage III elongations obtained under constant load conditions. Copper-tin gave about the same values in both types of test.

In the case of copper, it has been argued (Barrett and Sherby, 1964) that the low ductility is a result of oxide formation at grain boundaries resulting in enhanced decohesion rates during tertiary creep, and a copper single. crystal was pulled to 44% elongation at 550°C in the Instron without any sign of failure during preliminary experiments in this laboratory. However, no evidence of a second phase in grain boundaries was ever observed in thin foils examined in the electron microscope. The tests conducted in a stream of Argon in the Instron machine, preserved a clean surface on the deformed copper, but did not improve the elongation to fracture, nor did it have any apparent effect on the creep curve or stationary creep Barrett and Sherby (1964) found that in copper the rate. ductility was only enhanced in a vacuum better than  $10^{-4}$ atmospheres. This particular figure does not appear to have theoretical significance relative to the idea of oxide formation since the theoretical decomposition partial pressure of oxygen for the reaction 4 Cu +  $0_2 = 2Cu_20$ -12 's temperature. at ·

## 5-5. Stress-Strain Rate Dependencies in the Constant Stress Region of the Instron Curves

The stresses reached in the Instron in the plateau region at various strain rates are shown in Figure 5-7 on a log-log plot for comparison with the stage II creep data. (Only the data points for the Instron tests are shown.) In the case of copper, steady states were not achieved above strain rates of about  $10^{-3}$ /min. At the higher speeds, therefore, the values shown in the figure refer to the stress at which the stress is decreasing at less than 0.015  $kg/mm^2$  per minute. This is an arbitrary criterion and it is recognised that it corresponds to a range of work hardening rates depending on the stress level (low work hardening rates at higher strain rates). The alternative would be to take measurements at the same $^{\sigma}$ hardening rate, but it was impossible to accurately determine the extremely small rates of loading at low strain rates.

The result of this procedure should be to give stresses which lie below the peak Instron values (which were not very reproducible) and the stage II creep values, by an amount which decreases with increasing strain rate. This is offset comewhat by the increase in strain to maximum load as the strain rate increases (which would tend to increase the error at high strain rates).





A rough estimate of the maximum error (neglecting machine effects) can be made as follows. At  $\epsilon = 5 \times 10^{-3}$  /min the rate of loading at the measured value gives a work hardening rate of 0.3 kg/mm<sup>2</sup> per percent elongation. The strain to maximum load is about 5% at most greater than that at the point of measurement, so that if the mean hardening rate is half that over the remainder of the stress-strain curve, the error will be about 0.75 kg/mm<sup>2</sup>. From Figure 5-7 it can be seen that the maximum difference between Instron and creep values is in fact about 0.70 kg/mm<sup>2</sup>, but that there is no consistent variation with strain rate, except that all the values above 2  $\times 10^{-4}$ /min are on the low side of the creep results. In general, the agreement between the two test methods is good and follows roughly the same stress-strain rate dependence.

## 5-6. Load Change Test Results

Figure 5-8 shows strain-time curves from specimens subjected to a sudden change in stress, corresponding to approximately a two-orders-of-magnitude change in the steady state strain rate. The strains are all referred to the point of load change, which is taken to correspond to a. Note the widely varying strain and





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time scales on the various figures.

Each test was repeated at least once under identical conditions. The reproducibility is quite good, though quantitative data is sometimes difficult to extract, since the order of magnitude of the measurement is in some cases not much more than the experimental accuracy. The tests were also repeated under rapid loading conditions, the load being changed directly by hand, and the shape of the upward transients could be dramatically altered. In copper and copper-tin this effect could reverse the type of transient observed, suggesting that qualitatively at least the results shown are correct.

#### 5-7. Strain Rate Change Test Results

Stress-strain curves showing the load response to increases of strain rate of varying magnitude from a common value are shown in Figure 5-9. The basic rate was  $2 \times 10^{-4}$  /min with increments of 10, 50 and 100-fold, from the constant load region. The crosshead travel has in each case been adjusted to allow for load cell deflection using the same procedure as described in Section 5-4. As with the Instron loading curves, the copper-tin and silicon steel are

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that is, the transients involve either low strains or short times. In copper, the initial rapid jump in load is very small and new steady states are, never achieved. A few additional tests were done on a small sample of decarburised iron (carbon 40 ppm by weight) and this also behaved like pure copper, though it did eventually reach new steady states.

The load instability noted in the loading curves was still apparent in silicon steel, but not in coppertin.

## 5-8. Discussion

## 5-8-1. Copper

The strain-time curve for copper is typical of pure metals and some class II alloys. The stress-strain curves show that the yield stress is not very sensitive to strain rate. This implies a high stress dependence of the dislocation velocity, so that large changes in velocity produce only small changes in stress. Johnston (1962) has shown that such a dependence, combined with a high work hardening rate and initial dislocation density, will prevent the development of yield points which otherwise result from the inter-

of ..... imen and testing machine.

The unloading transient for copper is similar to that of pure aluminium described in Section 2-7. An upward change in stress brings about a transient resembling a primary creep curve (this is also true for the other two materials). The strain rate change test in copper shows essentially the same features as the basic Instron curves, notably a small rapid stress response and a high work hardening rate. In the analysis of Mecking and Lucke described in Section 3-5 this corresponds to a high stress dependence of the dislocation velocity, or a high velocity.

In regard to the stress dependence of the steady state creep rate, the exponent n confirms the class II designation for copper. In Figure 5-10 the results for copper and silicon steel are compared with published data from constant stress tests. Allowing for an expected underestimation of the stress values for the constant load data, the agreement is  $\rho$ good, particularly in regard to the slopes. There is no published data on copper-tin at this temperature.

#### 5-8-2. Copper-Tin

The strain-time curves for copper-tin suggest class I behaviour with a particularly strongly inverted curve at low stresses. Inverted primary creep and the changeover at ha in a few instances, notably

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Figure 5-10. Log-Log plots of secondary creep rate as a function of stress - comparisons with published work.

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on Al-Mg (Ahlquist and Nix, 1971) and Fe-Mo (Fuchs and Ilschner, 1969). The peaks and discontinuities in the stress-strain curves from the Instron are in good qualitative agreement with the creep curves, as are the observations of relative ease of achieving steady state. Where the approach to stage II in the constant load tests involves a decreasing strain rate, in the Instron tests the load plateau is approached from a region of increasing load and vice versa. This suggests that the deformation modes are compatible and that the observed discontinuities are not machine effects.

The yield points in copper-tin closely resemble those observed by Horiuchi et al (1965) in Al-Mg and Al-Cu at temperatures and strain rates above  $300^{\circ}$ C and 2.5 x  $10^{-2}$ / min, respectively. As in Figure 5-6(b) yielding occurs slowly, involves strains of the order of two percent, which increases with strain rate, and there is no work hardening after the lower yield stress is reached. The height of the yield drop increases with strain rate at high temperatures, but near the critical temperature (which increases with increasing strain rate) the height of the drop decreases, and disappears as the strain rate increases. At 2.5 x  $10^{-2}$ /min, for example, the yield drop disappears at 0.6 T<sub>m</sub> in Al-Mg (compare Figure 5-6(b) which is also at 0.6  $T_m$ ). This correlation with homologous temperature sugrance of the yield point is due to a at the '

\_ limiting diffusion controlled process.

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The most obvious explanation is that at low strain rates, where solute atmosphere drag is the rate controlling process, the yield point results from the low velocity dependence of the flow stress. Likewise, the relative infrequency of dislocation interaction prevents work hardening. At high strain rates the solute atmospheres cannot diffus¢ fast enough, and dislocations escape them. As a result, the velocity dependence of the flow stress is increased and the yield point disappears. There must be some frictional drag on the dislocations even at high strain rates, because the stress dependence of the strain rate is not greatly changed, there is no evidence of inhomogeneous flow, and the yield stress is still fairly rate sensitive.

The unloading transients for copper-tin are similar to those identified in Section 2-7 for a class I material. In the strain rate change tests the small yield point at  $2 \times 10^{-3}$ /min does not reappear because the increase in dislocation density is smaller after the strain rate change than after loading directly at the higher rate. The low stress exponent of the strain rate in copper-tin implies a viscous-glide controlling mechanism.

The most commonly used model for the solute-dislocation interaction is due to Cottrell and Jaswon (1949) involving

At temperatures and/

or low strain rates the atmospheres can diffuse fast enough to prevent breakaway. At higher strain rates and effective stresses the solute atmosphere becomes progressively more dilute and the drag force decreases. This is the situation causing the disappearance in the yield point and the apparent increase in velocity-stress exponent at high strain rates. The inhomogeneous flow and sharp stress drops observed during tensile tests at lower temperatures (i.e., at about 130°C in copper-tin) are no longer observed.

Regardless of the precise mechanism, the solutedislocation interaction leads to a linear velocity-stress relationship for atmosphere dragging. In Weertman's viscous glide model, the steady state strain rate is given by:

$$\dot{\varepsilon}_{s} \simeq \frac{2(1-\nu)}{AG^2} \sigma_{app}^3$$
 (5-2)

where A is the constant for the model interaction. For the Cottrell-Jaswon model:

$$A \propto \frac{e^2 b^5 c G^2}{k T \bar{D}}$$
(5-3)

where  $\overline{D}$  is the solute diffusion coefficient, e is the solute-solvent size difference, and c is the solute concentration. In principal, A can be computed and compared with the value determined from the stress dependence of the steady

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Instead, an estimate of the range of velocities can be derived. Experimentally the constant in the strain rate applied stress equation was found to be 7 x  $10^{-6}$  (kgm/mm<sup>2</sup>)<sup>-3</sup> %min)<sup>-1</sup>. From equation (5-2), taking G = 3850 kgm/mm<sup>2</sup> and v = 0.3, A is found to be 0.0365 kg/mm<sup>2</sup>/min and thus taking  $V = \sigma b/A$ , the range of velocities becomes of the order of  $10^{-8}$  to  $10^{-7}$  cm/sec.

## 5-8-3. Silicon Steel

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The primary creep curves of silicon steel are difficult to characterise. They are similar to those of Pahutova et al (1972), but other authors have reported normal primary creep (Barrett, Nix and Sherby, 1966; Solomon and Nix, 1970). The sigmoidal type curve arises from simultaneous changes in dislocation density and velocity. Haasen and his co-workers have described the sigmoidal curves of silicon and germanium in terms of a decreasing mobile density. This is shown schematically in Figure 5-11, where the lower curve is derived by graphical integration.

In the linear portion of the transient, the increase in density is compensated by a decrease in effective stress and velocity. The simplest prediction of the model is that the ratio  $\varepsilon_m t_m/\varepsilon_m$ , where  $\omega$  refers to the point of inflexion,

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Figure 5-3 the values are: a)  $l_{x}^{i}$  31, and b) 1.38. Implicit in Haasen's approach is an increasing internal stress with strain. In the case of silicon steel, the sigmoidal curves are found at all stress levels, so that the creep mechanism is quite different from that of copper-tin. The model does not invoke a particular dislocation mechanism or solute interaction.

The yield effect in silicon steel, like that in copper-tin, is gradual, involves quite large strains, and suggests a small stress dependence of the dislocation velocity. However, there are important differences, notably that in silicon steel the yield does not appear at low strain rate, increases with strain rate, even at the lower homologous temperature, and does not involve an actual stress drop. In addition, it is followed by a period of work hardening.

One possible source of this kind of yield behaviour is a low initial density, which has been shown by Johnson (1962) to have a greater effect on the appearance of the yield point than does the velocity stress dependence. Such low densities have been reported by Lytton (1965). In this case, an increasing yield effect with increasing strain rate would be expected, and a higher velocity and velocitystress dependence would tend to suppress the stress drops,

to the .... of work hardening. Note that in

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marked as in copper-tin. However, after a strain rate change the yield point is reproduced at all strain rates and has essentially the same appearance as in the stress-strain curves. To produce a yield after a small strain rate change, the rate of dislocation multiplication must be greater than in copper-tin.

For silicon steel, it has been suggested (Pahutova et al, 1972) that the mechanism giving rise to the sigmoidal curves involves a short range order interaction. An alloy system known to exhibit similar creep curves is partially ordered Fe-Al, which has a similar phase diagram. The effect of order on the high temperature deformation of Fe-Al solutions has been studied by Lawley et al (1960) and Schmatz and Bush (1968). The former concluded that the diffusion of atoms to maintain the equilibrium ordered structure determined the creep behaviour, which is glide controlled. The inverse transient was explained by a stress directed short range order superimposed on the isotropic order. This provides relatively "easy" paths for dislocation movement and an increasing creep rate with strain. Schmatz and Bush observed very low initial dislocation densities (due to growth of ordered domains during annealing). They concluded that during initial stressing, the rapid refinement of the domains occurred, and the resulting rapid i / i id a Johnson type yield,

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typified by an S-shaped curve, without reference to a glide controlled mechanism.

The degree of order in 34% silicon steel will not approach that in the iron-aluminium alloy, so that it is tempting to conclude that extensive secondary recrystallisation has reduced the initial dislocation density. However, the strain rate change curves tend to refute this argument. The high n value, the inverted transient after a stress drop and the extensive "normal" primary creep region imply some form of substructural activity which is absent in copper-tin, so that overall the silicon steel behaviour is intermediate between the other two materials.

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#### CHAPTER 6

#### DETAILED ANALYSIS OF STRAIN TRANSIENTS

## 6-1. Introduction

In this Chapter a model is described, the purpose of which is to provide a quantitative description of the strain-time transients incorporating variable material behaviour in terms of mobile dislocation density and dislocation velocity. Initially, however, a brief description is given of some earlier models based on the hardeningrecovery creep concept. It would not be practical here to give a full description of these models; rather, only the underlying assumptions and some indications of the mathematical approach have been given. In the previous Chapter, a wide variety of creep behaviour has been exposed and some qualitative explanations proposed. The model outlined in Section 6-3 seeks to provide a unified view whereby such effects as solute-atmosphere dragging and short range order can be incorporated in the form of differing dislocation velocities and velocity-stress dependencies without loss of generality in the description.

## 6-2. Strain-Time Relationships Derived from Recovery Models

Lagneborg (1969b) used a refined recovery-creep model to obtain expressions for the strain-time relationship during primary and secondary creep. The h and r terms in equation (3-6) were expressed in terms of the increase and decrease in internal stress due to strain hardening and recovery, viz.:

$$\dot{\varepsilon}(t) = \dot{\varepsilon}_{0} \exp - \left\{ \frac{Ab(\sigma(t)_{h} - \sigma(t)_{r})}{kT} \right\}$$
(6-1)

where  $\sigma(t)_{h} - \sigma(t)_{r}$  represents the activation barrier for further straining. To describe the hardening term he used equation (2-8) and a linear dependence of strain on the total dislocation density and developed an expression of the form:

$$\frac{d\sigma(t)}{dt} \propto \rho(t)^{-\frac{1}{2}} \dot{\epsilon}(t) \qquad (6-2)$$

The corresponding expression for  $d\sigma(t)_r/dt$  was obtained by assuming a network growth model with a  $\rho(t)^{3/2}$ dependence of the rate of recovery. In addition, the recovery process defines the quantity of dislocations lost and thus the net density at any instant. The assumed linear dependence of strain and net density leads to an

(C-1) for the ' in rate.

The model thus results in a system of four equations which can be manipulated to provide computed comparisons with experimental results for the time dependence of both  $\rho(t)$  and  $\dot{\epsilon}(t)$ . For a 20% Cr - 35% Ni steel Lagneborg was able to derive a reasonable "normal" shaped primary creep curve, although the model also predicts a continually increasing dislocation density which is in contradiction to most experimental observations (see Section 3-3).

Apart from the fact that the model invokes the network growth concept, there are other problems associated with its use to describe other materials. The equations cannot be solved analytically and contain parameters such as M, the dislocation mobility, and  $\rho_{o}$  and  $\dot{\epsilon}_{o}$ , the dislocation density and strain rate produced on loading. These cannot easily be measured, so that further assump-  $\Im$ tions become necessary regarding (for example) their stress dependence. Correspondingly, it is difficult to use this approach to describe strain transients. A similar model due to Barrett et al (1970) describes transients in silicon steel (which had a well-defined pub-boundary structure). Using the same type of recovery term, they were able to compute suitable curves while at the same time clearly indicating that extensive substructural change occurred during the transients. A question thus arises as to whether the qualitative agreement between theory and experiment in

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these models is fortuitous, arising from essentially dimensional considerations rather than from a genuine view of structure. It may thus be possible to devise simpler models without reference to a particular dislocation network or to separate hardening and recovery terms.

Gittus (1971) expressed the balance between hardening and recovery as a balance between strain induced immobilisation of dislocations and dislocation annihilation resulting from thermal recovery, thus:

$$d\rho = \left(\frac{\partial \rho}{\partial \varepsilon}\right) d\varepsilon \div \left(\frac{\partial \rho}{\partial t}\right) dt$$
 (6-3)

He chose the following expressions for these terms:

$$\frac{\partial \rho}{\partial \varepsilon} = -\lambda \left(\rho - \rho_{\infty}\right) \frac{\varepsilon_{s}}{\varepsilon}$$
(6-4)

and

$$\frac{\partial \rho}{\partial t} = \gamma \left( \rho_0 - \rho \right) \tag{6-5}$$

where  $\lambda$  and  $\gamma$  are constants,  $\rho = \rho_0$  when t = 0, and  $\rho \neq \rho_{\infty}$ as  $t \Rightarrow \infty$  when  $\gamma = 0$ . By substituting these equations into equation (6-3) and differentiating, an expression can be generated which can be solved analytically for the time dependence of the mobile dislocation density. If the dislocation velocity is assumed constant, then equation (3-1) can be integrated to yield the desired strain-time relationship:

$$\varepsilon(t) = \left\{ \frac{\dot{\varepsilon}_{s} (\rho_{i} - \rho_{L})}{(\lambda \dot{\varepsilon}_{s} + \gamma) \rho_{L}} \right\} (1 - \exp - ([\lambda \dot{\varepsilon}_{s} + \gamma]t)) + \dot{\varepsilon}_{s}t \quad (6-6)$$

where  $\rho_{i}$  and  $\rho_{r}$  are the initial and steady state values. Note that this equation has the same form as the empirical relationship (2-1). It predicts a linear relationship ` between the empirical coefficient a and the steady state creep rate, a correlation which was verified by Gittus for stainless steel. In principal, equation (6-6) can be solved to give the increments of creep strain contributed during each of a succession of time periods at a succession of stress levels. In practice, this equation cannot be used for stress increments since it makes no allowance for the sudden rapid increase in dislocation density at the start of the transient, it considers only the decay in density from an initial peak value. It thus cannot, as it stands, be applied to a material such as copper where the dislocation density within sub-grains is an increasing function of stress.

#### 6-3. Derivation of an Alternative Strain-Time Relationship

The following description is based on the concept that the time needed for a dislocation to traverse a given area of slip plane will be dependent on both the time of flight  $t_f$  between obstacles and the waiting time  $t_w$  spent in the vicinity of the obstacles. The waiting time is that needed for the dislocation to receive a thermal fluctuation of sufficient magnitude to overcome the obstacle and is thus a function of the attempt frequency and the activation energy. The time of flight will be a function of the mean velocity of a gliding dislocation and of the mean spacing of the obstacles (see Lloyd et al, 1970).

Sargent (1973) has suggested an approach by means of which this concept could be used to define the time dependence of the mobile dislocation density, this being an essential requirement for any model based, like this one, on the integration of equation (2-1).

The net change dN in mobile dislocation density (i.e., in those actually moving) during an interval dt is the difference between the density becoming stuck, N  $(dt/t_f)$ , and that managing to free itself (where dt is small relative to  $t_f$ ). The latter quantity is  $dt/t_w$  times the density of dislocations held up. At this juncture, the concept of a density N<sub>o</sub> of "potentially mobile dislocations" is introduced. This is simply the sum of those held up and those actually moving. To clearly define this quantity it would be necessary to provide a mechanistic description of the distribution of dislocations and/or of the obstacles to dis-

For example, the approach of Lagneborg (1973) could be used to define  $N_0$ . In this case it is assumed that there exist both thermal and athermal barriers to dislocation motion. The potentially mobile density (what he calls the mobile density) is comprised of all those for which the athermal obstacle can be overcome by the applied stress. This means that it includes all those dislocations situated in positions where the structure is locally coarser than some critical spacing. In a non-substructure forming material  $N_0$  might thus be expected to be a much larger fraction of the total density than in a subgrain former.

For the purposes of this analysis it is desirable to maintain generality so as to be able to apply the model to materials of strongly differing behaviour within which  $N_0$  would vary considerably relative to the total number of dislocations. It is assumed that  $N_0$  is constant at constant stress. Effectively, this means that any net increase in total dislocation density during primary creep or during a transient can be thought of as becoming permanently held up at athermal barriers. It is difficult to estimate the degree to which this assumption is justified because the corresponding experimental evidence does not clearly indicate the net effect on the distribution of athermal barriers.

The net change in mobile density in time dt is thus:

$$dN = (N_0 - N) \frac{dt}{t_w} - N \frac{dt}{t_f}$$
(6-7)

where the first term describes the number mobilised, the second the number immobilised.

' an instantaneous density N:

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$$\frac{dN}{dt} = \frac{N_o}{t_w} - N \left(\frac{1}{t_w} \div \frac{1}{t_f}\right)$$
(6-8)

Integration of this expression defines the mobile density as a function of time, assuming  $t_w$  and  $t_f$  are constant:

$$N = \frac{1}{a} \left( \frac{N_{o}}{t_{w}} - \exp((c-at)) \right)$$
 (6-9)

where  $N = N^{O}$  when t = o, and:

$$a = (\frac{1}{t_w} + \frac{1}{t_f}), c = \ln (\frac{N_o}{t_w} - aN^o)$$

the creep rate is given by:

$$\dot{\epsilon} = \frac{\alpha b V}{a} \left[ \frac{N_o}{t_w} - \exp(c - at) \right],$$
 (6-10)

where V is the average dislocation velocity, which leads to:

$$\varepsilon = \frac{\alpha b V}{a} \left[ \frac{N_o t}{t_w} - \frac{1}{a} \left( \frac{N_o}{t_w} - a N^o \right) (1 - e^{-at}) \right]$$
(6-11)

Note that this equation is of the same form as equation (2-1) and provides a more analytical basis for the coefficients than does that of Gittus.

The shape of a primary creep curve depends critically on the relative magnitudes of  $t_w$  and  $t_f$ . At low stresses, if the time of flight between obstacles is long a  $\approx 1/t_w$  and:

$$\varepsilon = \alpha b V t_{W} \left( \frac{N_{O} t}{M_{O}} - (N_{O} - N^{O}) (1 - \exp^{-t/t_{W}}) \right)$$
 (6-12)

If  $N_0 > N^0$  (as in an annealed material) the creep curve is of the inverse type, the steady state being defined by  $\dot{\epsilon} = \alpha bV N_0$  which means that all the potentially mobile dislocations are mobile. In addition, the transient strain:

$$\varepsilon_{t} = \dot{\varepsilon}_{s} \frac{(N^{O} - N_{O})}{aN_{O}}$$
(6-13)

which is identical to the representation of Gittus.

The term  $t_f$  decreases with stress at a greater rate than  $t_w$ , and in the limit a  $\approx 1/t_f$ , and:

$$\varepsilon = \alpha bVt_f \left( \frac{N_o t}{t_w} - t_f \left( \frac{N_o}{t_w} - \frac{N^o}{t_f} \right) \left( 1 - \exp^{-t/t_f} \right) \right) \quad (6-14)$$

The shape of the primary creep curve now depends on  $(N_0/t_w - N^0/t_f)$ , and for a series of tests, as the stress increases, the curve can change from "inverted" to "normal" in shape. The latter case is extreme, since it implies "barrier"  $\cdot$  control, but this is not a requirement for normal curvature. Note that the approximation of constant velocity would be invalid if a change in mechanism were to occur during a test, and the resulting discontinuities in mobile density and velocity would lead to more complex curves. For copper the identity "at" was calculated from a series of curves at different stresses. A plot of "at" against t was linear except at very low strains and close to the steady state. The calculated a values are shown in Table 6-1 as a function of  $\dot{\epsilon}_{e}$ .

The basic creep curves for copper-tin in Figure 5-2 can be expressed as:

$$\varepsilon = 7.77 \times 10^{-4} t - 0.011 (1 - e^{-0.075t})$$
 (6-15)

and:

$$\varepsilon = 8.33 \times 10^{-3} + 0.018 t + 0.012 (1 - e^{-10.41t})$$
 (6-16)

respectively. Thus, while the a values for copper and copper-tin are comparable at low strain rates, that for copper-tin is more rate sensitive. Comparable values for silicon steel cannot be obtained because of the more complex type of creep curves produced by this material.

The "normal" shaped curves for copper reflect the relatively high velocity of dislocations in a pure material where the friction stress is low. Thus, even at low stresses the time of flight is sufficiently small to approximate to situation of equation (6-14).
# TABLE 6-1

# CALCULATED VALUES OF THE

COEFFICIENT a IN EQUATION (2-1)

	<u> </u>	έ <sub>s</sub> /min	a/min
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1)	Copper		
		$2.65 \times 10^{-5}$	0.0053
		6.17 x 10 <sup>-5</sup>	0.0137
		$1.50 \times 10^{-4}$	0.0295
		5.38 x $10^{-4}$	0.0602
		$1.70 \times 10^{-3}$	0.155
		$3.70 \times 10^{-3}$	0.370
		$1.04 \times 10^{-2}$	1.443
	×	$8.63 \times 10^{-2}$	12.55

ii) Copper-tin

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7.77	x	10-4	0.075
1.80	x	10 <sup>-2</sup>	10.41

In copper-tin the inverted creep curves at low stresses imply an increasing effective stress. The corresponding decline in internal stress has been detected during primary creep of aluminium-magnesium (Ahlquist and Nix, 1971). Since an increasing total dislocation density implies an increase in internal stress, then provided that the velocity is not strongly stress dependent, the increase in strain rate at constant stress is the result of an increasing mobile fraction of dislocations at ostensibly constant total density. This is essentially the glide controlled situation of equation (6-12). At high stresses, i.e., those above the initial internal stress, there is an initial rapid extension as in pure copper. In this case the higher density of dislocations, decreasing during primary creep, transforms the transient to one of declining strain rate, as indicated by equation (6-14).

### 6-5. Load Change Transients

The single stress change curves of Figure 5-8 can be analysed in the same way as for the creep curves by treating any initial rapid extension as  $\varepsilon_0$  (identified with a rapid increase in dislocation density). On each of these figures the best fit curve of the form of equations (2-1) and (6-11) has been determined and as can be seen from the

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calculated points in each figure, the agreement is very close.

#### 6-5-1. Copper

The stress increment transient in Figure 5-8(d) has the form:

$$\varepsilon = 0.0116 + 0.0146 (1 - e^{-5.65t}) + 0.0214 t$$
 (6-17)

where t is in minutes. The a value at this strain rate predicted from the creep data in Table 6-1 is about 3.1, which, in view of the experimental scatter compares well with the 5.6 above. A fit to the data using fixed values for a and  $\dot{\epsilon}_{s}$  (predicted and measured respectively) diverges at short times.

In equation (6-11):

$$\epsilon_{t} = \frac{\alpha b V}{a^{2}} \left( \frac{N_{o}}{t_{w}} - a N^{o} \right) = \frac{\epsilon_{s}}{a} \left( 1 - \frac{a N^{o}}{N_{o}} t_{w} \right)$$
(6-18)

from the above values,  $aN^{O}/N_{O} t_{W} \approx 3.85$ .

To see if this gives sensible values for  $t_w$ , a value for  $N_0^O/N_0$  is needed. If it is assumed that the peak in mobile dislocation density during a transient corresponds to the point at which all the potentially mobile dislocations are mobile (which must be an underestimate for  $N_0$ ) then  $N_0^O/N_0$  can be put equal to unity, where  $N_0^O$  is the peak value. Thus at<sub>w</sub> = 3.85 and since a =  $(1/t_w + 1/t_f)$ ,  $t_w/t_f \approx 3$ . At this stress, equation (2-8) predicts a value of  $10^9/cm^2$ for the mobile dislocation density, so that, from equation (2-1) the mean velocity is about  $10^{-3}$  cm/min. This is orders of magnitude greater than that estimated for coppertin in Section 5-8. If the time  $t_f$  is that required for a dislocation moving across a sub-grain of diameter  $10^{-3}$  cm then  $t_f$  and  $t_w$  are of the order of 1 and 3 minutes, respectively.

For the unloading transient in Figure 5-8(a), there is an initial delay period of apparently zero strain rate. During this time, the local effective stress will vary in both magnitude and sign. Only after some form of recovery will a positive strain rate be established and up to this point the effective mobile dislocation density will be zero.

Using the expression:

$$\varepsilon = \dot{\varepsilon}_{s} (t-t_{o}) + \varepsilon_{t} (1 - e^{-a(t-t_{o})})$$
 (6-19)

where  $t_0$  is the delay time and  $\varepsilon_t$  is now negative the data in Figure 5-8(a) gives a best fit at  $\dot{\varepsilon}_s = 2.4 \times 10^{-4}$ /min,  $t_0 =$ 14 mins,  $\varepsilon_t = 0.0086$  and a = 0.0281. The fit is not very sensitive to  $t_0$  and no unique solution was obtained. The predicted points are shown in the figure. The predicted value of a at the reduced stress level is 0.035 at 2.4 x  $10^{-4}$ /min and the measured  $\dot{\epsilon}_s$  is 1.85 x  $10^{-4}$ /min. The agreement is again quite reasonable.

According to the model, for lower velocity equation (6-13) gives  $\varepsilon_t = -\dot{\varepsilon}_s/a$  when  $N^0 = 0$ . For  $\dot{\varepsilon}_s = 2.4 \text{ x}$  $10^{-4}/\text{min}$  and a = 0.0281,  $\varepsilon_t$  is found to be 0.0085, which is exactly that found.

Thus the primary and transient data for copper can be explained semi-quantitatively by the model in terms of changes in mobile dislocation density.

### 6-5-2. Copper-tin and Silicon Steel

The load change data for copper-tin gives from Figures 5-8(e) and 5-8(b):

 $\varepsilon = 0.066t + 0.0084 (1 - e^{-49.3t})$  (6-20) for the upward change, and:

$$\varepsilon = 3.06 \times 10^{-4} t + 2.96 \times 10^{-4} (1 - e^{-0.946t})$$
 (6-21)

for the load reduction.

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Comparison with the creep data shows that while the exponential coefficient for the upward change in stress is of the <u>magnitude</u> for that strain rate, the value for the reduction in stress is one order of magnitude too high. While care must be taken in interpreting this data because the strains involved are very small, this observation suggests that changes in dislocation mobility associated with stress changes are much more important in copper-tin than in copper. In copper, there is apparently no history effect in the coefficient a. In copper-tin, however, on dropping the stress, dislocations have greater mobility than in the steady state because initially they are still free of atmospheres, so that this history dependence of velocity shows up in a higher a value.

The curves in Figure 5-8(f) and 5-8(c) can be expressed roughly as:

$$\varepsilon = 0.0166t - 0.0015 (1 - e^{-9.36})$$
 (6-22)

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and:

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$$\varepsilon = 1.47 \times 10^{-4} t - 7.39 \times 10^{-4} (1 - e^{-0.206t})$$
 (6-23)

respectively. While there is no primary creep data to compare this result with, it is worth noting that these a values are intermediate between those for copper and copper-tin.

#### 6-6. Conclusions

The foregoing model provides a semi-quantitative framework for dealing with strain transients, whether they arise from load changes or from a single load creep test. The coefficient a has been shown to be a sensitive material parameter related to the mobility of dislocations while the transient strain parameter  $\varepsilon_t$  is chiefly dependent on the changes in mobile dislocation density. While in certain circumstances the resulting equations resemble those of Gittus there is a fundamental difference in approach. Thus, the model developed here makes no reference as such to hardening and recovery as separable processes which directly influence the deformation. In addition, the dynamic processes of dislocations gliding and being held up at obstacles is essentially "non-structural" and runs contrary to the network growth view of creep.

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The model suggests that the substructure does not in fact influence directly the mobility of dislocations, since, for example, it could be shown that, in a structural material such as copper, the parameter a was not sensitive to the state of the structure, and thus is the same at a given stress level both during a primary creep curve and after a load change. Such a conclusion greatly facilitates the comparisons of materials of varying creep responses, since the most important differences arise from effects on dislocation mobility. In the following chapter, the problem of microstructural influence on mobile dision behaviour will be of primary interest.

### CHAPTER 7

### MICROSTRUCTURAL OBSERVATIONS

### 7-1. Introduction

The study of microstructures is made necessary by the fact that different materials develop widely different structures after creep. The study of the various forms of mechanical response would thus be incomplete without some knowledge of the substructural arrangements and the ways in which they could influence the mechanical behaviour. It has already been suggested that many mechanical transients have been interpreted in terms of a resistance to flow originating within sub-boundaries, and one purpose of the examination of substructures is the assessment of the extent to which the observed features could provide such a strengthening. Since the basic relationship of any creep study is that between the strain rate and the applied stress, any model of substructural arrangements should be formulated to translate these mechanical data into a prediction of some structural parameter.

The objects of the experimental observations described here were: to determine the degree of uniformity of the microstructures, to perform a detailed analysis of the most representative features, and to assess the basic dif-

ferences between the structures of the three materials studied.

There are several microstructural features which provide evidence of non-uniformity, most notably, the existance of slip bands, deformation bands, and grain boundary distor-The extent to which these non-uniformities persist in tion. (say) secondary creep is a strong indication of the scale on which the substructural changes which lead to the establishment of a new steady state can occur. In a material within which slip lines persist during secondary creep, for example, the structural change accompanying a change in stress may not simply be a uniform response in sub-grain size or mobile dislocation density; rather it could conceivably take the form of a change in slip line spacing. The existance of such structural non-uniformity indicates a response to local differences in strain and also the extent to which a creep model based on uniform dislocation activity throughout a material can be expected to apply.

The detailed analysis of the more microscopic features includes measurement of the sub-grain size, the degree of equiaxiality of the sub-grains, and the magnitude and variability of sub-grain misorientation. The sub-grain size is the structural parameter most commonly used as a measure of the mechanical resistance provided by the sub-boundaries. The misorientation provides a good indication of the strain inhomo-

encity since it describes the total accumulation of dislocations within a given boundary. The dislocation density not associated with sub-boundaries is also of interest, being commonly associated with the mobile dislocation density.

## 7-2. Experimental Observation on Copper

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### 7-2-1. Optical Microscopy

Photographs 7-1 and 7-2 show the surface markings on a copper single crystal and a polycrystalline sample both deformed 10% at  $550^{\circ}$ C at a crosshead speed of 2 x  $10^{-3}$ /min. The tests were performed in a flow of Argon to prevent the formation of an oxide layer. Slip bands can be seen in both specimens. In the single crystal, they follow the trace of the primary slip plane. The slip band spacing is of the order of 10 - 20 microns in each case, which is considerably finer than that of Clauer et al (1970) on molybdenum and is at the low end of the spacings reported for aluminium by Garofalo (1965). In Photograph 7-2, some coarser bands can be seen at points A. Deformation bands were not observed, possibly because the surfaces were not clean enough to reveal such thermal etching effects. After straining well, into stage III



Photograph 7-1. Slip lines observed on a copper single crystal after 10% deformation at 2 x 10<sup>-3</sup>/min (X 150)



Photograph 7-2. Slip lines observed on a copper polycrystal after 10% deformation at 2 x  $10^{-3}$ /min (X 150)

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the overall grain shape in copper was similar to that in the annealed specimens. Some grain boundaries were a little distorted but the annealing twin boundaries still retained their sharp corners. No grain boundary cavities were found except in broken specimens.

## 7-2-2. Substructural Observations in Copper Single Crystals

After 1% strain, in (111) sections a fine dislocation debris was found, the longer lengths of dislocation lying along the primary edge direction [121]. The dislocation density was  $1.3 \pm 0.3 \times 10^9$ /cm<sup>2</sup> (based on nine observations with an assumed foil thickness of 0.225 µ). In (101) sections there were a few narrow boundaries aligned in the primary slip plane trace.

After 11% strain in (111) sections, the only additional feature was some small networks aligned parallel to the primary screw direction [ $\overline{101}$ ] their ends running towards [ $\overline{121}$ ], and containing sets of parallel dislocations. Elsewhere, the dislocation density at 1.4  $\pm$  0.3 x 10<sup>9</sup>/cm<sup>2</sup> was unchanged.

In (121) sections, well defined sub-boundaries were in evidence lying in the traces of both the primary and cross slip planes.

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After 39% strain in (111) sections, the substructure was composed of well developed, roughly equiaxed subgrains and extensive hexagonal networks adjacent to the boundaries. The sub-boundaries were aligned along  $[1\bar{2}1]$  and  $[\bar{1}10]$ , i.e., the primary edge and conjugate trace directions. The subgrain diameter was of the order 5 - 10 microns, but was quite variable, and Kikuchi line shifts across the subboundaries were very small, indicating misorientations of only a few minutes. Burgers vector analyses were performed on the networks by observing the contrast at different operating reflections. The network consisted of two parallel sets of screw dislocations of burgers vector a/2 [ $\bar{1}01$ ] and a/2 [ $1\bar{1}0$ ] which are the two most highly stressed coplanar (111) systems and are inclined at  $60^\circ$  to each other.

In ( $\overline{101}$ ) sections at 39% strain the subgrains were more regular and elongated along [ $1\overline{21}$ ] with occasional cross linkages (Photograph 7-3). However, the structure was inhomogeneous with clumps of subgrains separated by large dislocation free areas. There was a very strong alternating contrast across the sub-boundaries normal to the primary slip plane trace. Kikuchi line analysis indicated that the misorientation also alternates and was of the order of  $1^{\circ}$ , comprised predominantly of a tilt across [ $1\overline{21}$ ].

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In some areas, the sub-boundaries ran into regions of coarser networks similar to those seen on the primary slip



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Photograph 7-3. Dislocation substructure in copper single crystal deformed 39% at 2 x 10-3/min. (IO1) section. (X 6000)



plane. The width of the networks seen in ( $\overline{101}$ ) indicated that they lay in the conjugate ( $\overline{111}$ ) plane. Burgers vector analysis showed that they were composed of two sets of parallel screw dislocations with burgers vectors a/2 [011] and a/2 [ $1\overline{10}$ ] (Photographs 7-4 and 7-5). Both these configurations are highly stressed, though since the nets are aligned along the primary slip plane trace, they could be formed by the interaction of ( $\overline{111}$ ) [011] with the highly stressed (111) [ $\overline{110}$ ] screw dislocations from the primary slip plane.

The above observations are in good agreement with those of previous workers on copper. The main features are the slow build up of essentially tilt walls perpendicular to the primary slip direction, and the formation of networks by the intersection of coplanar (111) screw dislocations. In addition, screw networks develop at the intersection of the ^ primary and conjugate planes adjacent to the tilt walls, with which they interact.

The strong alternating contrast seen in (101) sections at high strains is considered to arise from the interaction of primary edge dislocations with forest dislocations of alternating sign. According to Steeds (1966) these forest dislocations would have [011] or [110] burgers vectors and Orlova and Cadek (1970) found that these boundaries contain a/2 [011] and a/2 [110] dislocations from the cross slip

plane as well as primary and coplanar (111) dislocations. The cross slip plane is perpendicular to the ( $\overline{1}01$ ) plane, so that such boundaries would be quite narrow in this section.

# 7-2-3. Substructural Observations in Copper Polycrystals

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The structure of the annealed material was almost featureless with a few short lengths of dislocation scattered throughout. The dislocation density varied from virtually zero to  $10^8/\text{cm}^2$ , but the method of measurement was insensitive to densities below  $10^7/\text{cm}^2$  so that an average could not be computed.

The structures of two series of specimens deformed 7 to 9% at 2.9 and 4.6 kg/mm<sup>2</sup> (with steady state strain rates of 3 x  $10^{-4}$ /min and 9.5 x  $10^{-3}$ /min) were extensively examined. A well developed subgrain structure was found in each, but several different types of subgrain groupings were observed. Qualitatively, the two strain rates resulted in similar structures, the following description concentrates on that obtained at the lower strain rate, comparisons being provided where appropriate.

### a) General Description

There was a pronounced variation from equiaxed (Photograph 7-6) to elongated bands of parallel subgrains. Both structures were found within the same grain and there is an overall sense of alignment in a slip plane trace. Occasionally the bands of parallel subgrains were separated by equiaxed regions roughly 10 - 20 microns wide, that is of the same separation as the observed slip lines. However, since the foils were randomly oriented it is not surprising that these observations were the exception rather than the rule. In addition, the directionality in the equiaxed zone was more pronounced adjacent to grain boundaries running approximately in the slip trace direction.

There was no evidence of a general pattern of groups of subgrains of small misorientation within higher angle sub-boundaries. Clumps of subgrains were found which were apparently equally misoriented about a common zone axis, so that the clump appeared uniform in contrast with respect to its surroundings. The misorientations within the group were found to be of the same magnitude as those between it and the surrounding subgrains.

The twin boundaries retained their sharp corners after deformation and no evidence of recrystallisation or grain boundary migration was ever seen. The substructure





Photograph 7-6. Dark field micrograph showing substructure in polycrystalline copper deformed 8% at 2.9 kg/mm<sup>2</sup>,  $\dot{\varepsilon}_{\rm g} = 2.4 \times 10^{-4}/{\rm min.}$  (X6000)

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after deformation at the higher strain rate showed similar variations in subgrain size and subgrain arrangements. The subgrain size was finer and the sub-boundaries were more ragged than at the lower stress. Photographs 7-7 and 7-8 show a roughly equiaxed region and a region of elongated subgrains, respectively.

### b) Sub-grain Size

For subgrain size measurements, twelve areas of foil comprising 5900 square microns with a total subgrain count of 715 were taken from several specimens deformed at 2.9 kgm/mm<sup>2</sup>. The average subgrain size was  $3.9 \pm 0.2$  microns where the limits indicate the 90% confidence interval for the mean. From foil to foil, the average varied from 3.45 to 4.5 microns and a histogram showed the distribution to be symmetrical about the mean. Determinations were also done on two areas of foil after creep at the higher stress, the average subgrain size in this case being 3.15 microns.

The deviation from equiaxiality was assessed on the same twelve areas of foil from the number of sub-boundary intercepts with a 10 cm line placed along and normal to the prominent (111) trace. At X11,000 the number of such intercepts varied from 1 to 10, a total of 107 such measurements being



Photograph 7-7. Dislocation substructure in polycrystalline copper deformed 9% at 4.6 kg/mm<sup>2</sup>  $\dot{\epsilon}_{s} = 1.0 \times 10^{-2}/min.$  (X 11,600)



Photograph 7-8. Dislocation substructure in polycrystalline copper deformed 9% at 4.6 kg/mm<sup>2</sup>

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made. The average intercepts were  $3.6 \pm 0.3$  parallel and  $5.05 \pm 0.45$  normal. There is thus a significant elongation of subgrains in the trace direction.

According to Bird et al (1969) copper has one of the smallest subgrain sizes found in crept material at equivalent stresses, being from  $10^{4}$ b to  $10^{5}$ b (b is the burgers vector) for stresses between  $10^{-3}$  and  $10^{-4}$  G (i.e., stresses between 1 and 11 kg/mm<sup>2</sup>). For b = 2.5 x  $10^{-8}$  cm, the subgrain size becomes 2.5 to 25 microns. Only a few subgrain size measurements have actually been carried out on copper thin foils. Barrett (1965) obtained a value of 3.3 microns at  $500^{\circ}$ C and 2.1 kg/mm<sup>2</sup>, and Lloyd and Embury (1970) found variations from 1 to 3 microns over the stress range 1.8 to 4.6 kg/mm<sup>2</sup>. More recently, Staker and Holt (1972) obtained an average value of  $3.37 \mu$  from 25 areas at  $550^{\circ}$ C with a strain rate of 5 x  $10^{-3}$ /min which is in excellent agreement with those above.

## c) Sub-grain Misorientation

Kikuchi line shifts were measured between groups of subgrains on four areas at the low stress level comprising 36 measurements, and on three areas at the high stress levels comprising 18 measurements. The average misorientation at

2.9 kg/mm<sup>2</sup> was  $38.4 \pm 6.5$  minutes and at 4.6 kg/mm<sup>2</sup>, 27.9  $\pm$  8.0 minutes. There was considerable range of values from 96 minutes to 12, but only three values exceeded 55 minutes. The difference between these means is not significant at the 10% level of significance.

The extent of tilt and twist was variable and complex and it was very difficult to get a clear overall view. A few characteristic arrangements do occur, one of which is shown in Photographs 7-8 and 7-9. In both these, the alternating contrast coincides with alternating tilt misorientations, and is typical of regions of elongated parallel subgrains. This structure is very similar to that observed in single crystals at large strains. In regions where the structure was more equiaxed, though still showing pronounced directionality, there was still the alternating tilt misorientation, but in addition an alternating twist can be detected about the cross-linking boundaries. This is shown schematically in Figure 7-1. Figure 7-2 shows an unusually clear example of this, Photograph 7-10 being from part of the same field. The length and direction of the arrows gives the relative magnitude and directions of the rotation of the subgrains. In other regions, the misorientations are much more variable, though tilt boundaries are still found more frequently than twists.

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Figure 7-2. Experimental results of misorientations taken from copper crept 9% at 2.9 kg/mm<sup>2</sup>,  $\dot{\epsilon}_{s} = 2.7$ x 10<sup>-4</sup>/min. Arrows indicate the relative directions and magnitudes of Kikuchi line

#### d) Sub-boundary Structure

Only rarely was the area covered by a sub-boundary large enough to permit any details of their dislocation content to be extracted. Practically all such networks were found from burgers vector analysis to be composed of two sets of parallel screw dislocations forming a crossgrid. An example is shown in Photograph 7-11, where the boundary is possibly made up of coplanar [011] and [110] screw dislocations lying in the (111) plane. In this indexing framework, they could also have coplanar burgers vectors [110] and [011]. Thus, as in the single crystals, the most frequently observed burgers vectors could be assigned to the primary and conjugate planes, though of course the traces cannot be assigned with certainty.

The difficulty in finding resolvable non-screw networks may arise from the greater mobility of screw dislocations so that screw networks can form on several intersecting (111) type planes. Tilt boundaries would be restricted to orientations close to ( $\overline{101}$ ) planes, so that the chance of finding a wide boundary in the foil is lessened.

The stability of the substructure was clearly demonstrated by the fact that after holding at the creep temperature for up to 200 hours following removal of the load during steady state creep, there were no significant sub-

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x 10<sup>-2</sup>/min. (X 40,( )

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$$g = [\overline{2}02]$$
  
 $b = [\overline{1}10]$   
 $b = [0\overline{1}1]$ 



structural changes in either character or size.

Observations of the transverse surfaces of broken specimens showed that fracture occurred by intergranular separation at low and intermediate stress and by necking and apparently transgranular fracture at high stresses.

### 7-3. Structural Observations in Copper-Tin

Photograph 7-12 shows copper-tin in the annealed condition and Photographs 7-13 and 7-14 were taken after 10% deformation at 2.7 kg/mm<sup>2</sup>. In these and the remaining photographs in this Chapter, the tensile axis is horizontal. The severe grain boundary distortions are in sharp contrast to the structure of copper after deformation. There are numerous cavities on transverse grain boundaries particularly at triple points and at twin boundary/grain boundary intersections. Extensive grain boundary migration has occurred and within the grains the twins are frequently bent and some of the twin boundaries in the largest grains have become ragged. Most of the cavities are located on grain boundaries, indicating a retarding effect on boundary mobility and suggests that at this strain, migration is still occurring locally rather than across entire grains.





Photograph 7-13. Micrograph of copper-tin after 10% strain at 2.7 kg/mm<sup>2</sup> (X 600)

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Photograph 7-14. Micrograph of copper-tin after 10% strain at 2.7 kg/mm<sup>2</sup> (X 1600)

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Photograph 7-15. Micrograph of copper-tin after 3% strain at 2.7 kg/mm<sup>2</sup> (X 600)

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The effect of strain was assessed by comparing these observations with those from specimens crept only 3% at the same stress. As can be seen in Photographs 7-15 and 7-16, the extent of boundary distortion is considerably reduced though still apparent, and the twin boundaries are undisturbed. However, it is remarkable that even at this strain cavities have already formed on many transverse boundaries and triple points. The structure observed after 3% at a higher stress (9.0 kg/mm<sup>2</sup>) is shown in Photographs 7-17 and 7-18. The boundaries are ragged but the twins are quite sharp and there are fewer cavities.

As stated previously, very few thin foils could be produced with copper-tin, and none of these contained any grain boundaries, which would have produced the most interesting observations. The presence of the cavities and the grain boundary distortions combine to give preferential thinning at these points. Photograph 7-19 shows a typical electron micrograph. There is no evidence of sub-boundary formation and the overall appearance is like that in the primary plane in copper single crystals at low strains.



Photograph 7-16. Micrograph of copper-tin after 3% strain at 2.7 kg/mm<sup>2</sup> (X 1500)



Photograph 7-17. Micrograph of copper-tin after 3% strain at 9.0 kg/mm<sup>2</sup> (X 250)

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Photograph 7-18. Micrograph of copper-tin after 3% strain at 9.0 kg/mm<sup>2</sup> (X 250)



Photograph 7-19. Electron micrograph of copper-tin after 3% strain at 2.7 kg/mm<sup>2</sup> (X 40,000)

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7-4. Structural Observations in Silicon Steel

The etch-pitted substructures observed in this material after deformation at the two stress levels were very different, and also showed considerable variation within a given specimen.

After 8% strain at 4.5 kg/mm<sup>2</sup>, the substructure varied between a) regions of random individual dislocations, b) regions of coarse equiaxed subgrains within partly completed sub-boundaries, and c) bands of sub-boundaries which were more pronounced adjacent and parallel to a grain boundary. Examples are shown in Photographs 7-20, 7-21 and 7-22. In the equiaxed regions, the subgrain size is about 10 microns, but between boundaries in the bands it varies from about 0.1 to 5 microns. The total dislocation density in those areas without sub-boundaries was found to average 7.0 x  $10^7$  $\pm 0.2/cm^2$  (the results on silicon steel are all averages of 9 measurements) compared with the as-annealed value of  $6.0 \times 10^6/cm^2$ .

The structure after deformation at 9.5 kg/mm<sup>2</sup> was also variable but only from grain to grain rather than within individual grains. In some grains the distribution was random (Photograph 7-23) at a density of 1.5 x  $10^8/cm^2$ . Elsewhere, parallel bands of high density separating narrow low density areas ran right across the grain. In most cases,



Photograph 7-20. Micrograph of etch pitted silicon steel after 8% strain at 4.5 kg/mm<sup>2</sup> (X 2,000)



Photograph 7-21. Micrograph of etch pitted silicon steel after 8% strain at 4.5 kg/mm<sup>2</sup> (X 2,000)

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Photograph 7-22. Micrograph of etch pitted silicon steel after 8% strain at 4.5 kg/mm<sup>2</sup> (X 2000)



Photograph 7-23. Micrograph of etch pitted silicon steel after 8% strain at 9.5 kg/mm<sup>2</sup> (X 1600)

the bands ran at about  $55^{\circ}$  anti-clockwise to the rolling direction (Photographs 7-24 and 7-25). Roughly perpendicular to these bands ran occasional short bands, which close to grain boundaries, resemble sub-boundaries. Where resolved the total dislocation density in the banded regions was 1.4 x  $10^8$ /cm<sup>2</sup> which is almost identical to that in non-

In neither test condition do the grain boundaries show the gross distortion of copper-tin, but serrations were observed, particularly in the boundary between a banded grain and a homogeneous one. In Photograph 7-24 for instance, the grain boundary contains pronounced serrations and opposite the bands there is intense dislocation activity. Serrations can also be seen in Photographs 7-26 and 7-27. However, features such as at B in Photograph 7-27 were also observed in annealed material. This photograph also shows a fan of high density associated with many triple points akin to the phenomenon of fold formation observed macroscopically in some materials. In a few cases, there is evidence of grain boundary migration accompanying serrations. In Photograph 7-26 the boundary has apparently moved up leaving a region of lower dislocation density behind it within which are trailing sub-boundaries. Small dislocation-free grains can also be seen growing out of this boundary which may now

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Photograph 7-26. Micrograph of etch pitted silicon steel after 8% strain at 9.5 kg/mm<sup>2</sup> (X 1000)



Photograph 7-27. Micrograph of etch pitted silicon steel after 8% strain at 9.5 kg/mm<sup>2</sup> (X 1600)

#### CHAPTER 8

## DISCUSSION OF THE MICROSTRUCTURAL OBSERVATIONS

### 8-1. Copper

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The non-uniformity of the substructure of copper after creep deformation makes it difficult to establish the general characterisation of the dislocation arrangements. The observations suggest that the distribution and size of sub-grains is related to the formation of slip bands. Certain well-defined boundaries develop as a result of the intensive localised slip in these bands. These boundaries appear to be fixed features throughout secondary creep and form a skeleton between which less well defined sub-grains are formed. The processes of primary and transient creep then involve changes in the scale of the substructure between the bands. Though different in appearance this duplex structure may be analogous to those reported by other authors on crept materials.

The different types of boundaries observed, primary tilt walls and nets of coplanar or primary/conjugate screw dislocations are basically similar to those formed at lower temperatures, although somewhat more regular in appearance.

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elv stress free, but

their regularity suggests that the stress fields are small.

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In a detailed study of copper at room temperature, Steeds (1965) has pointed out that the formation of tilt walls and screw networks of alternating sign in an arrangement such as that in Figure 7-1, requires secondary dislocations. In particular, simple coplanar screw networks cannot produce this situation. One consequence of such an interaction between primary and secondary dislocations is that the crystals maintain a memory of their initial orientation, so that such a mode of structure formation, given a capacity for dislocation annihilation, could lead to the establishment of a quasi-steady state.

Although a partial reduction in applied stress is known to be followed by structural changes leading to a coarser sub-grain size, it has been shown here that no such coarsening occurs when the stress is totally removed. This implies that the recovery process leading to coarsening is not driven by a long range internal stress, i.e., one extending over an entire sub-grain, otherwise coarsening would be most pronounced in the absence of an applied stress. Thus, the motion of dislocations between cell walls driven by the applied stress must induce a dynamic instability and be an essential feature of substructural reorganisation. The consequences of this situation are quite profound. The

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source of internal resistance is misleading. Rather, the sub-boundaries, in this framework, are seen to be a form of debris, the strengthening effect of which is critically dependent on the instantaneous mobile dislocation density and distribution. It is thus not surprising that, in Chapter 6, the transients resulting from a stress change could be successfully modeled in a semi-quantitative fashion without reference to sub-grain size, the important parameters being the mobile density and velocity.

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At this point, there is still a need to develop models for describing the processes of sub-grain formation at least to the extent that some predictions could be made on the sub-grain size developed at a given stress level. Holt (1970) has examined the formation of a cell structure by the breakdown of a random array of dislocations due to a process akin to spinodal decomposition. In this type of substructure, the internal stress between adjacent cell walls is small relative to that within the walls. Furthermore, boundaries of opposite sign alternate across the substructure in accord with experimental observation.

Unfortunately, during high temperature creep subboundaries do not develop from the random array of dislocations visualised by this model. Slip occurs inhomogeneously and there is evidence to suggest that during primary creep

of irregular dislocation groupings formed shortly after stress application. The description of the dynamics of mobile dislocations given in Chapter 6 suggests that in copper, when dislocations move between obstacles, they do so rapidly relative to the waiting times at obstacles, so that any approximation to a random array is at best a very poor one.

It has been shown that the average sub-boundary misorientation is independent of the applied stress level, and thus the steady state at a given temperature is characterised by a limiting value of the mean sub-boundary energy. In the absence of a long range internal stress the driving force for coarsening arises from the need for sub-boundaries to reduce their energy towards a local equilibrium value. Without an applied stress, there is little motion of dislocations between cell walls so that the increases in boundary energy needed to maintain the recovery process are absent, and little coarsening can occur.

The question thus arises as to whether a model could be devised in which the rates of dislocation accumulation and removal implied by the above can be balanced in such a way as to predict the scale of the cell structure. To do this the steady state is defined as the establishment of a constant cell structure assuming a constant value for the misorienta-

of

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dislocations in the cell

walls leads to a refinement of the sub-grains, and recovery simultaneously allows the climb of dislocations with subboundaries and sub-grain coarsening.

To describe the refinement process consider an array of cubical sub-grains of side L and misorientation  $\theta$ . The dislocation density of this array is:

$$\rho = \frac{3\theta}{bL}$$
(8-1)

and for constant  $\theta$ :

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$$\dot{\rho} = -\frac{3\theta}{bL^2} \dot{L}$$
 (8-2)

The strain rate can be expressed as:

$$\dot{\varepsilon} = \frac{bL}{2}\dot{\rho}$$
(8-3)

where the average slip distance is identified as L/2 and is constant, thus:

$$\left(\frac{dL}{dt}\right)_{refinement} = -\frac{2\tilde{\epsilon}L}{3\theta}$$
 (8-4)

The value of the proportionality constant will vary according to the shape of the sub-grains and the location of the dislocation sources. Since it is energetically favourable for two small angle boundaries to combine to form one of a larger angle, the above refinement would be difficult to maintain in the presence of a recovery mechanism. However, it is an experimental fact that the average misorientation is stress independent while the mean subgrain size varies inversely with stress. Consequently, the precise method of forming a new boundary cannot easily be specified. In the steady state, with which this model is concerned, the subgrain size could in principal be held constant and the angle varied, but the results should be equivalent for the same basic mechanism.

There are several possible ways of describing the coarsening process. Li (1962) has considered the kinetics of subgrain coalescence. This process involves the rotation of adjacent subgrains so that a common boundary gradually fades away. One feature of such a process is that because of their larger energy/unit area, twist boundaries tend to be eliminated in favour of tilt boundaries. The observed dominance of tilt boundaries in creep substructures may result from coalescence during subgrain formation.

The following treatment parallels that of Li but considers dL/dt with  $\theta$  constant rather than  $d\theta/dt$  with L constant.

Consider a sub-boundary, misorientation  $\theta$  coarsening by the climb of dislocations at each end. It is assumed that there is a supply of fresh dislocations at the centre of the wall which serves to maintain the overall density in the boundary and thus its

It is assumed that the rate controlling

another. Thus, the movement of material needs to be described rather than the details of the dislocation structure.

To get an expression for the rate of coarsening, the vacancy flux which must be maintained can be calculated and the corresponding energy consumption can be equated to the change in stored energy of the boundary due to the coalescence process.

The rate at which dislocations climb past a given atom row in the boundary is (1/h) (dL/dt), where h is the dislocation spacing and dL/dt is the rate of coarsening. Since  $\theta = b/h$ , the number of vacancies per row which must be supplied or removed in unit time is  $(\theta/b)$ (dL/dt). This quantity represents a number per atom in the boundary, the number per unit area being:

$$\frac{dn}{dt} = \frac{\theta}{b^3} \frac{dL}{dt}$$
(8-5)

The driving force, F, per unit length of the boundary is related to this flux by the basic diffusion equation  $J = (D/\Omega kT)$  F, so that for  $\Omega = b^3$ 

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the driving force for diffusion is:

$$\mathbf{F} = \frac{\mathbf{k}\mathbf{T}\theta}{\mathbf{D}} \frac{d\mathbf{L}}{d\mathbf{t}}$$
(8-6)

operating over a distance L.

The rate of free energy consumption is thus:

$$\frac{dE}{dt} = \int_{0}^{L} L \frac{dn}{dt} F dL = \frac{L^2}{2} \frac{\theta^2 kT}{D\Omega} \left( \frac{dL}{dt} \right)^2$$
(8-7)

For a boundary length L the corresponding decrease in boundary free energy is:

$$\frac{dE}{dt} = E \frac{dL}{dt}$$
(8-8)

where for a tilt boundary:

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$$\mathbf{E} = \mathbf{E}_{\mathbf{O}} \boldsymbol{\theta} \quad (\mathbf{A} - \mathbf{ln} \ \boldsymbol{\theta}) \tag{8-9}$$

where  $E_0 = Gb/4\pi(1-\nu)$  and A is a constant  $\nu$  0.4. Equating (8-7) and (8-9):

$$\left(\frac{dL}{dt}\right)_{\text{coarsening}} = \frac{2 E_0 (A-\ln\theta) D\Omega}{L^2 k T \theta}$$
 (8-10)

· · · is twice this rate because it is

Equating (8-10) and (8-4) gives the required prediction for L:

$$L = \left(\frac{6 E_0 (A-\ln\theta) D\Omega}{kT\dot{\epsilon}}\right)^{1/3}$$
(8-11)

For copper,  $G = 3,850 \text{ kg/mm}^2$ ,  $b = 2.5 \times 10^{-8} \text{ cm}$ ,  $\theta = \frac{1}{2}^{\circ}$ ,  $\Omega = b^3$ , and  $D = 7.04 \times 10^{-14} \text{ cm}^2/\text{sec}$ ; thus:

$$L = \left(\frac{5.25 \times 10^{-5}}{\epsilon}\right)^{1/3}$$
 (8-12)

where L is in microns and  $\dot{\epsilon}$  in sec<sup>-1</sup>.

The predicted L values for copper for a range of strain rates are shown in Figure 8-1 as a function of  $\sigma_{\rm app}/G$ using the strain-rate/stress measurements of Table 5-1. Also shown are the two values measured during the thin foil experiments plus some values reported elsewhere. The predicted values are of the right order at low stresses, but are up to a factor of five times too small at high stresses. There are a number of reasons why the predicted values might be too low. Firstly, the refinement term is probably an overestimate since it makes no allowance for loss of disloca-<sup>5</sup> tions within sub-grains or locally at sub-boundary walls. Likewise, the coarsening term is a minimum because the diffusion process is assumed to be occurring through the lattice

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Fig. 8-1 Predicted and experimental values of subgrain size in copper at 550°C, as a function of applied stress.

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According to Mukherjee et al (1969) from equations (2-5) and (2-6), there should be a relation of the form:

 $\frac{\dot{\epsilon}kT}{DGb} = A^{*} \left(\frac{b}{L}\right)^{n}$ 

where A' for copper is 
$$\sim 10^{10}$$
 and n is close to the stress  
dependence of the strain rate. Equation (8-11) can be  
rearranged to give:

$$\frac{\dot{\epsilon} kT}{DGb} \simeq B' \left(\frac{b}{L}\right)^3$$
(8-14)

where B' is a constant  $\sim$  3. Thus, the functional form of equation (8-11) agrees with that observed, although there is the problem of the low exponent which is analogous to that of predicting the stress dependence of strain rate from theoretical models. It is possible to arrive at a higher dependence in the hardening term using a more detailed description of dislocation interactions and source distribution, for example by describing the hardening term in terms of a model similar to those used to describe stage II tensile deformation at lower temperatures. Unfortunately, the extra dependence appears as the ratio b/L so that the predicted values are even lower in this case.

One other possibility is that at high stresses the interaction of large numbers of rapidly moving mobile disloca-

(8 - 13)

tions with cell walls occurs by much more complex processes than envisaged in this simple model. A more precise measurement of sub-grain size over a wide range of stresses might well detect a non-linearity in the stress-cell size relationship.

## 8-2. Copper-Tin

Creep in copper-tin obviously involves an interaction between dislocation transport processes within the grains on the one hand, and grain boundary sliding, grain boundary migration and cavity nucleation on the other. Recent reviews on grain boundary sliding and creep cavitation (Stevens, 1966; Perry, 1974) testify to their complexity, and it is not yet possible to predict for example the effect of composition or type of alloying element on the extent of sliding. It is beyond the scope of this report to offer more than a few qualitative comments on the deformation of copper-tin, related to conclusions drawn from published work on other alloy systems.

An initial problem is to assess the extent to which grain boundary processes influence the shape of creep curves and creep transients. It has already been shown that these features can be explained, at least qualitatively, by the

on mobility

resulting from variations in stress. It is generally accepted that grains do not slide as a whole relative to each other, the displacements along boundaries being non-uniform. Such non-uniform sliding must mean that deformation is occurring within grains and sliding is seen to serve primarily as an accommodation process. It is not obvious how a material within which deformation was controlled by grain boundary sliding could exhibit the stress sensitive primary creep curves shown by copper-tin. Likewise, models of grain boundary sliding predict a linear rate dependence of the stress rather than the higher values found in creep.

Experimental evidence shows that grain boundary sliding is an essential feature in the nucleation of cavities during creep. The cavities form on grain boundaries which are normal to the tensile stress. Grain boundary sliding occurs in bursts and sets up stress concentrations at irregularities in the boundary which provoke cavitation whenever plastic flow or diffusion is limited. Grain boundary migration, the formation of servations on boundaries, and plastic flow at points where sliding is arrested, all reduce these stress concentrations and correspondingly cavitation. Fold formation at triple points can also delay intergranular fracture. If the shear strength of the matrix is comparable with that of grain boundaries then grain boundary sliding will be pronounced.

The cavitation and grain boundary distortion in copper-tin is much more pronounced than is usually found in published micrographs on single phase material. The observation of reduced cavitation at the higher stress is in accord with normal experimental observation (see Nemy and Rhines, 1959). It has been argued that at low stresses grain boundary sliding is highly confined to a narrow boundary zone. Thus, the resulting shear is more strongly focused on, for example, a triple point and a higher hydrostatic tension will be attained. Similarly, a high rate of grain boundary migration could act to reduce the sliding, thus ragged boundaries with less cavities are observed.

In pure copper, density measurements indicate the presence of sub-microscopic voids in early stage II creep (Soettner and Robertson, 1961), and intergranular fracture occurs at low and intermediate stresses. These observations, even in the presence of a dislocation substructure which undoubtedly acts to relieve stress concentrations at grain boundaries, have some important implications. In copper, there is apparently little capacity for matrix hardening by alloying without the onset of enhanced grain boundary sliding. The increased resistance to the boundary sliding in copper-tin combined with a probable lack of sub-grain formation represents such a matrix hardening resulting in the enhancement of

# 8-3. Silicon Steel

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The microstructural observations reported here on silicon steel differ from those of previous investigators in regard to the fact that different structures were found at different stress levels. The low stress observations are similar to those of Barrett et al (1966), and those at high stress levels resemble the results of Lytton et al (1965). Both these investigators also encountered grain boundary serrations, and small sub-grains and poligonised walls at grain boundaries.

The orientation of the bands found at the high stress level relative to the rolling direction corresponds to the trace of the most highly stressed slip system for material with the (110) [001] texture. On the other hand, the simple uniform distribution of dislocations observed in some grains implys an orientation for multiple {112}<111> 'slip. It thus appears that the material used in this work has an incomplete secondary recrystallization texture, or at least, a distribution of orientations of the [001] axis in the plane of the sheet about the rolling direction. Commercially, the control of the preferred texture is achieved by an appropriate fine dispersion of a second phase, notably of manganeso gulphide, the precise details of which are unavailable. A comparison of steady state strain rates at comparable stresses indicates that this material was intermediate in strength between that of Barrett et al (randomly oriented) and of Lytton et al (textured), the latter being the weakest. Since there is no grain size dependence of the creep rate, these differences can be linked to differences in texture.

The dislocation densities given here are in excellent agreement with published data. At the low stress level the above authors obtained 1.7 x  $10^7$  and 1.6 x  $10^7$  compared with 1.7 x  $10^7/\text{cm}^2$ . At the high stress level Lytton reported 1.2 x  $10^8$  compared with 1.5 x  $10^8/\text{cm}^2$ .

In contrast to copper-tin, grain boundary sliding is restricted in silicon steel by both grain boundary migration and servation formation, and also by a relatively easy plastic flow adjacent to grain boundaries and in particular at triple points. However, even at low stresses the substructure is nowhere near as well defined as in pure copper and pure iron.

Barrett et al supposed that, being regular planar arrays, sub-boundaries in creep would form more easily when more than one slip system was operating. They maintained that the formation of slip bands in their material was restricted to relatively coarse grains oriented for single slip. The very gradual way in which the sub-grains build up, and the tendency for the partially formed sub-boundaries to be aligned in one cr, at most, two directions, does not support this

argument. It seems more plausible that the sub-boundaries form by the climb polygonization of dislocations, most of which are from a single slip system. The situation envisaged by Barrett et al may be applicable close to grain boundaries where stress concentrations provoke multiple slip and fine sub-grains. The fact that when slip bands form, they do so much more rapidly than sub-boundaries, suggests that subboundary formation is simply a consequence of the absence of localised slip in bands. The slow development of sub-grains is thus primarily a reflection of the lack of both obstacles to dislocation motion and of the limited availability of dislocations of different burgers vectors. Most of the gliding dislocations manage to approach the grain boundaries where, as a result, most of the polygonized walls are found. Note that in the textured material, grains in the multiple slip orientation contained only random dislocations, elsewhere slip bands form. The implication is that the effect of increasing the applied stress is the promotion of slip band activity. At high stresses the high dislocation mobility prevents pile ups within grains which would otherwise lead to polygonfsation.

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## CHAPTER 9 SUMMARY AND CONCLUSIONS

The classification of single phase metals into two groups based on the stress exponent of the secondary creep rate does not make it possible to predict the form of the primary or transient strain-time curve. Likewise the shape of the primary curve at a given stress level does not necessarily provide a characterisation of a given metal over the entire stress range of interest.

While the magnitude of the secondary strain rate itself is independent of the deformation history, the magnitude and form of a strain transient connecting two steady states is highly sensitive to the immediate stress history. Consequently the study of transients provides much more information of fundamental significance. The analysis of transients is much more likely to provide a true material parameter upon which a classification of material behaviour could be based.

Nost creep models, being based on a steady state hardeningrecovery balance address themselves solely to the steady state. In this study, the very variable transients observed have been analysed on the basis of a modified mobility model, where creep is seen simply to be a process of dislocations gliding between obstacles, material variations arising from the relative magnitudes of the time of flight between obstacles and of the waiting times at obstacles.

Extension of this approach requires a better fundamental understanding of those microstructural features which control the density and mobility of dislocations. Nuch substructural examination

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in materials deformed in steady state creep would provide an indication of these features. The observations suggest that this may be much more difficult than was foreseen. Characterisation of crept microstructures is hindered by the fact that slip is not homogeneously distributed at high temperatures. This is evidenced not only by slip lines but also by the irregular disposition of subgrains. Measurements of such parameters as subgrain size and dislocation density within subgrains become very approximate and lose much of their potential significance. The fact that subgrains do not coarsen in the absence of an applied stress and that the transients could be analysed without reference to subboundaries implies that substructure is not an essential participant in the process.

The behaviour of sub-boundaries has been modelled so as to predict subgrain size. While there is an approximate agreement, it is recognized that this is a less direct description than the glide approach. In this sense it resembles those recovery models where the hardening and recovery terms are considered separable, and like them, it is very difficult to extend the treatment to non-steady state conditions, where the processes leading to subgrain redistribution may not be themselves substructure dependent.

At this juncture, it is appropriate to indicate the approach which could be taken in a future program to augment that adopted in this thesis. There is a need for much more work on the assessment of the inhomogeneity of slip and substructure. In the initial phase this would require detailed study of single crystals

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in regard to slip line spacings and shape changes during deformation. This would necessarily lead to detailed examination of the inter-relationship in both size and distribution of slip bands and subgrains, and in particular of the way in which these interact during a transient.

There is also a need for more complete data on the form of primary creep curves and creep transients for a larger number of metals. This could be used with a model of the type presented here for transient analysis to develop material parameters such that the classification of metals and alloys can be directly related to the process of strain accumulation.

#### POSTSCRIPT

Kocks has recently presented an analysis of work hardening laws which includes a number of ideas which run parallel to those presented in this thesis. Dealing initially with low temperature deformation he has assumed that recovery is truly dynamic in the sense that it is proportional to the number of times a potential recovery site is contacted by a moving dislocation, and the resulting change in flow stress is strain rather than time dependent.

Using the same starting point as in Chapter 6, the net change in dislocation density, he has redefined the net hardening rate, using essentially dimensional expressions. The result differs fundamentally from the conventional expressions arising from Equation 3-4. Kocks' approach leads to a more empirical law for transient creep which has yet to be tested.

#### Reference:

Kocks, U.F., "Laws for Work Hardening and Low-Temperature Creep" On Micromechanical Modelling of Flow and Fracture,

# APPENDIX A

# THE BASICS OF THERMALLY ACTIVATED DISLOCATION MOTION

It has long been recognised that in a manner analogous to thermal effects in chemical reactions, the thermal motion of the crystal lattice can aid dislocation glide. If the glide process requires the dislocation to surmount an energy barrier  $\Delta F$ , where  $\Delta F$  is the activation energy for the controlling mechanism(s), then the Boltzmann relation predicts that the fraction activated at a given moment, and hence the probability of the event occurring per unit time is:

$$f = \exp^{-\Delta F/kT}$$
 (A-1)

and since the strain rate is proportional to the number of elementary units of flow occurring in a given time, it will also be related to the activation energy by such an Arrhenius equation.

If the structure is assumed to remain constant, then for a single rate process, one can write:

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$$\dot{\epsilon} = \dot{\epsilon}_{0} \exp^{-\Delta F/kT}$$

as the simum attainable creep

(A-2)

rate (when  $\Delta F=0$ ), being a function of the mobile dislocation density and the maximum attainable velocity  $V_0$ . Equation (A-2) is a restatement of the well-known strain rate equation:

$$\dot{\epsilon} = \rho b V$$
 (A-3)

where  $\rho$  is the mobile dislocation density (constant at constant structure), b is the burgers vector and V the average dislocation velocity. Thus, equation (A-2) implies that:

$$V = V_{o} \exp^{-\Delta F/kT}$$
 (A-4)

The applied stress can contribute to the activation free energy by helping a dislocation to pass an energy barrier resisting creep deformation. If the external shear stress  $\tau_{app}$  includes a term  $\tau_{int}$  due to long range internal stresses, then the dislocation is acted upon by an effective stress  $\tau_{eff} = \tau_{app} - \tau_{int}$  attempting to move it over the barrier. Li (1968) and Balasubramanian and Li (1970) have described the kinetics of dislocation motion past such barriers. Figure A-1 shows schematic representations of the free energy profile and the corresponding back stresses  $\tau_b$  experienced by a dislocation due to the repulsive barrier, as a function of out by the moving dislocation. The effec-



Fig. A-1 Schematic description of barriers to dislocation motion (After Nichols (1971))

tive shear stress causes the dislocation to reach position (b), thermal activation being required to allow further movement to position (c), whence continued forward motion is possible solely under the action of  $\tau_{eff}$  to the corresponding (b) position at the next barrier.

If the total energy required to overcome the barrier is  $\Delta F_0$  (considered as an activation enthalpy, the entropy of activation being ignored) then the amount required by thermal activation for forward dislocation motion is:

$$\Delta F_{f} = \Delta F_{o} - b \qquad (A-5)$$

where  $A(\tau_b)$  is the width of the back stress curve,  $A(\tau_{eff})$ being defined as the activation area, i.e., the area swept out by the dislocation during the activation event. For movement in the backward direction:

$$\Delta F_{b} = \Delta F_{o} + b \int_{O}^{\tau} A d\tau_{b} \qquad (A-6)$$

corresponding to thermal activation from position (c) to (b), work being done against the effective stress.

An average velocity of the dislocations is thus:

$$V = 2V_{o} \exp^{-\Delta F_{o}/kT} \sinh \frac{b}{kT} \int_{0}^{T_{o}} A d\tau_{b}$$
 (A-7)

At low stresses, this equation yields a linear velocity-effective stress dependence, whilst at large stresses, the backward rate becomes negligible and the velocity is obtained by substituting equation (A-5) into (A-4). At constant structure, it can be seen that the activation area can be expressed in terms of the effective stress dependence of the strain rate, and its magnitude should provide some indication of the type of obstacle to dislocation motion which is controlling the deformation, and thus of the rate controlling process.

Experimentally, the activation area may be determined by measuring  $\tau_{eff}$  over a range of strain rates, from the change of stress with time in a relaxation test, or by carrying out abrupt changes in either stress or strain rate to get  $\partial \ln \dot{\epsilon}/\partial \tau_{eff}$ . A is found to be almost independent of temperature, but approximately inversely proportional to stress, varying in magnitude from 3000 b<sup>2</sup> to 80 b<sup>2</sup> (Li, 1968). The three types of measurement will only agree provided that the mobile dislocation density does not change when the effective stress changes, and that the nature of the obstacles does not depend on strain rate. At high temperatures, the first two methods are unlikely to correspond to this situation.

The inverse stress dependence of the activation area implies a velocity-stress relation:

$$V = B (\tau_{eff})^{m*}$$
 (A-8)

where B and m\* are independent of stress but may be functions of temperature.

Nichols (1971) has criticised the expression (A-6) on the grounds that backward movement of the dislocations requires thermal activation against the effective stress between barriers in addition to that required to surmount the barrier. Thus, in a system where frictional effects are neglected, so that dislocations move rapidly between barriers, the backward movement involves motion from a (b) position back to another (b) position (Figure A-1). The area swept out during the activation effect, in the reverse direction . includes that between barriers and Nichols' expression becomes:

$$\Delta F_{b} = \Delta F_{o} + b \int_{o}^{\tau_{eff}} A' d\tau_{b} \qquad (A-9)$$

where  $A'(\tau_{eff})$  is the corresponding activation area. It is apparent that the activation area in the forward direction is much more stress dependent than that in the reverse direction, and that the precise form of the velocity-stress relation no longer corresponds to that of Li's analysis. For example, the constants B and m\* in equation (A-8) cannot in Nichols'

It appears that depending on the -

level of the effective stress, linear, power law and exponential dependencies will result irrespective of the rate controlling mechanism. Thus, the different regimes of stress dependence of strain rate cannot be conclusively taken as an indication of a change in the mode of dislocation motion.

The most commonly employed method of measuring the internal stress at high temperatures is that involving, socalled "dip" tests. These involve straining at constant strain rate to a given stress level (or to the steady state) and then suddenly adjusting the cross-head position to reduce the stress. The form of the load transient observed at constant total strain immediately following this reduction is taken as an indication of the level of mean internal stress. Theorétically, if the load were reduced instantaneously to that corresponding to the internal stress, then the strain "rate will go to zero and there will be no observable transient. After a reduction to a value above the internal stress forward straining will continue and lead to a relaxation in stress and a decreasing load transient. A reduction below the internal stress reverses the sense of flow and yields an increasing load transient, as the elastic strain component increases. In each case, the internal stress eventually begins to decline due to relaxation processes, so that the load decreases. Thus, only the first few seconds of the

transient can be monitored and several stress reductions may be needed before an accurate enough estimate of the internal stress can be made. This generally restricts the method to measurements of the steady state values where the same structure can be re-established following each "dip". For reasonably accurate estimates, a closed loop type testing machine is essential. A similar procedure can be followed whereby the strain transients following a load reduction in a creep test can be monitored.

# APPENDIX B

### PROBLEMS ASSOCIATED WITH THE USE

### OF FOILS FOR TRANSMISSION ELECTRON MICROSCOPY

## B-1. Foil Preparation

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During foil preparation dislocations may be both lost or rearranged due to the relaxation of long range stresses, or introduced by mechanical damage. The localised rearrangements are usually only a major problem where the fine scale features of isolated dislocations are being studied, the general character of the substructure being unaltered.

A more serious problem in the kind of work described in Chapters 5 and 7 was the loss of dislocations immediately adjacent to the perforation, that is, in the thinnest areas of the foil. For a distance of roughly 2 microns from the edge, the dislocation density was practically zero and a reduced density often persisted for about 5 microns in all. The well developed sub-boundaries and networks withstood this removal much better than did those within subgrains, so that the substructure tended to look considerably "cleaner" near the edge.

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the observations as much as possible to foil area beyond the affected region.

The accidental introduction of dislocations by mechanical damage or rough handling during foil preparation and location in the microscope was found to be easily achieved. Spark erosion damage was not anticipated since the slices so-prepared were greater than 1 mm although in copper, such damage has been reported to penetrate up to 0.3 mm.

The mechanical smoothing of the spark-cut slices may, however, also introduce some dislocations. Severe abrasion to 0.25 mm was tried at one point to reduce the time involved in chemically polishing the slices. This was found to introduce an extensive "fatigue-like" cell structure of high density (Photograph B-1). Although this arrangement was easily distinguished from the creep substructure, being parallel to the foil surface it suffered even more from loss during thinning. The creep subboundaries were mostly inclined to the foil and thus were "locked" in to a greater extent. Thus, in regions where the creep structure was normally looked at, the "fatigue" structure had been partially removed, the remnants being coarser and more intermittant and while still more ragged than the normal substructure, care must be



Photograph B-1. Electron micrograph of annealed copper severely abraided during preparation (X 10,000)



Photograph B-2. Electron micrograph taken near the edge of rely

、 196 taken to avoid confusion (Photograph B-2). The easiest distinguishing characteristic lies in the fact that there is virtually no misorientation across the "fatigue" cells. The copper discs were therefore subjected only to a light smoothing on 600 grade emery to remove the spark machine surface roughness, after which they were chemically thinned.

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# B-2. Damage to the Foil During Examination

The structure of a foil can be changed during examination in the electron microscope by both the heating and irradiation effects of the electron beam and by surface contamination by hydrocarbon vapours. The contamination, which otherwise leads to a gradual loss of intensity transmitted through the foil, can be controlled by the use of a "cold finger" which can easily be inserted into the microscope column adjacent to the specimen holder and which helps considerably in preserving the foil for prolonged examination. This can, however, lead to a more subtle and perhaps misleading artifact resulting from irradiation effects. Numerous small black spots were observed on micrographs taken in areas of foil which had undergone same feature can be detected in published micrographs in the literature, not only in creep, but also in tensile deformation and fatigue.

Detailed examination of these spots showed that when the foil was set for the exact Bragg diffracting condition the characteristic contrast was found to be a black-white lobe shape (Photograph B-3). In dark field similar contrast was observed, the sense of the black-white image remaining the same (Photograph B-4). It has been shown (McIntyre and Brown, 1966) that this is characteristic of defects at the top surface of the foil only. Careful examination of several specimens with over a hundred defects provided no exception to this observation.

Although there was a great variation in the density of defects observed from grain to grain, in all cases the density increased with time (Photographs B-5, B-6). However, the size of an individual defect did not appear to vary (points A in Photographs B-5, B-6). The variation of the orientation of the line separating the black and white lobes showed that the defects did not have a spherically symmetrical strain field, and is consistent with the defects being dislocation loops (L.M. Brown, 1971). No attempt was made to establish whether the defects were vacancy or interstitial in nature.



Photograph B-3. Bright field micrograph of annealed copper (X 50,000)



Photograph B-4. Dark field micrograph of the same area as (X 50,000)


Photograph B-5. Bright field micrograph of annealed copper - left 15 minutes in the electron beam (X 50,000)



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B-6. Bright field micrograph of the same area as

Pashley and Presland (1961) showed that ions, emitted from a coated filament, or generated at the grain, could produce damage in thin foils of gold. Howe et al (1963) also observed damage in copper foils. These results were reported by Hirsch et al (1965). While these defects can be ignored in examination of dislocation structures, the increasing use of anti-contamination devices, permitting long-time examination of the same foil area can result in high ion dosages and is consequently increasingly likely to be observed.

The heating effect in thin foils is manifested by the development of slip traces resulting from dislocations moving due to the stresses set up by the electron beam. Such a trace is seen in Photograph 7-6 near point A. In copper, this motion results in the visible part of the slip plane becoming uniformly lighter or darker than the background, and the traces apparently persist indefinitely. The contrast results from the inability of the dislocation to leave the foil due to the impedance of either a contaminating film or a pre-existing surface (oxide) layer. Such traces are readily visible and can easily be discounted, though occasionally a nuisance if the dislocation is one of a group being studied.

## B-3. Errors in the Measurement of Dislocation Density and Subgrain Size

In a material within which dislocations are uniformly distributed, the most serious error probably arises from loss of dislocations from the foil during preparation, or their introduction by mechanical damage. There are, however, other aspects which may lead to significant mismeasurement.

Firstly, the estimation of the foil thickness, needed when using an intercept method, may be difficult and subject to significant error (if the foil thickness were in error by one extinction distance in a value of 6, there would be an automatic error of 15% or so). Secondly, there is a human error involved in the choice of magnification at which the measurement is made. Ideally, this should be such as to clearly resolve individual dislocations over the range of densities being monitored, and the magnification adjusted accordingly. At high densities there is a problem in magnifying every dislocation allowed by a particular lattice reflection. If there is any degree of inhomogeneity, then the high magnification required permits only very small areas to be dealt with and large number of counts become necessary. The line length or grid must be adjusted along with the magnification.

As an example, at X10,000 the lowest density which could be measured with a suitable grid (i.e., that giving 1 intercept) was about  $10^6/\text{cm}^2$ . If the magnification were much lower the identification of individual dislocations becomes difficult. At densities greater than about  $10^9/\text{cm}^2$ it would also be hard to clearly resolve the dislocations.

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There is an additional error due to the fact that some dislocations are out of contrast at practically any given reflection. Hirsch and Steeds (1964) estimate that these may be up to 50% of the total. No correction has been applied to the results in this thesis, because the measurement is considered only to indicate the order of magnitude, but for critical work a correction would have to be applied.

The measurement of subgrain size is basically similar to that of normal grain size, with corresponding statistical and geometrical errors. The most serious difficulty in such a measurement is clearly identifying the individual subgrains. In a material showing wide variations in subgrain size a magnification must be chosen which gives fields of view which include a reasonable number of small and large subgrains. Frequently, the small subboundaries within clearly imaged subgrains especially when inclined to the foil at a smallish angle are difficult to

distinguish from areas of locally high dislocation density within subgrains. They sometimes also run out of the foil or are apparently partly removed during preparation. In the micrographs, this shows up as sub-boundaries which do not lead anywhere, but simply end in the middle of a subgrain. The identification of subgrains thus becomes very subjective, particularly near the edge of many micrographs where the focus and contrast may be less sharp.

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## REFERENCES

Ahlquist, C.N., and Nix, W.D., Actal. Met., 19, 373 (1971) Andrade, E.N. da C., Proc. Roy. Soć. A84 (1910) Balasubramanian, N., and Li, J.C.M., J. Mat. Sci., <u>5</u>, 434 (1970) Barrett, C.R., Acta. Met., 13, 1088 (1965) Barrett, C.R., Ahlquist, C.N., and Nix, W.D., Met. Sci. J., 4, 41 (1970) Barrett, C.R., and Nix, W.D., Acta. Met. 13, 1247 (1965) Barrett, C.R., Nix, W.D., and Sherby, O.D., Trans. ASM, 59, 3 (1956) Barrett, C.R., and Sherby, O.D., Trans. AIME, 230, 1322 (1964) Bird, J.E., Mukherjee, A.K., and Dorn, J.F., Proc. Haifa Conf. (1969) Boettner, R.C., and Robertson, W.D., Trans. AIME, 221, 613 (1961) Brown, L.M., Electron Microscopy in Materials Science Ed U. Valdre (Academic Press) p. 360 (1971) Cannon, W.R., and Sherby, O.D., Met. Trans., 1, 1030, (1970) Challenger, K.D., and Moteff, J., Met. Trans. 4, 749 (1973) Clauer, A.H., Wilcox, B.A., and Hirth, J.P., Acta. Met. 18, 381 (1970) Cottrell, A.H., and Aytekin, V., J.I.M., 77, 389 (1950) Cottrell, A.H., and Jaswon, M.A., Proc. Roy. Soc., A199, 104 (1949) Daily, S., and Ahlquist, C.N., Scripta Met., 6, 95 (1972) Davies, P.H., and Dennison, J.P., J.I.M., <u>90</u>, 53 (1951) Evans, H.E., and Williams, K.R., Phil. Mag., 26, 1399, (1972) Evans, W.J., and Wilshire, B., Métal Sci. J., 4, 89 (1970) Met. Trans., 1, 2133 (1970) Exell, S.F., and Marrington, D.H., Phil. Mag., 26, 1121 (1972) Friedel, J., Dislocations, Pergamon Press, London (1954) Fuchs, A., and Ilschner B., Acta. Met., 17, 701 (1989) Fullman R.L., Trans. AIME, 197, 447 (1953)

Garofalo, F., Fundamentals of Creep and Creep-Rupture in Metals (MacMillan Series in Materials Science), 1965

Gasca-Neri, R., Ahlquist, C.N., and Nix, W.D., Acta Met., <u>18</u>, 655, 663, (1970)

Gibbs, G.B., Phil. Mag., <u>23</u>, 771 (1971)

Gittus, J.H., Phil. Mag., 23, 1281 (1971)

Gupta, I., and Li, J.C.M., Met. Trans., <u>1</u>, 2323 (1970)

Gupta, I., and Strutt, P.R., Canad. J. Phys., <u>45</u>, 1213 (1967)

Hansen, M., "Constitution of Binary Alloys", McGraw-Hill (1958)

Hartley, H.O., Technometrics, <u>3</u>, 269 (1961)

Hasegawa, T., Sato, H. and Karashima, S., Trans. Japan Inst. Metals, <u>11</u>, 231 (1970)

Hirsch, P.B., Kowie, A., Nicholson, R.B., Pashley, D.W., and Whelan, M.J., "Electron Microscopy of Thin Crystals", London Butterworths 1965

Kolt, D.L., J. App. Phys., <u>41</u>, 3197 (1970)

Koriuchi, R., Yoshinaga, H., and Hama, S., Trans. Japan Inst. Metals, 6, 123 (1965)

Howe, L.M., Gilbert, R.W., and Piercy, G.R., App. Phys. Letter, <u>3</u>, 125 (1953) Immarigeon J-P.A., and Jonas, J.J., Acta Met., <u>19</u>, 1053 (1971)

Johnston, W.G., J. App. Phys., <u>33</u>, 2716 (1962)

Jones, B.L., and Sellars, C.M., Net. Sci. J., 4, 96, (1970)

Karashima, S., Likubo, T., Watanabe, T., and Oikawa, H., Trans. Japan Inst. Metals, <u>12</u>, 369 (1971)

Keh, A.S., and Keissman S., Electron Microscopy and Strength of Crystals, J. Wiley, New York (1963)

Koster, H., Z. Metallkde, 39, 1, (1948)

Lagneborg, R., Metal. Sci. J., <u>3</u> 18 (1959a)

Lagneborg, R., Hetal Sci. J., 3, 161 (1959b)

Lagneborg, R., Int. Met. Rev., 17, 130 (1972)

Lagneborg, R., Forsén, B.H., and Hiberg, J., Proc. of the Conf. on th in Steels and High Temperature Alloys" Sheffield

Lawley, A., Coll, J.A., and Cahn, R.W., Trans. AIME, 218, 166 (1960) Li, J.C.M., J. App. Phys., <u>33</u>, 2958 (1962) Li, J.C.M., 'Recrystallization, Grain-growth and Textures', A.S.M. 1966 Li, J.C.M., Dislocation Dynamics, edited by Rosenfield, A.R., Hahn, G.T., Benet, A.L., and Jaffee, R.I., (McGraw Hill Books Co.) 1968 Lloyd, D.J., and Embury, J.D., Metal Sci. J., 4, 6 (1970) Lloyd, D.J., Worthington, P.J., and Embury, J.D., Phil. Mag. 22, 1147, (1970) Lubahn, J.D., and Felgar, R.P., "Plasticity and Creep of Metals", (J. Wiley) 1961) Lytton, J.L., Barrett, C.R., and Sherby, O.D., Trans. AIME, (233, 1399 (1965) McIntyre K.G., and Brown, L.M., J. Physics, <u>27</u>, C3 178 (1966) McLean, D., Report on Progress in Physics, 29, 1 (1966) McLean, D., High Temperature Materials: The Controlling Physical Process, Edited by Kennedy, A.J., (Oliver and Boyd) 1968 Mecking, H., and Lucke, K., Scripta Met., <u>4</u>, 427 (1970) Mitra, S.K., and McLean, D., Metal Sci. J., 1, 192 (1967) Morris, C.E., Metal Progress, 56, 696 (1949) Mukherjee, A.K., Bird, J.E., and Dorn, J.E., Trans. ASM, <u>62</u>, 155 (1969) Myshlyaev, M.M., Olevskii, S.S., and Maksimov, S.K., Phys. Stat. Sol. (a) <u>15</u>, 391 (1973) Nemy, A.S., and Rhines, F.N., Trans. AIME, 215, 992 (1959) Nichols, F.A., Hater. Sci. Eng., <u>8</u>, 108 (1971) Oden, A., Lind, E., and Lagneborg, R., Proc. of the Conf. on 'Creep Strength in Steels and High Temperature Alloys', Sheffield 1972 Oikawa, H. Iikubo, I. and Karashima, S., Int. Conf. on "The Science and Technology of Iron and Steel" Tokyo, Sept. 1970 Orlova, A., and Cadek, J., Phil. Mag., 21, 509 (1970) Orlova, A., Pahutova, M., and Cadek, J., Phil. Wag. <u>23</u>, 303 (1971) A., Pahutova, N., and Cadek, J., Phil. Mag. 25, 865 (1972)

Pahutova, M. Cadek, J., and Rys, P., Phil. Mag. 23, 509 (1971) Pahutova, M., Hostinsky, T., and Cadek, J., Acta Met., 20, 693 (1972) Pashley, D.W., and Presland, A.E.B., Proceedings European Conf. on Electron Microscopy, Delft 417, 429 (1960) Perry, A.J., J. Mat. Sci., 9, 1016 (1974) · Pickering, H.W., Acta Met., 13, 437 (1965) Raymond, L., and Dorn, J.E., Trans. AIME, 230, 560 (1964) Reppich, B., J. Mat. Sci., 6, 267 (1971) Sargent, C., Private Communication (1973) Schmatz, D.J., and Bush R.H., Acta Met., 16, 207 (1968) Sherby, O.D., and Burke, P.M., Progr. Mat. Sci., 13, 325 (1987) Sherby, O.D., Trozera, T.A., and Dorn, J.E., Proc. ASTM, 56, 789 (1956) Solomon, A.A., Ahlquist, C.N., and Nix, W.D., Scripta Met., 4, 231 (1970) Solomon, A.A., and Nix, W.D., Acta Met., 18, 863 (1970) Staker, M.R., and Kolt, D.L., Acta Met., 20, 569 (1972) Steeds, J.N., Proc. Roy. Soc., A292, 343 (1966) Steeds, J.W. and Hirsch, P.B., Relation Between the Structure and banical Properties of Metals, N.P.L. Symp. No. 15, p. 39 London, KMSO (1964) Stevens, R.N., Met. Rev., 11, 129 (1966) Hatanabe, T. and Karashima, S., Trans. Japan Inst. Metals, 11, 159 (1970) Katt, D.F., Ph.D. Thesis, McMaster University 1967 Keertman, J., J. App. Phys., 28, 362, 1185 (1957) Veertman, J. Trans. ASM, 61, 681 (1958)