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EXPERIMENTAL MEASUREMENT AND MICROMECHANICAL MODELLING
OF THE CREEP DEFORMATION BEHAVIOUR OF AISI TYPE
310 STAINLESS STEEL AND PURE POLYCRYSTALLINE MAGNESIUM

by

Longquan Shi

A Dissertation
Submitted to the Faculty of Graduate Studies and Research
Through the Engineering Materials Program of
the Department of Mechanical Engineering
in Partial Fulfillment of the Requirements for
the Degree of Doctor of Philosophy at the
University of Windsor

Windsor, Ontario, Canada
1994
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ABSTRACT

Experimental creep measurements on an AISI type 310 stainless steel and high purity polycrystalline magnesium have been conducted within the test temperature and applied stress ranges of 650 °C to 800 °C and 110 MPa to 320 MPa for the 310 stainless steel, and 150 °C to 250 °C and 20 MPa to 50 MPa for pure magnesium. The results show that: (i) the creep strain rate ratio, F, between the initial creep rate upon loading and steady-state creep rate decreases from a high value to about unity when the test temperature and/or applied stress are increased; (ii) the apparent activation energy for creep, $Q_c$, upon loading is lower and is closer to the activation energy for pipe diffusion, $Q_p$, than the value at steady-state which is of the order of the activation energy for volume diffusion, $Q_v$; and (iii) the initial creep rate is less strongly dependent upon the applied stress than the steady-state creep rate.

The experimental results are discussed in terms of dislocation network models for creep and the internal and effective stresses. The analysis indicates that the high value for $F$ at low temperatures and/or low stresses is in agreement with the predictions of exhaustion theories where multiplication is not taking place and easily-moved dislocations are eventually exhausted. The low value measured for the apparent activation energy for creep upon loading (creep strain < $10^{-4}$) can be attributed to the contribution of pipe diffusion due to the large effective stress present at the onset of creep. The stress dependence of the initial and steady-state creep rates is a consequence of a change in both internal and effective stress during creep deformation, and the stress dependence of the initial strain rate offers an indirect measurement for investigating the stress dependence of the dislocation velocity in creep tests.
Creep and stress relaxation test results for pure magnesium show that the strain-hardening coefficient, $H$, during steady-state creep remains essentially constant with a magnitude of $0.27E$ ($E$ is the elastic modulus) at 200 °C and is independent of the applied stress. Examination of the experimental results lends support to the theoretical models based on dislocation link length distribution (dislocation network) models for recovery creep. This analysis gives the stress dependence of the steady-state creep rate, $\dot{\varepsilon}_s$, the dislocation annihilation rate, $\dot{\rho}_s$, the average effective dislocation velocity, $v$, the recovery rate, $R$, and the strain-hardening coefficient, $H$, respectively, during high temperature recovery creep of crystalline materials.

Micromechanical modelling was carried out for three aspects of creep, namely (i) the grain size effect in the creep and superplastic deformation of polycrystalline materials; (ii) simulation of the experimental creep results for a 310 stainless steel using the Öström-Lagneborg creep model; and (iii) a dislocation link length statistics model for the plastic deformation of crystalline materials. It is shown that: (i) during superplastic deformation, a solid polycrystalline material can be visualized as a two "phase" mixture, one flowing according to Newtonian viscous flow and the other deforming by power law creep; (ii) the simulation results for the 310 stainless steel obtained using the Öström-Lagneborg creep model are in good agreement with the experimental results and other independent studies on dislocation network models for creep; and (iii) the dislocation link length statistics model produces reasonable values for the strain-hardening coefficient and the recovery rate during a constant stress creep test, and for a strain rate change during a constant strain rate tensile test.
DEDICATION

To my parents.
my wife Lanying Ouyang.
and my son David.
ACKNOWLEDGEMENTS

The author of this dissertation wishes to thank Dr. Derek O. Northwood, for guidance from the onset to the completion of this study, and for both academic and non-academic advice during his study at Windsor.

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LIST OF SYMBOLS

1. English Letters:

A a dimensionless materials constant (A₁ and A₂ are numerical constants);
Aᵢ an area swept out by the i-th dislocation link;
Aᵦ a constant;
Aₑ a structure-dependent parameter;
A the activation area for dislocation glide processes;
BCC body-centred cubic crystal structure;
b the Burgers vector;
C(t) the probability function during dislocation link collision;
CMC ceramic matrix composites;
c (i) the dislocation network geometry factor; (ii) the local equilibrium vacancy concentration;
D (i) an appropriate diffusion coefficient for different processes (Dₖ for grain boundary diffusion; Dₚ for dislocation pipe diffusion; Dₚh for grain boundary phase diffusion; Dᵥ for lattice (or volume) self-diffusion);
D (ii) grain size (Dₑ for critical grain size; ȳ for the average grain size; ȳ for the normalized grain size (ȳ = D/me)); ȳ for the normalized geometric mean (ȳ = 0.931);
DS dispersion strengthened (alloys);
Dₒ a diffusion frequency factor;
d (i) grain size (diameter); (ii) an average particle size;
E the elastic modulus or Young’s modulus;
Eₐ the activation energy for dislocation detachment process;
exp the base of natural logarithms (exp = 2.7183);
F creep rate ratio between the initial and steady-state creep rates;
FCC face-centred cubic crystal structure;
f(D) a frequency function for grain size distribution;
f_p a crystallographic orientation factor (f_p = 0 to 1);
H strain-hardening coefficient;
H(x) the Heaviside unit function (H(x) = 0 or 1 when x < 0 or ≥ 0);
HCP hexagonal closed packed crystal structure;
ΔH activation entropy;
h a constant (h = 1/λₑ);
lᵥ the volume integral constant;
i, j integer numerical numbers;
Kₚ the power law creep constant;
k (i) Boltzmann’s constant (k = 1.381 · 10⁻²³ J/K or 8.620 · 10⁻⁵ eV/K); (ii) an integer numerical number;
k₁ the multiplication rate constant for the dislocation kinetics;
k_R a relaxation parameter;
kₑ a proportionality constant (kₑ = 0 to 1);
k(t) the network geometry function;
L (i) the obstacle spacing during dislocation glide; (ii) a stress dependent factor;
lim the limit sign;
ln the natural logarithm sign;
ℓ the internal stress exponent for the mobile dislocation density;
M the Taylor factor (M = 3.1 for cubic crystals, M = 2 for preferential glide on slip systems where the resolved shear stress attains its maximum value);
MMC metal matrix composites;
M' the mobility of dislocations;
m (i) the stress exponent for recovery rate; (ii) the strain rate sensitivity;
N (i) a stress exponent; (ii) the total number of dislocation links;
n (i) the strain rate sensitivity; (ii) the apparent stress exponent; (iii) an integer numerical number;
n_0 the stress exponent for recovery rate;
ODS oxide dispersion strengthened (alloys);
p (i) the exponent of the inverse grain size for steady-state creep rate; (ii) the effective stress exponent for average dislocation velocity;
Q the activation energy for diffusion;
Q_0 an activation energy term (Q_0 for the apparent activation energy for creep; Q_n for strain-hardening process; Q_r for recovery process);
Q_0 an energy term;
q a constant;
R (i) gas constant (R = 8.314 J/K * mole); (ii) dynamic recovery rate;
r (i) particle radius; (ii) the ratio of transient creep rate to the transient creep strain; (iii) average radius of circular dislocation loops; (iv) r_{Kc} f_0 * correlation coefficient;
S_x a subgrain parameter;
SFE stacking fault energy;
T the absolute temperature (T_c for the lowest temperature at which the threshold stress is zero; T_m for the absolute melting (or decomposition) temperature in degrees Kelvin);
TD throia dispersed (nickel alloys);
t creep time;
Δt incubation time for a stress decrement during steady-state creep;
u the normalized dislocation link length (u = <\ell>/\ell_0);
u_{th} the normalized threshold link length (u_{th} = \ell_{th}/\ell_0);
V_c the volume fraction of coarse grains;
V_f the volume fraction of fine grains (V_c + V_f = 1);
V_0 a constant;
V the activation volume for dislocation glide processes;
\dot{\gamma} the average effective dislocation velocity;
x a variable related to dislocation geometry and distribution;
y a function for strain rate sensitivity;
Z (i) the Zener-Hollomon parameter; (ii) the ratio between mobile and total dislocation densities.
II. Greek Characters:

α (i) an average Schmid strain resolution factor; (ii) a parameter (α for dislocation interaction during its motion; α is a constant);
β a constant dependent on dislocation geometry and dislocation:
Γ line energy (tension) of the dislocation in the matrix;
Γ_p line energy (tension) of the dislocation at the particle interface:
Δ the increment or decrement operator;
∇^2 a Laplacian;
∂ the partial differential operator;
δ thickness (δ_0 for grain boundary; δ_ph for grain boundary phase);
ε creep strain (ε(t) for creep strain at a given time; ε_i for the instantaneous strain upon loading; ε_i for the strain limit for transient creep);
ε̇ the deformation strain rate and/or creep strain rate at time t (ε̇_i for the initial creep strain rate; ε̇_m for the reference strain rate; ε̇_s for the steady-state creep rate);
ε_0 frequency constant (ε_0 for initial creep rate; ε_0 for steady state creep rate);
η̇ the viscosity;
λ (i) the planar spacing between particles; (ii) the dislocation link length (λ_c for the critical link length; λ_m for the link length where φ attains its maximum value; λ_th for the threshold link length; <λ> for the average link length);
μ the shear modulus;
ν Possion’s ratio (ν = 1/3 for metals); (ν' and ν'' for thermal activation frequencies);
Π the product operator;
ρ dislocation densities (ρ_i for the immobile dislocation density; ρ_m for the mobile dislocation density; ρ_o for the initial dislocation density; ρ_i for the total dislocation density);
ρ_a dislocation annihilation rate;
Σ the summation operator;
σ the applied uniaxial tensile stress (σ for the average effective stress; σ_i for the average internal stress; σ_o for the threshold stress; σ_p for the applied-stress-independent average resisting stress due to particles; σ_σ for the resisting stress due to the matrix solid solution phase);
σ̄ the shear modulus normalized applied stress (σ̄_eq for the normalized transition stress; σ̄_o for the normalized internal stress);
σ_g the geometric standard deviation in grain size distribution (σ_g = 1.460);
τ the shear stress (τ_c for the local climb stress; τ_d for athermal detachment stress; τ_e for the average effective stress; τ_i for the average internal stress; τ_n for the Orowan stress or creep threshold stress);
ϕ(λ, t) the dislocation link length distribution frequency function;
ϕ_0 the initial dislocation link length distribution frequency function;
Φ(u) the universal dislocation link length distribution frequency function;
χ a stress enhancement factor;
ψ a constant dependent on the dislocation distribution;
Ω the atomic volume.
CHAPTER 1 INTRODUCTION

Creep, together with fatigue and static fracture, are important from both economic and academic points of view. Creep is one of a number of fields where improvements would lead to the possibility of rapid technological advance, in terms of a more reliable and a safer performance, or the attainment of higher energy efficiency. There is a demonstrated need for the development and improvement of materials for various engineering applications at elevated temperatures, and engineers and scientists worldwide are devoting their time and energy to the study and solution of creep-related matters.

Generally, the creep process is described in terms of a strain-time relationship (both the applied stress and test temperature are kept constant during a creep experiment). There are three stages in strain-time behaviour in the creep of metals and alloys, namely (i) a primary creep stage where the strain is increasing with time, and the rate is decreasing with time (or in some cases increasing); (ii) a secondary stage where the strain is increasing with time at a constant rate (steady-state creep); and (iii) a tertiary stage where the creep is accelerating until fracture [1-3]. For polycrystalline materials creep rupture is generally associated with void formation and the coalescence of microcracks within grains or at grain boundaries.

Since creep is a slow deformation process, long term tests must be conducted on selected samples before an improved mechanical design is practically attainable and a component is assembled in an operating machine. It is for this reason that the steady-state creep stage, and its connection to final fracture, as well as the behaviour when the applied stress is varying and environmental effects come into play (creep-fatigue interactions and
creep-corrosion interactions) are emphasized. It is therefore clear that the creep performance of an engineering material is determined by both the material's properties and its operating environment. Such technical (creep) data can be obtained in a number of ways, such as (i) in a long term laboratory test under a variety of simulated environments; or (ii) a statistical study of an operating component; or (iii) the observation of a natural phenomenon. This could involve measuring the specimen length under a sustained load at elevated temperatures, or recording the creep life time of identical components made from different materials in a machine, or studying geographic maps drawn at different periods of time. The creep properties of a material also depend on its operating environment, and may differ markedly from those properties determined in vacuum (they can be either worse or better depending on the material and its environment and the interaction between them).

It was not possible to conduct the required long period testing in our laboratory, where typically a large number of creep-test setups are operating simultaneously and often higher applied stresses and test temperatures (than in practice) are used to accelerate creep deformation. However, over the years the data in the literature has shown that all the three stages of creep deformation are interrelated, and information on the early stages, i.e. the primary and secondary stages, can be used to predict the final fracture properties [4-6]. As will be discussed in the literature review section, studies on creep of pure metals and solid solution alloys can provide an important understanding of creep mechanisms, and provide important "clues" to high temperature alloy design, since the alloy matrix plays a major role in determining the creep resistance of both dispersion strengthened
(DS) alloys and metal matrix composites (MMCs).

Creep studies attain increasing importance as operating temperatures increased for energy efficiencies. Much of the creep work was concentrated on empirical studies and materials characterization. Creep data has been measured for a wide range of engineering materials over a period of about one century. However, the creep mechanism studies have lagged far behind the advances in the empirical studies and the technological development of high temperature alloys [7-12]. The development of high temperature alloys has, in the main, been the result of painstaking empirical investigation, and it is really only in retrospect that principles underlying these developments have become gradually evident [1]. For example the empirical recovery creep equation in its present form was obtained some two decades ago but until very recently there was no satisfactory physical mechanism explanation. Often a mechanistic study can provide information useful in technological development, and instances of this can be found in many science and engineering disciplines. It is therefore the aim of the work described in this dissertation to study some selected aspects of recovery creep both by making experimental measurements on a pure metal and an alloy, and by analyzing the creep results using a statistical treatment of the dislocation network models for recovery creep deformation of crystalline materials.

The creep tests and micromechanical analyses are concentrated on the primary and secondary creep stages. The materials used in this study were an AISI type 310 stainless steel and high purity polycrystalline magnesium. They represent a Class II alloy and a pure metal in the creep material classification of Sherby and Burke [7]. Several aspects
of creep behaviour are discussed and analyzed. These include: (i) the apparent activation energy for creep; (ii) the stress dependence of initial and steady-state creep rate; (iii) the stress dependence of strain-hardening coefficient and recovery rate during steady-state creep; and (iv) micromechanical modelling of the superplastic deformation of polycrystalline materials, the Öström-Lagneborg creep model, and the dislocation link length statistics for plastic deformation of crystalline materials. The first three aspects are related to the dependence of creep behaviour on test conditions, namely temperature and applied stress, which are of technological importance. This will be discussed in Chapter IV. The last aspect is concerned with the micromechanical mechanisms of, and the latest statistical treatment of, the creep of crystalline materials at elevated temperatures. This constitutes the subject matter of Chapter V. The role of the alloy matrix in determining creep resistance, and the statistical treatment of the creep deformation are two important aspects of the current main stream in creep studies. In Chapter V, superplastic deformation of polycrystalline materials is also briefly discussed. The connection between creep and superplastic deformation for polycrystalline materials is of importance from the technological point of view for elevated temperature deformation and materials shaping.

For the convenience of the reader, this dissertation employs the following presentation format. Each section of the main chapters begins with a brief introduction to that subject, reports on the experimental results or model calculations, discusses these results and concludes with a brief summary. Chapter VI summarises the main conclusions from this study, and the dissertation concludes with recommendations for further work.
CHAPTER II REVIEW OF THE LITERATURE AND OBJECTIVES OF PRESENT STUDY

§1. Literature Review

A brief overview of creep will first be given concentrating on the general aspects and technical importance of the phenomenon and illustrating this by examples of industrial applications and natural deformation phenomena involving creep. The development of superalloys for gas turbine engine [8-10], the sintering and hot isostatic pressing (HIP) of powder metallurgy (PM) compacts [11-15], and the modelling of the movement of geological formations (e.g. icebergs and the earth mantle) are all dependent on a knowledge of creep [16, 17]). Considerable data has been compiled on the general aspects of creep deformation [16-19], and on the creep properties of various metals, alloys, intermetallic compounds, carbides, oxides, nitrides, and other ceramics [20-24], although the mechanisms of creep still need to be further studied and improvements made in mechanistic modelling. Despite their obvious disadvantages at high temperatures such as high vapour pressure, lack of oxidation resistance, and lack of resistance to nitrogen (in air) and possibly to carbon and to sulphur (in chemical plant applications), metals are the principal materials used to carry tensile stress at high temperatures, while ceramic materials are excellent for high temperature applications to support compressive stress [25]. Various surface coating techniques have, however, been developed to improve this situation to some extent with respect to aggressive creep environments [26]. Thus, this review will mainly discuss creep of metallic materials, although other materials will also be referred to for comparative proposes.
1.1. General Aspects of Creep

This section briefly introduces some relevant experimental creep techniques and definitions. A creep test is used to measure the creep rate, $\dot{\varepsilon} = \frac{d\varepsilon}{dt}$, and the time to fracture, $t_f$, where $\varepsilon$ is creep strain, and $t$ is the creep time. In terms of the creep curve, i.e. $\varepsilon$ versus $t$ curve, the creep rate, $\dot{\varepsilon}$, is the slope of this curve. The initial creep rate, $\dot{\varepsilon}_i$, is the creep rate at the onset of creep, i.e. $\dot{\varepsilon}_i = \frac{d\varepsilon}{dt}|_{t=0}$, or the initial slope of $\varepsilon$ versus $t$ curve. The steady state creep rate, $\dot{\varepsilon}_s$, is the creep rate when the slope of $\varepsilon$ versus $t$ curve is a constant. An actual creep curve of pure magnesium is given in Fig. 1 for a creep test at 150 °C and 35 MPa, where $\dot{\varepsilon}_i$ and $\dot{\varepsilon}_s$ are given by the slopes of dotted line and by dashed line, respectively. It is seen from Fig. 1 that $\dot{\varepsilon}$ decreases with time from $\dot{\varepsilon}_i$ to $\dot{\varepsilon}_s$. And this type of creep curve is called normal type (N-type) creep curve.

The stress dip test is carried out in the steady state creep stage and involves rapidly reducing the applied stress, $\sigma$, from the steady state value by an amount $-\Delta \sigma$ and then carefully measuring the strain rate immediately after the stress reduction. Immediately after a load reduction, the sample contracts elastically and subsequently deforms by plastic flow. For a small stress reduction the plastic strain rate, instantaneously after reducing the applied stress, is positive. However, after a large stress reduction the instantaneous strain rate is negative. At an intermediate level of stress reduction the plastic strain rate is momentarily zero. This level corresponds to the point at which the new reduced applied stress is equal to the mean internal stress, $\sigma_i$. After a short time recovery events reduce the internal stress giving a positive strain rate once again. The mean internal stress, $\sigma_i$, is the driving force for dislocation recovery. The mean
Fig. 1 A typical creep curve for pure magnesium crept at 150 °C and 35 MPa

(Instantaneous strain is subtracted).
Fig. 2 Schematic diagram illustrating the stress dip test for measurement of the average internal stress $\sigma_i$ and the average effective stress $\sigma_e$: (1) $\Delta \sigma_1$, (2) $\Delta \sigma_2$, (3) $\Delta \sigma_3$,

where $\Delta \sigma_1 < \Delta \sigma_2 < \Delta \sigma_3$, $\sigma = \text{applied stress}$, $\sigma_i = \sigma - \Delta \sigma_2$ and $\sigma_e = \sigma - \sigma_i = \Delta \sigma_2$. 
effective stress, $\sigma_e$, is given by $\sigma_e = \sigma - \sigma_i$, and is the driving force for dislocation glide [27, 28]. A schematic diagram shows different behaviours for various stress reductions, $\Delta \sigma$, Fig. 2.

The creep behaviour of a given material is in general, determined by three factors, namely (i) the material's intrinsic properties (atomic bonding, melting temperature, elastic modulus, diffusion coefficient; they are interrelated with each other); (ii) its microstructural features (such as chemical composition, phase microstructure, and grain size; these factors can be changed by the metallurgical processing); and (iii) the operating environment (temperature, applied stress and or strain, and test media such as gas or fluid types). Once a relationship is established between the creep behaviour and these material properties and environmental factors by means of a creep study, it can then be used as a guide to material selection, design and processing for a specific operating environment.

An improved creep resistance can be obtained by a proper combination of those material properties, which can be adjusted through a proper choice of materials, and an application of a variety of metallurgical processes, such as alloying additions, controlled casting, mechanical processing, heat treatment, and powder metallurgy, to optimize the microstructural features. From this accumulated data on alloy development and mechanical properties measurements, some general guidelines, the Honeycombe rules [29], have been put forward as an aid in the development of creep resistance in materials. These "rules" are as follows:

1. The creep resistance at a given temperature is higher in metals and alloys of high melting point, because a metal of high melting point has a lower rate of self-
diffusion at that temperature than a metal of lower melting point. In cases where power
law creep is important, as the rate of dislocation climb is proportional to the rate of self-
diffusion, this process will be more difficult in a metal of higher melting point. The
limiting temperature below which climb cannot occur readily (i.e. atomic self-diffusion
is negligible) is about 0.5T_m, where T_m is the melting (or decomposition) temperature in
degrees Kelvin. This relationship can be shown from a thermodynamic consideration of
the atomic self-diffusion process [18].

(2) Creep resistance is greater in a matrix of low stacking fault energy (SFE) (creep rate is proportional to the third power of the SFE [7, 30-32]), because the
dislocations are dissociated, and thus find it more difficult to cross-slip and to climb, in
order to avoid obstacles. The stacking fault energy of a pure metal can be lowered by
solute additions. For this propose solutes of high valency are best because they more
readily increase the electron to atom ratio, and thus decrease the stacking fault energy.
Fortunately, such solutes also tend to raise the flow stress more markedly than solutes of
lower valence.

(3) Solid solution hardening is a useful contribution. This is best achieved by use
of solutes differing markedly in atomic size and valency from the parent metal.
Unfortunately, these factors mitigate against extensive solid solubility.

(4) Long-range order in solid solutions provides a further contribution to the creep
strength of solid solutions, because the superlattice dislocations are paired to preserve
order across the slip plane, and are thus similar to extended dislocations.

(5) Precipitates are essential to further increase the creep strength of a solid
solution. The Orowan (precipitate strengthening) theory provides an estimate of the critical spacing of a dispersion for the optimum strength as that which is just small enough to prevent dislocations from bowing around the particles (approximately $10^{-8}$ m). Unfortunately, such fine dispersions are usually not stable at high temperature because rapid diffusion of the alloying elements through the matrix allows coarsening [33-37]. This can be minimized in several ways [36, 38], namely (a) choose elements in the precipitate which diffuse slowly (low diffusion coefficient); (b) use a dispersed phase which is practically insoluble in the matrix, so that the re-solution of fine particles and the growth of coarser particles is slow (low solid solubility); and (c) select a precipitate which is crystallographically closely matched to the matrix and so remains coherent longer (low interfacial energy).

(6) Use of precipitates in association with crystal defects. Some precipitates form more readily than others on dislocations, and thus are important sources of strengthening, both at low and elevated temperatures. Precipitates which form during creep are particularly useful if they nucleate on dislocations. Nucleation in association with stacking faults is another form of strengthening. Precipitation at grain boundaries is useful in reducing grain-boundary sliding, but in many cases this leads to early cavity formation and premature intergranular cracking. High creep strength is thus often achieved, but at the expense of creep ductility. It is possible that grain boundary precipitates with low energy interfaces with the matrix are less likely to cause intergranular failure.

As can be seen from preceding discussion, the numerous empirical studies have been utilized to make high temperature superalloys and other engineering materials a
reality. Due to its economic and academic importance, and a need for a wide range of structural materials for various engineering applications at high temperatures, creep is receiving increasing attention recently, as can be seen from the American Society for Metals (ASM) seminars on "Deformation, Processing, and Structure" [38], and "Flow and Fracture at Elevated Temperatures" [39], and the Institute of (Metals) Materials (the Swansea) creep conferences [40-42]. In spite of extensive studies, our understanding of creep is far from complete, and the creep properties of engineering materials can still be further improved. This is seen from the early development of the time law for creep which was based upon the shape of the creep strain-time curves [18, 43] to the recent creep models based on more detailed microstructure observations and micromechanical models [44, 45].

1.2. Creep of Metals and Alloys

Researchers have been concentrating in two major directions with respect to the creep mechanisms of metals and alloys, namely (i) the applied stress and test temperature dependence (i.e. test conditions); and (ii) the microstructure dependence (of materials properties) of steady-state creep rate. The creep behaviour of single-phase crystalline materials has been classified into two categories [7, 28, 44], namely Class I (n = 3) and Class II (n = 5), in terms of the stress exponent, n, of steady-state creep rate which characterizes the rate determining mechanism during creep deformation. The mechanism for Class I creep behaviour is a viscous glide of dislocations dragging a solute atmosphere along their glide path. This Class I behaviour has been fairly well modelled over the past four decades [41, 46-54], by considering diffusion associated with a moving potential
wall, drag of a Cottrell atmosphere or a Snoek atmosphere by a dislocation, and other solute effects on dislocation motion. The Class II creep is believed to be controlled by a recovery process (dislocation climb) carried out by vacancy diffusion. This view is supported by the experimental results which show that the activation energy for high temperature creep is equal to the activation energy for vacancy diffusion in the materials [55-62], and that for creep of various crystals, the activation volume for creep is similar to that for self-diffusion, indicating a creep mechanism involving vacancies [57]. Thus the temperature dependence of Class II creep rate should follow the Arrhenius relationship which contains a Boltzman factor [63-65]. The empirical constitutive equation has been established to describe the power law creep [59, 66, 67], although the recovery creep process has not yet been analyzed satisfactorily [22]. In order to model the recovery rate process during elevated temperature creep deformation, the following three factors should be taken into account, namely (i) the configuration of dislocations and the character of their motion; (ii) the forces acting upon dislocations; and (iii) the sources and sinks of vacancies.

In terms of microstructure, grain size is an important factor in polycrystals, such as alloys and ceramics. In polycrystals, creep is a far more complex phenomenon, since the onset of diffusion simultaneously makes a large number of mechanisms of deformation available in addition to dislocation glide and climb. Both lattice mechanisms and boundary mechanisms are operating during high temperature creep deformation. An examination of more than twenty mechanisms by Cannon and Langdon [23, 24] has led to an expression for the steady-state creep rate, $\dot{\varepsilon}$, of the form:
\[ \dot{\varepsilon}_s = \frac{AD\mu b}{kT} \left( \frac{b}{d} \right)^p \left( \frac{\sigma}{\mu} \right)^n \]  

where A is a dimensionless constant, D is the appropriate diffusion coefficient, \( \mu \) is the shear modulus, \( b \) is the Burgers vector, \( k \) is Boltzmann's constant, \( T \) is the absolute temperature, \( d \) is the grain size, \( p \) is the exponent of the inverse grain size, \( \sigma \) is the applied stress, and \( n \) is the stress exponent. The diffusion coefficient is given by:

\[ D = D_0 \exp \left( -\frac{Q}{kT} \right) \]  

where \( D_0 \) is a frequency factor, and \( Q \) is the activation energy for the appropriate diffusion process. For lattice mechanisms of creep, which are based on intragranular glide and climb of dislocations, \( p = 0 \), and in general the predicted value of \( n \) is within the rather limited range from 3 to 4.5 when the activation energy is equal to the value for lattice self-diffusion (or volume diffusion), \( Q_v \), although it is possible to obtain higher values of \( n \) at lower temperatures by invoking pipe diffusion with an activation energy of \( Q_p (\approx 0.6Q_v) \). For boundary mechanisms of creep, \( p \geq 1 \).

There are two grain boundary processes [68], namely (i) Lifshitz sliding [69] (grain boundary displacement associated with grain elongation); and (ii) Rachinger sliding [70] (grain boundary displacement not associated grain elongation). Examples of Lifshitz sliding are: (i) sliding accommodated by either lattice (or volume) or grain boundary diffusion -- Nabarro-Herring creep [71, 72] \((n = 1, p = 2, Q = Q_v)\) and Coble creep [73] \((n = 1, p = 3, Q = Q_{gh} \text{ or } Q_p)\); and (ii) sliding accommodated by intragranular flow
(diffusion) across the grains [74] \((n = 1, \ p = 1, \ Q = Q_{gb}\) or \(Q_{p}\)). Examples of Rachingers sliding are: (i) sliding with a continuous glassy phase at boundary [75] \((n = 1, \ p = 1, \ Q = Q_{gb}\)); and (ii) sliding without a glassy phase -- sliding accommodated by formation of grain boundary cavities [76] \((n = 2, \ p = 1, \ Q = Q_{c}\) and sliding accommodated by formation of triple-point folds [77] \((n = 3.5, \ p = 2, \ Q = Q_{s}\). In the above relationships, \(Q_{gb}\) is the activation energy for grain boundary diffusion, and \(Q_{p}\) is the activation energy for diffusion through grain boundary phase.

Analysis of a wide range of experimental data of the creep of ceramic materials has shown two major points [23, 24]. First, there are many similarities between the creep of ceramics and metals. These similarities include (i) diffusion creep with a stress exponent of \(n = 1\) at low stresses, and power law creep with \(n \geq 3\) at high stresses; (ii) an inverse relationship between the subgrain size and the applied stress, \(\sigma\); (iii) a dislocation density within the subgrains varying with \(\sigma^2\); and (iv) a contribution from grain boundary sliding (GBS) which increases with a decrease in stress and/or grain size. The major differences between the creep of ceramics and the creep of metals is the enhanced role of diffusion creep in ceramics because of small grain size and high lattice resistance which suppresses power law creep. The division of creep behaviour of ceramics into two categories in power law creep with stress exponents of \(\sim 5\) and \(\sim 3\), respectively. The behaviour with \(n = 5\) is interpreted as fully ductile behaviour as in pure face-centred cubic (FCC) metals, and the behaviour with \(n = 3\) is interpreted as dislocation climb from Bardeen-Herring sources (creation of dislocation rings from an edge dislocation which is fixed at two points and which has a slip plane normal to the extra plane of
atoms) [78, 79] under conditions where there is either a lack of five independent slip systems and general inhomogeneous deformation or, if five independent slip systems are available, a lack of interpenetration of these systems, in that dislocation cross slip is difficult. The obvious inference is that boundary mechanisms play a larger role in the creep of ceramics than of metals [24]. This would be in accord with the inherently lower mobility of dislocations in ceramics by the Peierls-Nabarro stress (the lattice resistance to dislocation glide), and with the differences in fabrication of metals and ceramics. These differences are likely to lead to thicker grain-boundary films in ceramics.

1.3. Creep of Dispersion Strengthened Alloys

A practical means of improving the (power law type) creep resistance of the alloy matrix is by the introducing a nondeformable second phase for high temperature alloys (such as dispersion strengthened (DS) alloys and (particulate reinforced) metal matrix composites (MMCs) [80-83]). It should be pointed out that the second phase particles (or reinforcements) are effective sources of creep strength only when they are thermally stable at elevated temperatures. In contrast to pure metals and single phase solid solution alloys, experimental results on dispersion strengthened (DS) alloys have shown two general characteristics, namely much higher apparent creep stress exponents, n, and activation energy, Q_c. For example, n values of 7 to 75 and apparent activation energies 2 to 3 times that for self-diffusion have been reported for precipitation and/or oxide-dispersion-strengthened (ODS) nickel-based alloys [83-86]. The activation energy for self-diffusion and that for creep of metals and other materials can be found in tabulated documents [22, 87-89].
There have been many studies on the creep mechanisms of dispersion strengthened (DS) alloys. Early studies have shown that the increased creep strength in these materials is caused by the obstacles resistance to the glide of edge dislocations which could be circumvented by climb [90], and by a decrease in the recovery rate, and this in turn is due to a decrease of the driving force for the recovery process and of the mobility of the climbing dislocations involved in the process [91]. The dislocation mechanism considered in the models is dislocation climb to bypass particles and then opposite sign dislocation pairs annihilate each other (recovery process). Only edge dislocations are considered in the models, because screw dislocations are able to move without vacancy diffusion. The steady-state creep rate will then be governed by the rate of dislocation climb and annihilation (recovery) process. Good agreement has been found by applying these models to dispersion strengthened (DS) alloys. These models can account for the very large stress (exponents) of the steady-state creep rate often found for alloys hardened by a nondeformable second phase, such as sintered aluminium powder (SAP) type alloys, Ni-Cr-Al alloys and an austenitic 20Cr-35Ni stainless steel containing ordered face-centred cubic (FCC) $\gamma'$-Ni$_3$(Al, Ti) precipitates. However, the high values found for the apparent activation energy have been left unanswered. Also, the type of interfaces between particle and matrix and the interaction between dislocations and interfaces are not considered in these early climb models.

Recent studies on DS alloys have utilized three threshold stresses for creep, namely (i) the stress required to cause dislocation bowing between particles (the Orowan stress), \( \tau_o = 0.84 \mu b(\lambda - d) \), where \( \lambda \) is the planar spacing between particles, and \( d \) is the
average particle size.) [92-94]: (ii) the extra back stress required to create the additional
dislocation line length as the dislocation segment climbs over a particle (the local climb
stress), \( \tau_n = 0.3\mu b/\lambda \) [95, 96]; and (iii) the stress required to detach the dislocation from
the particle after climb is completed in an attractive dislocation-particle interaction (the
athermal detachment stress), \( \tau_d = (\mu b/\lambda)(1 - k_R^2)^{1/2} \), where \( k_R \) is given by \( \Gamma_p = k_R \Gamma \) in the
attractive interaction model, with \( \Gamma = \mu b^2/2 \) and \( \Gamma_p \) are the line energies of the dislocation
in the matrix and at the particle interface, respectively. The parameter \( k_R \) (0 \( \leq k_R \leq 1 \)) can
be thought of as a relaxation parameter. For \( k_R = 1 \), no attraction between particle and
dislocation exists (no relaxation); for \( k_R < 1 \), an attractive interaction results, which
becomes stronger with diminishing \( k_R \) (increasing relaxation) [97-100].

Models that take into account the above three threshold stresses have led to the
following important findings: (a) The measured threshold stresses for creep in DS alloys
are less than the values predicted from the equation of the Orowan stress by a factor
ranging from 1.25 to 2.5 [95, 101]. More significantly, it was suggested [99] that the
concept of the Orowan stress may not be applicable to the deformation of DS alloys at
high temperatures and low stresses, since the moving dislocations can bypass hard
particles by climbing under these experimental conditions. (b) Deformation processes
based on dislocation climb alone cannot provide a satisfactory explanation for the creep
of DS alloys in terms of a threshold stress. Firstly, although models based on "local"
climb [95, 96] yield threshold stresses that are in good agreement with those estimated
from experimental data on DS alloys, "local" climb, if it occurred, would result in a sharp
dislocation curvature (at the point where the dislocation meets the particle) [102] that can
be rapidly relaxed by diffusion, leading to more "general" climb. Second, models based on "general" climb [102-104] predicted either an insignificant threshold stress or a threshold stress that is proportional to the applied stress. (c) The possibility that an attractive interaction between dislocations and particles exists is supported by both theoretical (Srolovitz’s attractive particle-dislocation interaction [105, 106]) and experimental studies [107-109]. For instance, theoretical studies by Srolovitz’s et al [105, 106] show that an attractive interaction between dislocations and incoherent particles would in fact be expected at elevated temperatures because the incoherent interface can relax parts of the dislocation’s stress field by slipping and rapid diffusion. And, transmission electron microscopy (TEM) observations [107-109] have revealed that the dislocations remain bound to the particles after climb has been completed. It has been demonstrated [97, 98] that such an attractive particle-dislocation interaction in DS alloy leads to a well-defined threshold stress for creep which must be exceeded in order to detach the dislocation from particle after climbing. Most recently, it has been suggested [99] that thermally activated dislocation detachment from the dispersion particle is the rate controlling mechanism in DS alloys and that the climb process can be regarded as sufficiently rapid. In this case, creep may occur below the athermal "detachment stress".

Circumstantial evidence seems to suggest that the ability of incoherent interfaces to attract dislocations at elevated temperatures may be related to poor bonding across that interface: carbide strengthened aluminium alloys exhibit a strong asymmetry of the flow stress and the creep strength with respect to the loading direction [99, 110]; at elevated temperature the strength is reduced considerably under tension compared to compression
(whereas no asymmetry is observed at room temperature). This asymmetry has been attributed to carbide decohesion from the matrix under tension (interfacial debonding) and thus points to poor interfacial bonding. This is consistent with the finding that carbides are more beneficial for creep strength than oxides, the latter of which does not give rise to this asymmetry effect on elevated temperature strength.

Assuming that thermally activated dislocation detachment from the particle-matrix interfaces is the rate controlling mechanism in dispersion strengthened (DS) alloys and that the climb process can be regarded as sufficiently rapid, the quantitative rate equation developed from this model does not include a "true" threshold stress [99], which has the form of an Arrhenius expression: \( \dot{\varepsilon} = \dot{\varepsilon}_0 \exp(-E_a/kT) \), where \( \dot{\varepsilon}_0 \) is the reference strain rate, and \( E_a \) is the activation energy for dislocation detachment process. Both \( \dot{\varepsilon}_0 \) and \( E_a \) have been calculated from the model. The reference strain rate, \( \dot{\varepsilon}_0 \), was estimated by applying nucleation theory [111]: the probability that the dislocation has reached the critical configuration is given by the exponential term. The result is \( \dot{\varepsilon}_0 = 3\pi\lambda\rho_m/b \), where \( \lambda \) is the mean free path between the obstacles, and \( \rho_m \) is the mobile dislocation density. An approximate analytical expression for the activation energy for dislocation detachment, \( E_a \), was obtained by analyzing the energetics of dislocation detachment process from an attractive dispersoid particle. The passage of a dislocation through a uniform array of spherical particles of radius \( r \) and spacing \( \lambda \) was considered in the model analyses. The activation energy for dislocation detachment \( E_a \) is approximately given by the expression:

\[ E_a = \mu b^2 r[(1 - k_R)(1 - \tau/\tau_d)]^{3/2} \]

Where the applied stress, \( \tau \), is equal to or larger than the "athermal detachment stress", \( \tau_d \), thermal activation is not necessary to detach the
dislocation from the particle. When, however, the applied stress $\tau$ is smaller than $\tau_0$ the dislocation will reach an equilibrium position at the particle back depending upon the applied stress, the line energy of dislocation, and the strength of the dislocation-particle interaction. A finite activation energy for detachment $E_a$ has then to be supplied in order to enable dislocation detachment from the attractive dislocation-particle interaction [99].

The quantitative creep rate equation developed from this model has been compared to the experimental creep data of some DS alloys, such as tungsten wire filaments strengthened by dispersions of potassium vapour-filled bubbles, DS nickel base superalloy Inconel MA6000 strengthened in addition by yttria ($Y_2O_3$) dispersoids, fine grained aluminium strengthened by aluminium carbide ($Al_2C_3$) and aluminium oxide ($Al_2O_3$) particles, and rapidly solidified Al-8Fe-4Ce alloy strengthened by Al$_{10}$Fe$_2$Ce and Al$_{13}$Fe$_4$ particles. In all these cases, except fine aluminium strengthened by Al$_2C_3$ and Al$_2O_3$ particles, good agreement has been found between theory and experiment. Suggestions have been made by the theory to alloy development. Particular attention should be paid to the properties of the particle-matrix interface. A high degree of dislocation relaxation can only be achieved at an incoherent interface with a high specific energy. It is expected from the model that thermally stable bubbles should be the most effective barriers for the motion of dislocations at high temperature since the line energy relaxation should be maximal in this instance. The lowest attainable relaxation factor is about $k_r = 0.77$. This implies that the high temperature strength imparted by any particle dispersion cannot exceed about 60% of the Orowan stress ($\tau_0 = \tau_0(1 - k_r^2)^{1/2}$). The model also suggests that carbide dispersoids in aluminium alloys are about equally efficient as pores, and are more
beneficial for creep strength than oxides. This is related to interfacial bonding which can be affected by chemical compositions and the processing route.

Also, the difference between the precipitation and dispersion has been discussed in terms of the dislocation detachment creep model for the attractive particle-dislocation interaction during elevated temperature creep [9]. In general it appears that precipitation hardened alloys behave like materials without attractive interaction whereas dispersion strengthened alloys, produced for instance by mechanical alloying, behave like materials with a strong attractive particle-dislocation interaction. An important difference between the two particle types is that precipitates, unlike particles dispersed by mechanical means, have to overcome a nucleation energy barrier and are thus forced to form low energy (i.e. strong bonded) phase boundaries (interfaces). Weakly bonded interfaces are however needed to allow for the relaxation of the dislocation stress field by atomic rearrangements and fast diffusion along the interface. This may be the reason why small precipitates appear to be less capable of relaxing the line energy than small dispersoids. In addition, incoherency across the interface can lead to some degree of core spreading once the dislocation has reached the particle. This would not be possible at a strongly bonded coherent interface where crystal periodicity has to be maintained. While the detailed understanding of the particle-dislocation interactions at high temperature is certainly not far advanced, it thus appears that on the basis of a varying degree of interfacial bond strength and of particle coherency the creep behaviour of the whole class of particle strengthened materials may be consistently understood. This in itself may constitute a possibly important advance over climb theories for which the type of interface does not
play a role [99].

Finally, an important consequence concerns use of this creep equation for extrapolating creep data into regions where experimental data are not available. The shape of the curves of \( \log(\dot{\varepsilon}/\dot{\varepsilon}_0) \) versus \( \log(\sigma/\sigma_0) \) suggests that only materials with highly attractive dispersoids \( (k_r < 0.9) \) show "threshold-like" behaviour. In general, uncritical use of a "threshold stress" concept may result in serious overestimates of the creep strength at low strain rates (or low applied stresses). This danger may be averted by using this model-based creep rate equation.

The above dislocation detachment creep model [99] is correlated with some available creep and structure data for aluminium alloys strengthened by \( Al_2C_3 \) and \( Al_2O_3 \) particles, namely alloys Al-2.5, Al-10.0, IN9021 and IN9052 [100]. It is shown that though the model requires applied stress dependent apparent activation energy of creep, the stress dependence of creep rate can be satisfactorily accounted for even when this activation energy is stress independent, admitting a strong stress dependence of the pre-exponential structure factor, i.e. of the mobile dislocation density. On the other hand, the model is not able to account for the temperature dependence of creep rate if the temperature dependence is significantly stronger than that of the coefficient of lattice diffusion (a higher activation energy) as is usually the case with alloys strengthened by incoherent particles in which the attractive particle-dislocation interaction can be expected. It is suggested that the interpretation of creep in IN9021 alloy in terms of interface controlled diffusional transport of matter may be taken as an alternative to that based on the thermally activated detachment control.
1.4. The Role of Alloy Matrix in Creep Strength

Another popular line of study is the role of the alloy matrix in the creep behaviour of DS alloys [112-114]. By utilizing and extending the resisting stress concept, the experimental data have been evaluated for many of DS nickel base superalloys with various levels of matrix solid solution strengthening. The alloys examined include thorium (ThO₂) dispersoid TD-nickel, the oxide-dispersion-strengthened (ODS) mechanical alloys MA 754 and MA 753 (trademarks of the International Nickel Company), the ordered FCC γ'-Ti₃Al strengthened superalloys Udiment 700 (trademark of the Special Metals Corporation), Nimonic 115 (trademark of Henry Wiggins Limited) and Mar M209 (trademark of Martin-Marietta Inc.), and mechanical alloy MA 6000E (trademark of the International Nickel Company), strengthened by inert yttria (Y₂O₃) dispersoids and a high volume fraction of γ'-Ti₃Al precipitates. A generalized expression has been developed for creep rates which separates the matrix contributions from the particle contributions to the resisting stress and creep strength of these alloys.

This generalized creep rate equation is \( \dot{\varepsilon}_s = A_p(1 - k_s)^N((\sigma - \sigma_p)/E)^N \exp(-Q/RT) \) [114], where \( A_p \) is a structure-dependent parameter; \( k_s \) is a proportionality constant which is in the range from 0 to 1 and increases in magnitude with the extent of solid solution strengthening in the matrix phase; \( N \) is a stress exponent; \( \sigma \) is the applied stress; \( \sigma_p \) is the applied-stress-independent average resisting stress due to the strengthening particles; \( E \) is Young's modulus (temperature dependent); \( R \) is gas constant. A resisting stress component due to the matrix solid solution phase which is proportional to the net applied stress experienced by the matrix is \( \sigma_s = k_s(\sigma - \sigma_p) \). The total resisting stress to creep in
these particle-strengthened systems is \( \sigma_f = \sigma_p + \sigma_s = \sigma_p + k_s(\sigma - \sigma_p) \). Substituting \( \sigma_f \) into a standard empirical creep rate equation \( \dot{\varepsilon}_s = A_1[(\sigma - \sigma_f)/E]^N \exp(-Q/RT) \) for particle-strengthened alloys, one obtains the generalized creep rate equation, where \( A_1 \) is a constant [114]. The above generalized creep rate equation allows one to express the particle contribution to creep resistance of the alloy through the \( \sigma_p \) term in the resisting stress, and through the contribution of the matrix phase through the \((1 - k_s)^N\) term. It is also shown that the apparent stress exponent \( n \) in the empirical creep equation \( \dot{\varepsilon}_s = A_2\sigma^n \exp(-Q/RT) \) is related to the \( N \) and applied stress \( \sigma \) by a relation \( n = (\partial \ln \dot{\varepsilon}_s/\partial \ln \sigma)_f = N/(1 - \sigma_f/\sigma) \). The larger the ratio \( \sigma_f/\sigma \), the more sensitive is the creep rate to changes in the applied stress: this is not desirable in most applications. The relationship between \( Q \) and \( Q_e \) and other parameters such as \( k_s \), \( E \) and \( \sigma_p \) can be obtained by taking logarithms of the above two creep rate equations and differentiating with respect to temperature at constant applied stress. The activation energy \( Q \) is shown to be related to the apparent activation energy \( Q_e \), and to the temperature dependencies of the dislocation relaxation parameter \( \partial k_s/\partial T \). Young's modulus \( dE/dT \), and the particle resisting stress \( d\sigma_f/dT \), namely \( Q = Q_e + f(dk_s/dT, dE/dT, d\sigma_f/dT) \), where the activation energy \( Q \) is defined by the expression \( Q = RT^2(\partial \ln \dot{\varepsilon}_s/\partial T)_{\sigma_f} \), and \( f \) is a function of the temperature dependencies of the three parameters, namely \( k_s \), \( E \) and \( \sigma_p \).

It was concluded that the major role of the alloy matrix in the creep behaviour of these alloys is in determining the apparent stress dependence of the creep rates. Specifically, the stronger the matrix becomes through solid solution strengthening, the lower the applied stress sensitivity of the creep rate of the alloy. This analysis established
the role played by solid solution strengthening of matrix in the creep resistance of high
temperature alloys which are predominantly strengthened by second-phase particles.

From an alloy design point of view, these findings are particularly significant
because of the availability of such versatile processes as mechanical alloying [115-118]
which has made possible the development of alloys with tailor-made compositional
specifications with controlled amounts of inert dispersoid strengtheners. For example, the
undesirable high applied stress sensitivity displayed by DS alloys is not an intrinsic
characteristic of these alloys but could be alleviated. In particular, increasing the solid
solution strengthening contribution to the creep resisting stress to a level comparable with
that of the particle contribution would make this change possible. Decreasing the applied
stress sensitivity is of practical value in improving the performance reliability of these
alloys under practical conditions where spiked overloads might be expected. The analysis
further indicates that even small amounts of specific elements, e.g. the refractory elements
tungsten and molybdenum, could be very potent in improving creep resistance through
solid solution strengthening rather than larger amounts of other elements. A ranking of
the solid solution strengthening potency of the usual alloying elements in these high
temperature alloys, perhaps based on atomic size misfit effects, effects related to electron
vacancy number differences or effects related to shear modulus differences (see [118-
121]), would serve as a useful guideline in particle strengthened alloy design for better
creep resistance through enhanced solid solution strengthening.

1.5. Summary

From the above discussion, it is seen that the technological development of high
temperature alloys and the mechanistic study of creep behaviour are interdependent [22-25]. Notably this can be seen in two areas, namely that (1) solid solution strengthening and particle or fibre reinforcement of the matrix are connected to the lattice mechanisms of creep and to the interactions between dislocations and solute atoms and reinforcements [46, 53, 99, 114, 122-131]; and (2) high temperature creep and superplastic deformation of polycrystalline metallic and ceramic materials are linked to the boundary mechanisms of creep with various accommodations (grain boundary and/or lattice diffusion, grain boundary glassy phase, intragranular flow across the grains, formation of grain boundary cavities) [71, 77, 132-144]. Grain boundaries are a potential source of weakness at high temperatures because they provide both nucleation sites for cavities and paths for the interlinkage of cavities to form intergranular cracks. In general, the cavities tend to develop preferentially on those grain boundaries lying more nearly perpendicular to the tensile axis.

Recent studies on both the lattice and the boundary mechanisms of creep have been developed to a stage where a statistical treatment is employed. This treatment is based on the experimental observations that either dislocation link lengths or grain sizes are not uniform but rather take on a whole spectrum of lengths. This mathematical principle and physical methodology has been applied to creep and superplasticity studies [135, 145-148]. Some "unique" behaviour can only be predicted by means of this statistical treatment. Typical examples are (i) the strain-hardening coefficient variation during primary creep and its high values of the order of Young's modulus during steady-state creep; (ii) the correlation between recovery rate and dislocation annihilation rate; (iii)
the stress change during creep deformation; and (iv) superplastic deformation of a polycrystalline material. As a convenient means, this statistical treatment should find application to powder metallurgy, and other engineering materials microstructural features and analyses in that a large number of features are assembled in the microstructure of a complex material system under consideration.

In closing this brief review, it is noted that fineness in grain size, second phase particle, and interlamellar spacing is an effective means of obtaining high strength at low temperatures. Particle fineness must also be maintained within the limit of melting temperature in order that an improved high temperature strength and creep resistance are developed for a solid at elevated temperatures, while larger grain size or monocristalline materials will have higher creep resistance as compared to its fine grained polycrystal counterparts [25, 149-154]. For multiphase DS alloys and MMCs, the properties of alloy matrix, the interfaces, and the interaction between the matrix and the reinforcements, are together the three factors that determine the creep properties of these materials. The functional performance of a component within an operating machine at elevated temperatures is in a large measure dependent on its microstructural stability and dimensional fitness. A more reliable and safer operation of an efficient machine for elevated temperature applications such as in energy generating component will dependent on both an improved mechanical design for the component and materials design and processing for the microstructure and elevated temperature properties.

§2. Objectives of Present Study

The purposes of this work are to study creep deformation mechanisms for two
model materials, namely an AISI type 310 stainless steel and high purity polycrystalline magnesium. The two materials represent Class II solid solution alloys (the 310 stainless steel) and pure metals (pure magnesium), respectively. The reason for these choices is that the alloy matrix plays an important role in creep performance for complex engineering alloys such as nickel based superalloys. The 310 stainless steel is a typical iron based solid solution alloy which is used in elevated temperature and corrosive environments for its excellent elevated temperature strength and corrosion resistance. Pure magnesium has its advantages of comparatively low melting point which makes it possible to carry out creep experiments of relatively low temperatures.

Two approaches are used in this work, namely the development of experimental techniques and theoretical modelling of the micromechanisms of creep. Experimental measurement techniques include creep, stress relaxation, and a combined creep - stress relaxation test. The tests for the two materials are carried out over a range of test temperatures and applied stress. Also, the recovery rate and strain hardening coefficient are measured by using the combined creep - stress relaxation test. This particular experimental method, i.e. combined creep - stress relaxation, is a very useful technique for studying strengthening mechanisms for elevated temperature plastic deformation and can be generally applied to more complex engineering alloys. For instance, it can be applied directly or with slight modifications to measure recovery rates during elevated temperature plastic deformation of metals and alloys. It can also be used in alloy design and development for elevated temperature applications by identifying effective strengthening alloying elements or processing procedures. Any factor that slows down the
recovery rate will have the potential for improving the elevated temperature strength of complex engineering alloys.

For the micromechanical modelling, the creep mechanisms are studied using detailed (statistical) dislocation models. A dislocation network model, i.e. the Öström-Lagneborg creep model, is first modified by the author to take into account subgrain formation during creep deformation, and is then applied to the creep of the 310 stainless steel. The appropriateness of the modification is checked by comparing the calculated results with the experimental results for 310 stainless steel, and incorporating recent elastic theories for two and three dimensional subgrain boundaries and experimental results on subgrain formation. For the first time ever, the author uses dislocation link length statistics to model the microstructure developed for several experimental deformation conditions, namely creep, stress change tests during steady state creep, and strain rate changes during a constant strain rate test. Various original results are obtained from this modelling which give a satisfactorily account of a variety of experimental observations such as the dislocation density and average dislocation link lengths during creep, the strain hardening coefficients found for a stress increase during creep, the Cottrell-Stokes law, and the Haasen relationship for the flow stress increase associated with an instantaneous strain rate increase during constant strain rate deformation. In addition, a creep mechanism model is proposed and applied to superplasticity of polycrystalline materials. This model is based on the consideration of a continuous grain size distribution. Such a model can used to predict the internal stress at the transition between power law creep an linear Newtonian viscous flow. This computed transition
stress is compared with the experimental results from creep tests and the agreement is found to be fairly good.
CHAPTER III EXPERIMENTAL MATERIALS AND PROCEDURES

§1. Experimental Materials

Experimental materials were an AISI type 310 stainless steel and a high purity magnesium (purity 99.99%).

The chemical composition of the 310 stainless steel was (wt%): 0.065 C, 0.65 Si, 1.43 Mn, 0.004 S, 18.08 Ni, and 25.11 Cr. A high chromium content in the 310 stainless steel imparts its good resistance to high temperature oxidation. The high nickel content improves the resistance to cyclic oxidation. The high alloy content of the 310 stainless steel contributes to its excellent high temperature strength [155]. The 310 stainless steel specimens were of circular cross section with a diameter of 6.35 mm and a gauge length of 38.0 mm, Fig. 3. The specimens were first annealed at 1000 °C for 15 minutes and water quenched. This was followed by a 10 hour heat treatment at 700 °C to stabilize the grain size of the specimens.

Magnesium is the lightest engineering metal with a density of 1.74 g/cm³ (as compared to that of aluminium 2.70 g/cm³), and has its potential applications in weight and energy saving structural components. High strength-to-weight ratios of magnesium alloys allow thin-section designs which retain good fatigue and creep resistance and are suitable for electromagnetic interference (EMI) shielding applications. Also, it is in the form of diecastings that the use of magnesium alloys is increasing most rapidly [156]. The pure magnesium specimens were rod type with a diameter of 5.6 mm and a gauge length of 25.4 mm. They were annealed at 200 °C for 317 hours prior to the creep and stress relaxation tests.
Fig. 3 Creep specimens for the 310 stainless steel and pure magnesium.
The grain size of both the 310 stainless steel and the pure magnesium were determined to be about 100 ± 20 μm by the line intersection method. The heat treatments for both materials were made to ensure that grain sizes were sufficiently coarse such that during subsequent creep and stress relaxation tests, grain sizes will not undergo large variations (they will grow during creep) and should not be an important consideration in our experiments. Furthermore, the creep test conditions (applied stresses and test temperatures) for the two materials were in the power law creep regime. In this regime, creep strain is mainly generated by dislocation glide. Only if the applied stress is sufficiently low and the test temperature is sufficiently high (in the diffusion creep regime), or if the material has ultra-fine grain sizes (a few micro-metres) will grain size be an important factor in controlling the creep rate.

§2. Creep Test Machine

The creep and stress relaxation tests were conducted using an Instron 8562 model testing machine, Fig. 4. The test system includes three parts, namely (i) the test frame, a microprocessor control for mechanical test commands; (ii) an X-Y recorder for load and displacement as well as time registration; and (iii) a self adaptive temperature control together with a furnace and a water cooling system for protecting load cell and machine stability. Before each of the tests on a new specimen, a calibration was made for load and displacement according to the operation procedures specified for the test machine. During the tests, the test temperature was held to within ± 0.5 °C and the load to ± 1.0 N. The room temperature was maintained at 25 ± 0.5 °C. The test temperature and applied stress ranges utilized were 150 °C to 250 °C (T/T_m = 0.46 to 0.57) and 20 MPa to 50 MPa (σ/μ
Fig. 4 Instron 8562 model creep testing machine.
for the pure magnesium, and 650 °C to 800 °C (T/T_m = 0.51 to 0.59) and 110 MPa to 320 MPa (σ/μ = 1.44 · 10^3 to 5.78 · 10^3) for the 310 stainless steel, respectively. The specimen was held at each of the set temperatures for about 3 hours prior to the tests in order to equilibrate the temperature of the furnace and the specimens. The creep tests took about 3 to 5 hours from transient creep to steady-state creep, depending on the applied stresses and test temperatures. For the stress relaxation tests of pure magnesium, the cross-head of the test machine was stopped by turning off the actuator when creep was in steady-state stage where a constant displacement was maintained during the test. The current load in the stress relaxation test (starting from the initial creep load) was recorded together with the relaxation time. The stress relaxation test took about 3 hours. This test was used to measure the dynamic recovery rate corresponding to the steady-state creep, since each of the stress relaxation tests was started when the specimen was crept into the steady-state stage. Great care was exercised during the experiment, especially at the onset of both creep and stress relaxation tests, in the operation of the test machine. This was done by first familiarizing oneself with the operating procedures specified in the operations manual for the test machine (Manual No. M11-98560-1(A), Operating Instructions for Instron Model 8562 Dynamic Testing System, Instron Corporation, 1988) and then by adequate "practice" with the test machine and some actual specimens. A step by step operating procedures for Instron 8562 test system are described in Appendix B for creep and stress relaxation tests.

The stress dependence of the initial creep rate is used as an indirect experimental measurement for the stress dependence of the average effective dislocation velocity during
recovery creep deformation. The combined creep and stress relaxation tests can be used to measure the steady-state creep, \( \dot{\varepsilon}_s \), and the dynamic recovery rate, \( R \). The strain-hardening coefficient, \( H \), can then be calculated from the Bailey-Orowan equation [75, 157], \( \dot{\varepsilon}_s = R/H \), for recovery creep. This provides a means to study the strain-hardening and recovery behaviour of a material during high temperature recovery creep deformation.

§3. Numerical Integration Methods

All experimental data were analyzed by using the SYSTAT package at the Computer Centre, University of Windsor. This SYSTAT package is a very practical and simple data analysis package. It provides polynomial fitting for a set of experimental data. For linear regression of experimental results, the correlation coefficient \( r_{LR} \) will be given for each set of data. Numerical integrations of model equations were performed on CMS main frame at the Computer Centre, University of Windsor. All computer programmes were written in FORTRAN IV. The numerical method was the standard finite difference method (FDM), see for example reference [158].

For numerical integration of the model equations and other related calculations, the following numerical methods were applied, namely:

(i) The Lagrange polynomial (ordinate form) interpolation (with the linear interpolation for the first 3 and last 4 nodes): The Lagrange interpolating polynomial (ordinate form) for \( n + 1 \) points of function \( y = f(x) \): \((x_1, y_1), (x_2, y_2), \ldots, (x_{n+1}, y_{n+1})\) can be written as:
\[ P_n(x) = \sum_{k=1}^{n+1} \left\{ y_k \prod_{j=1}^{n+1} \frac{(x-x_j)}{(x_k-x_j)} \right\} \]  

(3)

(ii) Simpson’s 1/3 Rule integration and Romberg’s integration methods:

\[ \int_{x_1}^{x_{n+1}} f(x) \, dx = \frac{h_1}{3} \left( y_1 + 4 \sum_{i=1}^{n/2} y_{2i} + 2 \sum_{j=1}^{n/2-1} y_{2j+1} + y_{n+1} \right) \]  

(4)

\[ \int_{x_1}^{x_{n+1}} f(x) \, dx = \frac{2}{15} \left\{ 16 \int_{\text{Trapezoid}} - \int_{\text{Simpson}} \right\} \]  

(5)

Where \( h_1 \) is the step size in the space variable \( x \), \( n \) is an even integral number in equation (4) for the Simpson’s 1/3 Rule integration, and the Trapezoid and Simpson in the braces of equation (5) refer to the two integration procedures of Romberg’s integration method, respectively. The step size for the trapezoid integration is refined by half \( (h_1/2) \) as compared to that for the Simpson’s integration \( (h_1) \). The first term of the trapezoidal integration in equation (5) for Romberg’s integration method is given by:

\[ \int_{\text{Trapezoid}}^{h_1/2} = \frac{h_1}{4} \left( y_1 + 2 \sum_{i=2}^{2n} y_i + y_{2n+1} \right) \]  

(5A)

and the second term of the Simpson’s 1/3 Rule integration in equation (5) is given by equation (4).

(iii) The Runge-Kutta method for the differential equations including the following
calculation: (a) five point derivative approximation for the space differentiation, \( \frac{df(x)}{dx} \); and the fourth-order PECEC method with (b) the predictor of the third-order Nyström method and (c) the corrector as in the Milne-Simpson method for the time differentiation: \( \frac{dy(t)}{dt} = f(y(t)) \). These are the standard numerical methods for practical engineering calculations, and the formulas are given as follows:

(a) Five-point formula for space differentiation:

\[
\frac{df(x)}{dx} = \frac{1}{12h_1} (f_1 - 8f_2 + 8f_4 - f_5)
\]  

(b) The third-order Nyström method (explicit) for the predictor:

\[
y_n = y_{n-2} + \frac{h_2}{3} (7f(y_{n-1}) - 2f(y_{n-2}) + f(y_{n-3}))
\]  

(c) The fourth-order Milne-Simpson method (implicit) method for the corrector:

\[
y_n = y_{n-2} + \frac{h_2}{3} (f(y_n) + 4f(y_{n-1}) + f(y_{n-2}))
\]

Where \( h_2 \) is the step size in the time variable t. The detailed descriptions of the above three standard numerical integration methods can be found for instance in reference [158]. In the calculation, the step size in time variable \( h_2 \) was taken to be the average of the two or three consecutive time steps for equations (7) and (8).
CHAPTER IV \hspace{1cm} RESULTS AND DISCUSSION

§1. Initial and Steady-State Creep

1.1. Introduction

While many experimental and theoretical studies are concerned with steady-state creep stage, it is interesting to note that early work of Garofalo et al [159] indicated that a relationship exists between the steady-state creep rate, $\varepsilon_s$, and the initial creep rate, $\dot{\varepsilon}_i$ (the initial slope of $\varepsilon$ versus $t$ curve), for an AISI 316 type austenitic stainless steel at temperature from 704 °C to 830 °C under applied stresses ranging from 20.7 MPa to 203.5 MPa. The relationship is $\dot{\varepsilon}_i \approx 3.3 \varepsilon_s$. Other investigators have shown a similar relationship to be valid for other materials [6, 160]. However, a much higher creep rate ratio, $F = \dot{\varepsilon}_i/\varepsilon_s$, was observed by Evans and Wilshire [161, 162] and Webster et al [163]. These authors pointed out that the Garofalo empirical creep equation:

$$\varepsilon(t) = \varepsilon_0 + \varepsilon_c (1 - e^{-rt}) + \dot{\varepsilon}_s t \hspace{1cm} (9)$$

does not adequately describe the creep data immediately after loading in Ni-0.18wt%Pd, Zn-0.41wt%Ag, $\alpha$-Fe, and Cu-15at%Al [161, 162], and that simple first-order reaction rate theory is not valid at the time of loading and immediately thereafter [163]. In equation (9), $\varepsilon(t)$ is the creep strain at a given time $t$, $\varepsilon_0$ is the instantaneous strain upon loading, $\varepsilon_c$ is the limit for transient creep, $r$ is the ratio of transient creep rate to the transient creep strain, and $t$ is the creep time.

Given the large differences reported for values of the creep rate ratio, $F$, further investigations were necessary. Our creep experimental tests were designed to study the
dependence of the creep rate ratio, \( F \), on both test temperature and applied stress for the two materials, namely the 310 stainless steel and pure magnesium. Therefore, a range of test temperatures and applied stresses were employed to the present creep tests. Both the creep strain rate ratio, \( F = \dot{\varepsilon}/\dot{\varepsilon}_e \), and the apparent activation energy for creep, \( Q_e \), were measured for the two materials.

1.2. Experimental Results

An actual creep curve is given in Fig. 5. The measured creep strain rate ratio \( F \) for both the 310 stainless steel and pure magnesium are shown in Figs. 6(a) and (b), and Figs. 7(a) and (b), respectively. Each test point is for a different specimen. From these results it can be seen that \( F \) depends on the applied stress at a constant temperature, and on the temperature under a constant applied stress. The applied stress and temperature dependencies of \( F \) for these two materials under the test conditions utilized are as follows:

(i) At a constant temperature, \( F \) decreases from a large value at low applied stresses to about unity at high applied stresses. For the 310 stainless steel, \( F \) decreases from 162.9 at 110 MPa to 2.5 at 320 MPa when crept at 700 °C, Fig. 6(b). For pure magnesium at 200 °C, \( F \) decreases from 33.6 at 20 MPa to 1.9 at 50 MPa, Fig. 6(a). (ii) If the applied stress is kept constant, \( F \) is large at low temperatures and small at high temperatures. For the two materials, the changes in \( F \) with temperature are similar. For the 310 stainless steel crept at 200 MPa, \( F = 99.7 \) at 650 °C and 4.3 at 800 °C, Fig. 7(b). For pure magnesium at 35 MPa, \( F = 63.8 \) at 150 °C and 1.7 at 250 °C, Fig. 7(a). These results show that the applied stress and temperature have similar effects on \( F \): at low applied stresses and/or test temperatures, \( F \) is large, while at high applied stresses and/or test
Fig. 5 A typical creep curve for the 310 stainless steel crept at 700 °C and 200 MPa.
Fig. 6 (a) The creep rate ratio, $F$, versus the applied stress, $\sigma$, for pure magnesium at 200°C and 20 MPa to 50 MPa;
Fig. 6 (b) The creep rate ratio, $F$, versus the applied stress, $\sigma$, for the 310 stainless steel at $700 \, ^{\circ}\!\mathrm{C}$ and 110 MPa to 320 MPa.
Fig. 7 (a) The creep rate ratio, F, versus the creep test temperature, T, for pure magnesium at 35 MPa and 150 °C to 250 °C;
Fig. 7 (b) The creep rate ratio, $F$, versus the creep test temperature, $T$, for the 310 stainless steel at 200 MPa and 650 °C to 800 °C.
temperatures, $F$ is close to unity.

The values of the apparent activation energy for creep, $Q_a$, have been measured for both materials. Also the change in $Q_a$ with creep strain $\varepsilon$ has been obtained from the creep strain-time curves at different creep strain values. The results are presented in Figs. 8(a) and (b), and Figs. 9(a) and (b) for the two materials, respectively. Figs. 8(a) and (b) are plots of strain rate $\dot{\varepsilon}$ versus the reciprocal temperature $1/T$ for the 310 stainless steel (200 MPa, 650 °C to 800 °C), Fig. 8(a), and pure magnesium (35 MPa, 150 °C to 250 °C), Fig. 5(b), at several creep strains. From these results, it was calculated that the initial value of $Q_a$, i.e. $Q_{ai}$, is smaller than the value of $Q_a$ at steady-state stage, $Q_{as}$. This is further illustrated in Figs. 9(a) and (b) which are plots of $Q_a$ versus creep strain for both materials. The two materials show the same tendency in the variation of $Q_a$ with $\varepsilon$. As the creep strain increases the apparent activation energy for creep, $Q_a$, increases from an initial value, $Q_{ai}$, to a steady-state value, $Q_{as}$. For the two materials, $Q_{ai} = 87$ kJ/mole and $Q_{as} = 106$ kJ/mole for pure magnesium, Fig. 9(a), and $Q_{ai} = 210$ kJ/mole and $Q_{as} = 341$ kJ/mole for the 310 stainless steel, Fig. 9(b), respectively. Correlation coefficient $r_{ui}$ values are within an interval between 0.995 and 0.997 for linear regression of $\ln\dot{\varepsilon}$ versus $1/T$ for the two materials. The activation energy for creep at steady-state is of the order of but smaller than the activation energy for volume diffusion, $Q_v$, for both materials, which are $Q_v = 270$ kJ/mole to 311 kJ/mole for FCC-Fe and $Q_v = 135$ kJ/mole for pure magnesium [18, 22]. For an austenitic stainless steel, $Q_v = 314$ kJ/mole [18].

1.3. Discussion of the Results and Summary

The large $F$ value at low temperatures and/or stresses has been predicted by Li
Fig. 8 (a) The natural logarithm of strain rate $\ln(\dot{\varepsilon})$ versus the reciprocal of test temperature $1/T$ plots, for pure magnesium at 35 MPa and 150 °C to 250 °C: (1) initial state, (2) $\dot{\varepsilon} = 4.92 \cdot 10^4$, (3) $\dot{\varepsilon} = 14.76 \cdot 10^4$, (4) $\dot{\varepsilon} = 24.60 \cdot 10^4$, (5) steady-state:
Fig. 8 (b) The natural logarithm of strain rate $\ln(\dot{\varepsilon})_c$ versus the reciprocal of test temperature $1/T$ plots for the 310 stainless steel at 200 MPa and 650 °C to 800 °C: (1) initial state, (2) $\dot{\varepsilon} = 3.0 \cdot 10^{-4}$, (3) $\dot{\varepsilon} = 1.21 \cdot 10^{-4}$, (4) $\dot{\varepsilon} = 2.70 \cdot 10^{-4}$, (5) steady-state.
Fig. 9 (a) Apparent activation energy for creep, $Q_a$, versus creep strain, $\varepsilon(t)$, for pure magnesium at 35 MPa and 150 °C to 250 °C.
Fig. 9 (b) Apparent activation energy for creep, $Q_c$, versus creep strain, $\varepsilon(t)$, for the 310 stainless steel at 200 MPa and 650 °C to 800 °C. Note that for both of the materials, the apparent activation energy for creep, $Q_c$, is smaller at the onset of creep ($\varepsilon \leq 10^{-3}$) than that measured at steady-state creep.
[164] and Garofalo [18] in considering Li's creep equation, which relates the creep rates at different stages of creep deformation process. This equation has the form:

\[
\dot{\epsilon}(\tau) = \dot{\epsilon}_s \left( 1 - \frac{\dot{\epsilon}_s - \dot{\epsilon}_i}{\dot{\epsilon}_s} \exp\left(-k_1 \tau\right) \right)^{-1}
\]  \hspace{1cm} (10)

where \( k_1 \) is the multiplication rate constant for the dislocation kinetics. By setting \( \dot{\epsilon}_i >> \dot{\epsilon}_s \) and \( k_1 << 1 \) for low temperatures and stresses, equation (10) reduces to a logarithmic relationship between \( \dot{\epsilon} \) and \( \tau \). According to Li's dislocation mechanism for creep [164], at high temperatures and stresses, the creep rate ratio \( F \) tends to unity and \( k_1 \) is very large which indicates a large multiplication rate for dislocations during creep deformation. In this case, equation (10) reduces to an exponential type relationship between \( \dot{\epsilon} \) and \( \tau \) as has been established experimentally [18].

The apparent activation energy at the onset of creep deformation is smaller than that in steady-state stage for both materials. This may be due to the variation of internal stress during transient creep. From numerous experimental measurements using stress dip technique made by Ahlquist and Nix [165] and by Northwood and Smith [166] on various pure metals and Class II alloys undergoing creep at elevated temperatures, the average internal stress \( \sigma_i \) during normal transient creep increases from an initial value close to zero to a steady-state value of a magnitude of 0.85\( \sigma \), Fig. 10. Such an analysis can be applied to our creep tests since all the creep strain-time curves for the 310 stainless steel and pure magnesium under the present test conditions were of the normal type (N-type) transient. In Fig. 10, the applied stress \( \sigma \) is a constant, and the average internal stress \( \sigma_i \).
Fig. 10 A schematic illustration of the variation of the average internal stress, $\sigma_i$, and the average effective stress, $\sigma_e = \sigma - \sigma_i$, during a normal transient creep, where the applied stress, $\sigma$, is kept constant.
increases during N-type transient creep to a steady-state value about 0.85σ. The effective stress \( \sigma_e = \sigma - \sigma_c \), which is the driving force for dislocation glide [165, 166], decreases from about \( \sigma \) upon loading to a steady-state value of the magnitude 0.15σ. A generalized rate equation for the flow process which describes the temperature and stress dependence of strain rate can be written in the following form [16, 53, 57]:

\[
\dot{\varepsilon} = \dot{\varepsilon}_0 \exp \left\{ \frac{-Q - bA^\tau}{kT} \right\} 
\]  

(11)

where \( \dot{\varepsilon}_0 \) is a frequency factor, \( Q \) is the activation energy for the glide process, \( A^\tau \) is the activation area for dislocation glide process, \( \tau \) is the shear stress in slip direction and is related to the uniaxial tensile stress \( \sigma \) by \( \sigma = M \tau \), where \( M \) is the Taylor factor (an orientation factor). For polycrystalline cubic metals \( M = 3.1 \). The term \( bA^\tau \) is the work done by the shear stress \( \tau \) during a unit step of the glide process. Equation (11) represents plastic deformation of crystalline materials carried out by the stress assisted thermally activated motion of dislocations. When there is no applied stress, the forward and backward motions of a dislocation have the same probability. If a shear stress, \( \tau \), is exerted on the dislocation, forward motion is more probable than backward motion. Under the action of the shear stress, forward and backward motion frequencies are given by \( v^* \propto \exp[-(Q - bA^\tau)/kT] \) and \( v \propto \exp[-(Q + bA^\tau)/kT] \), respectively, where \( Q \) is the activation energy for zero applied stress, and the term \( bA^\tau \) represents the work done by the applied stress during the thermal activation. The inequality relationship \( v^* > v \) signifies that a finite shear stress assists forward motion and retards backward motion.
The plastic strain rate, \( \dot{\varepsilon} \), generated by this stress assisted thermally activated motion of dislocations is then given by

\[
\dot{\varepsilon} = \dot{\varepsilon}_0 (v^* - v) = 2\dot{\varepsilon}_0 \sinh(bA^* \tau/kT) \exp(-Q/kT).
\]

When \( bA^* \tau/kT \geq 1 \), which is generally valid for power law creep and tensile tests, backward motion can be neglected for \( v << v^* \). Hence the plastic strain rate generated is approximately given by equation (11).

In the case of dislocation creep, the shear stress \( \tau \) is equal to the effective shear stress \( \tau_e \) which drives dislocation glide and thus generates the creep strain. For high temperature creep, the activation area \( A^* \) is about 20b\(^2\) to 100b\(^2\) (where \( A^* \) is a decreasing function of stress \( \tau \) and the term \( bA^* \tau = 32 \) kJ/mole for the creep of single crystal Mg-12%Li alloy) [57]. Recent creep measurements on various alloys has shown that the activation area \( A^* \) is an inverse function of the effective shear stress \( \tau_e \) during recovery creep deformation [62]. The measured apparent activation energy for creep, \( Q_e \), is then equal to:

\[
Q_e = Q - bA^* \tau \tag{12}
\]

At the very beginning of loading, the effective stress is almost equal to the applied stress \( \tau_e = \tau \) (c.f. Fig. 10), and the term \( bA^* \tau \) is large but gradually becomes smaller in the case of normal N-type transient creep such as for the present creep experiments on the two materials. For the 310 stainless steel crept at 200 MPa and 700 °C, the contribution of the term \( bA^* \tau \) is estimated as 13.3 kJ/mole to 66.7 kJ/mole by taking \( A^* = 20b^2 \) to 100b\(^2\) and \( \tau_e = \tau \). Similarly for pure magnesium at 35 MPa and 200 °C, \( bA^* \tau = 4.5 \) kJ/mole to 22.5 kJ/mole. It is thus expected that at the onset of creep, \( Q_e \) would be 13.3 kJ/mole to 66.7
kJ/mole smaller for the 310 stainless steel, and 4.5 kJ/mole to 22.5 kJ/mole smaller for pure magnesium than the values measured in steady-state. In steady-state stage creep, the effective stress \( \sigma_e \) is very small as compared to that of the onset of N-type transient creep and thus \( Q_e \) is closer to \( Q \). Our experimental results for the 310 stainless steel and pure magnesium, Figs. 9(a) and (b) show this trend of an increasing \( Q_e \) from the transient to steady-state creep stages, which is in agreement with the activation analysis described above; quantitatively, however, the agreement is still poor.

The creep of pure metals and Class II alloys is diffusion climb controlled, and the apparent activation energy for creep \( Q_e \) is typically equal to the activation energy for volume diffusion \( Q_v \) [25, 55-61, 167]. The measured apparent activation energy for steady-state creep for pure magnesium, \( Q_{es} = 106 \) kJ/mole, is smaller than \( Q_e = 135 \) kJ/mole. This is probably due to a temperature dependence of \( Q_e \). For example, it has been reported that the ratio \( Q/Q_v = 1 \) when \( T/T_m \geq 0.7 \), whereas \( Q/Q_v = 0.6 \) to 1 when \( T/T_m = 0.5 \) to 0.7 [60]. The temperatures employed for pure magnesium were in the range from \( 0.46T_m \) to \( 0.57T_m \). Thus, the measured \( Q_e \) for pure magnesium (\( Q/Q_v = 0.79 \)) may reflect a temperature dependence of \( Q_e \) (change of mechanism). Our results for the 310 stainless steel, \( Q_e = 341 \) kJ/mole, is approximately equal to the value reported by Garofalo [18] for an austenitic stainless steel, \( Q_e = 314 \) kJ/mole (\( \sim 8\% \) difference). And the range of \( Q_e \) for FCC-Fe was 270 kJ/mole to 311 kJ/mole. This difference may be due to the alloying elements in the stainless steel which are involved in the diffusion climb process and the interaction between solute atoms and dislocations during recovery creep deformation and stacking fault energies.
In summary, the above experimental results have shown that for the two materials, the creep rate ratio $F$ is dependent on both the creep temperature and the applied stress. Generally, an increase in the temperature and/or the applied stress gives rise to a decrease in $F$. These results are supportive of Li's dislocation mechanism for creep [164] in which $F$ can be very large at low temperatures and/or applied stresses and is equal to unity at high temperatures and/or applied stresses. It is further shown that the apparent activation energy for creep, $Q_c$, at the onset of creep (creep strain $\leq 10^{-4}$) is smaller than the value of $Q_c$ determined during steady-state creep. This change in the apparent activation energy for creep may be due to the variation of the internal and effective stresses during transient creep.
§2. Stress Dependence of Creep Rates

2.1. Experimental Results

The stress dependencies of the initial and steady-state creep rates measured for the 310 stainless steel and pure magnesium are reported in this section. The experimental results are discussed in terms of the internal and effective stress creep theory and recent dislocation network models of recovery creep.

The dependence of the creep rate $\dot{\varepsilon}$ on the applied stress $\sigma$ for pure magnesium is shown in Figs. 11(a) and (b), for both the initial and steady-state stages of creep. The applied stress dependencies of the initial and steady-state creep rates can be described by the equations:

\[
\dot{\varepsilon}_I = \dot{\varepsilon}_{I0} \sigma^{n_I} \tag{13}
\]

and

\[
\dot{\varepsilon}_S = \dot{\varepsilon}_{S0} \sigma^{n_S} \tag{14}
\]

respectively, where $\dot{\varepsilon}_{I0}$ and $\dot{\varepsilon}_{S0}$ are numerical constants. For pure magnesium at 200 °C, $n_I = 2.8$ with correlation coefficient $r_{I,R} = 0.946$ and $n_S = 5.9$ with $r_{S,R} = 0.989$, respectively. The relationship between the initial and steady-state creep rates can be described by the equation $\log \dot{\varepsilon}_I = 0.48 \log \dot{\varepsilon}_S$ with $r_{I,R} = 0.972$ for the pure magnesium under these creep conditions (200 °C, 20 MPa to 50 MPa).

For the 310 stainless steel, the initial creep rate is also dependent on the applied stress as given by equation (13), see Fig. 12. The value of $n_I$ was determined as 2.1 with
Fig. 11  (a) Log-log plots of the initial creep rate, $\varepsilon_i$, versus applied stress, $\sigma$, for pure magnesium creep tested at 200 °C under applied stresses ranging from 20 MPa to 50 MPa.
Fig. 11 (b) Log-log plots of the steady-state creep rate, $\dot{\varepsilon}$, versus applied stress, $\sigma$, for pure magnesium creep tested at 200 °C under applied stresses ranging from 20 MPa to 50 MPa.
Fig. 12 Log-log plots of (a) the initial creep rate, $\dot{\varepsilon}_i$; and (b) the steady-state creep rate, $\dot{\varepsilon}_s$, versus applied stress, $\sigma$, for the 310 stainless steel creep tested at 700 °C under applied stresses ranging from 100 MPa to 320 MPa. Note the non-linear relationship (solid curve). The dotted line represents a linear plot.
\( r_{LR} = 0.884 \). The log-log plot of the steady-state creep rate versus the applied stress shows a non-linear behaviour, indicating that the stress exponent for the steady-state creep \( n \), in the power law creep equation, equation (14), increases with the applied stress from 3 to 16 when \( \sigma \) increases from 110 MPa to 320 MPa at 700 °C. This non-linear behaviour can also be seen in the \( \log \dot{\epsilon}_i \) versus \( \log \sigma \) plot, Fig. 12. This type of behaviour, i.e. an increase in \( n \) with \( \sigma \), has been observed by a number of investigators in a range of materials, including aluminium, nickel, and platinum [168], and an austenitic stainless steel [169]. This increase in the stress exponent, \( n \), is explained in terms of the average internal stress, \( \sigma_i \), the average effective stress, \( \sigma_e \), and the network growth kinetics (at elevated temperatures, the average link length of the network increases with time), in that the larger the applied stress \( \sigma \) to test temperature \( T \) ratio, \( \sigma/T \), the larger the \( n \) value [170]. The stress exponent \( n \) is expressed in terms of \( \sigma_i \) and \( \sigma_e \) as \( n = (1 + \sigma_e/\sigma_i)/[0.25(2pt) \kappa T / \sigma_i b A^* (\sigma_e/\sigma_i)] \), where \( A^* \) is activation area, and \( b \) is Burgers vector. Temperature affects \( \sigma_e/\sigma_i \) and hence \( n \) (\( \sigma_e/\sigma_i \) decreases with temperature) [170]. If a linear plot is used for the \( \log \dot{\epsilon}_i \) versus \( \log \sigma \) data for the 310 stainless steel, then \( n_s = 4.6 \) with \( r_{LR} = 0.947 \). This is within the range of \( n \) values previously reported for austenitic stainless steels [171]. For the 310 stainless steel, the initial creep rate, \( \dot{\epsilon}_i \), is related to the steady-state creep rate, \( \dot{\epsilon}_s \), by the relationship \( \log \dot{\epsilon}_i = 0.33 \log \dot{\epsilon}_s \) with \( r_{LR} = 0.978 \) for the present experimental conditions (700 °C, 110 MPa to 320 MPa).

2.2. Discussion of the Results in terms of the Internal and Effective Stresses Model

To discuss the results reported herein, the well-known Taylor-Growan equation
[172, 173] is applied to both the initial and steady-state creep. The equation relates the macroscopic strain rate, \( \dot{\varepsilon} \), to the mobile dislocation density, \( \rho_m \), and the average effective dislocation velocity, \( v \), via:

\[
\dot{\varepsilon} = ab \rho_m v
\]  

(15)

where \( a = 0.5 \) is an average Schmid strain resolution factor [53]. This theory is particularly suited to the analysis of the creep phenomenon and has been utilized by many authors, including among others Webster et al [163], Li [164], Gilman [174], and Ajaja and Ardell [175]. For steady-state creep, the total dislocation density, \( \rho \), is related to the applied tensile stress, \( \sigma \), by the Taylor relationship [172, 176, 177]:

\[
\sigma = \alpha_1 M_0 b \rho^{1/2}
\]  

(16)

where \( \alpha_1 \) characterizes the interaction of dislocations and is dependent on the geometrical arrangement of the dislocation structure [178, 179], and on the character of dislocation motion as a whole [65, 176]. For several different dislocation structures, such as Taylor lattice, Seeger pile-ups or glide zones, Basinski intersecting dislocations, Mott-Hirsch dragging of superjogs, and Kuhlmann-Wilsdorf bowing-out in dislocation network, \( \alpha_1 \) is calculated to be in the range from 0.3 to 0.6 [178]. Experimental data on pure metals and some solid solution alloys [179] shows that \( \alpha_1 \) takes values within the interval from 0.1 to about 1.0. According to recent network models for recovery creep deformation, the mobile dislocation density \( \rho_m \) is linearly related to the total dislocation density \( \rho \) during
the steady-state creep, i.e.:

$$\rho_m = Z \rho$$  \hspace{1cm} (17)

where \( Z \) is a constant about 0.2 \( [176, 180] \). From equations (15) to (17) and the experimental results for our two materials \( (n_c = 5) \), it appears that the average effective velocity of mobile dislocations is related to the applied stress by an equation of the form:

$$v = v_o \sigma^p$$  \hspace{1cm} (18)

where \( v_o \) is a constant. For our experimental results for the 310 stainless steel and pure magnesium, it is estimated that \( p = 3 \) by considering that the initial creep rate, \( \dot{\varepsilon}_c \), is proportional to the average dislocation velocity, \( v \). This stress dependence will be discussed in full detail in the section 3.4 of Chapter IV on creep and stress relaxation results for pure magnesium with respect to recovery rate data. The results from experimental measurement agree with predictions from these network models.

The stress dependence of the initial creep rate for the two materials will now be discussed by using the internal and effective stress theory \( [165, 166, 181, 182] \). Ahlquist et al \( [181] \) have developed an equation for the stress exponent \( n \) in the power law equations (c.f. equations (13) and (14)) relating the creep strain rate \( \dot{\varepsilon} \) and the applied stress \( \sigma \). In this relationship \( n \) is dependent on the average internal stress, \( \sigma_i \), and the average effective stress, \( \sigma_e \), during the creep test, as follows:
\[ n = \frac{(\sigma_o/\sigma_\xi + 1) (m + 1)}{[(m + 1 - \ell)/p] (\sigma_o/\sigma_\xi) + 1} \]  

(19)

where \( m \) is an exponent in the relation \( R \approx \sigma_i^m \). \( R = - (\partial \sigma / \partial t)_c \) is the recovery rate, \( \ell \) is given in \( \rho_m \approx \sigma_i^\ell \), and \( p \) is in the relation \( v \approx \sigma_c^p \). From our measurements for the 310 stainless steel and pure magnesium, \( p = 3 \) (here we assume that the average effective stress \( \sigma_c \) is proportional to the applied stress \( \sigma \)), and \( m = 6.5 \) and \( \ell = -1 \) have been measured by means of the stress dip test [165]. From the experimental measurements made by Ahlquist and Nix [165] and by Northwood and Smith [166] on various pure metals and solid solution alloys, the following relationships have been found for pure metals and Class II alloys undergoing creep at elevated temperatures:

\[ (\sigma_o/\sigma_\xi)_i > 1 \]  

(20)

and

\[ (\sigma_o/\sigma_\xi)_s = 0.2 \]  

(21)

where the outer subscripts \( i \) and \( s \) refer to the initial and the steady-state creep, respectively. This situation is shown schematically in Fig. 10. The internal stress during normal transient creep increases from an initial value close to zero, to a steady-state value having a magnitude of 0.85\( \sigma \). Such an analysis can be applied to our creep results since all of the creep strain-time curves for the 310 stainless steel and pure magnesium were of the normal transient type. The large value of \( (\sigma_c/\sigma_i) \) is due to a very small \( \sigma_i \) (close
to zero) and \( \sigma_c = \sigma \) upon loading. In steady-state creep \( \sigma_i = 0.85\sigma \) and \( \sigma_c = 0.15\sigma \), so that \( (\sigma_i/\sigma_c)_e = 0.2 \). With these data, estimates of the stress exponent values for the initial and the steady-state creep can be made. The results are \( n_i = 2.6 \) and \( n_c = 5.7 \) for our two materials. Given that a degree of uncertainty exists in the values of \( m \) and \( (\sigma_i/\sigma_c) \), these estimates for \( n_i \) and \( n_c \) agree well with our experimental measurements on the 310 stainless steel and pure magnesium. If one selects \( m = 3 \) as given by McLean’s network recovery creep model [183], then the estimates of the stress exponents are \( n_i = 2.4 \) and \( n_c = 3.6 \) which are still reasonable as compared to other creep experimental results on applied stress dependence of creep rates for pure metals and Class II alloys.

Equation (19) is based on the Bailey-Orowan recovery creep model [175, 157] and incorporates the internal and effective stresses [181]. For elevated temperature creep, strain hardening is controlled by the rate at which dislocations come into close proximity (storage of dislocations) through glide, while recovery occurs through dislocation rearrangement and annihilation which are controlled by dislocation climb. The applied stress, \( \sigma \), is composed of two terms, namely the internal stress, \( \sigma_i \), and the effective stress, \( \sigma_e \). That is \( \sigma = \sigma_i + \sigma_e \), where \( \sigma_i \) is associated with the recovery - strain hardening balance and determines the recovery rate, and \( \sigma_e \) is responsible for dislocation glide. Equation (19) is phenomenological in nature and describes both primary and steady state creep stages through the variation of the internal and effective stresses during creep experiment.

One point should be noted from the above discussion, namely that the initial creep rate is related to the applied stress by a power law equation with a stress exponent about
n, = 2.5. Because the mobile dislocation density $\rho_m$ in equation (15) is a constant for all applied stresses, the stress dependence of the initial creep rate is then solely due to a stress dependence of the average effective dislocation velocity, $v$. Therefore, measurement of the applied stress dependence on the initial strain rate can provide an indirect experimental means to study the applied stress dependence of the average effective dislocation velocity during recovery creep deformation at elevated temperatures. This point will be further discussed in the section 3.4 of Chapter IV on strain-hardening and recovery processes, in terms of the results predicted by using network models for recovery creep. The experimental approach and the theoretical approach arrive at very similar results with respect to the applied stress dependence of the average effective dislocation velocity. On the other hand, the applied stress dependence of the steady-state creep strain rate is determined by the stress dependencies of both the average effective dislocation velocity and the mobile dislocation density.
§3. Strain-Hardening and Recovery Processes

3.1. Introduction

In this section, the results are reported for the creep and stress relaxation tests that were used to study the strain-hardening and recovery behaviour during recovery creep of pure magnesium at elevated temperatures. The experimental results are discussed with respect to the dislocation network models for recovery creep. In the analysis, the applied stress dependence of steady-state creep rate, \( \dot{\varepsilon}_s \), the recovery rate, \( R \), the strain-hardening coefficient, \( H \), the average effective dislocation velocity, \( \nu \), and the dislocation annihilation rate, \( \dot{\rho}_w \), are obtained for recovery creep deformation of crystalline materials at elevated temperatures.

3.2. Creep Test Results

The typical creep results for pure magnesium are shown in Figs. 13(a) and (b). The creep strain-time curves were all of the normal type (N-type), i.e. decreasing creep rates with time from a larger initial creep rate upon loading to a smaller steady-state creep for all of the tests. This N-type transient creep curve is typical for pure metals and Class II alloys in which creep rate is determined by a recovery process (dislocation climb carried out by vacancy diffusion). This N-type of creep behaviour is also termed recovery creep. Figs. 13(a) and (b) show that creep rate is increased with an increase in either the applied stress at a constant temperature, or an increase in test temperature under a constant applied stress. The stress and temperature dependence of the steady-state creep rate shown in Figs. 14(a) and (b) give an applied stress exponent, \( n = 5.9 \), for pure magnesium crept at 200 °C under an applied stress range from 20 MPa to 50 MPa.
Fig. 13  (a) Typical creep strain versus time curves showing a normal type (N-type) transient for pure magnesium crept at various test conditions: T = 200 °C and $\sigma$

= (1) 20 MPa, (2) 25 MPa, (3) 35 MPa, (4) 40 MPa, (5) 50 MPa;
Fig. 13  (b) Typical creep strain versus time curves showing a normal type (N-type) transient for pure magnesium crept at various test conditions: $\sigma = 35$ MPa and $T$

$= (1) 150 \, ^\circ C, (2) 200 \, ^\circ C, (3) 225 \, ^\circ C, (4) 250 \, ^\circ C$. 
Fig. 14 Temperature dependence of the steady-state creep rate, $\varepsilon_s$, for pure magnesium: $\ln \varepsilon_s$ versus $1/T$ given an activation energy for creep, $Q_c = 106$ kJ/mole for tests under $\sigma = 35$ MPa and $T = 150$ °C to 250 °C. Applied stress dependence of the steady-state creep rate, $\varepsilon_s$, for pure magnesium: $\log \varepsilon_s$ versus $\log \sigma$ gives a stress exponent $n = 5.9$ for tests at $T = 200$ °C and $\sigma = 20$ MPa to 50 MPa, see Fig. 11(b) (p. 60):
This stress dependence of steady-state creep rate is typical for recovery creep and will be discussed in more detail later. Fig. 14(b) is a plot of the natural logarithm of the steady-state creep rate, $\ln \varepsilon_s$, versus the reciprocal absolute creep temperature $1/T$. The linear relationship between $\ln \varepsilon_s$ and $1/T$ gives an apparent activation energy for creep, $Q_c = 106$ kJ/mole. This measured value is smaller than that for self-diffusion, $Q_c = 135$ kJ/mole, and is close to, but slightly higher than, that for grain boundary diffusion or dislocation core diffusion, $Q_{gb} = 92$ kJ/mole for pure magnesium ($Q_{gb}$ is measured at lower temperatures) [22]. As has been discussed in section 1.3 of this chapter, the magnitude of the apparent activation energy, $Q_c$, for a given crystalline material is dependent upon the homologous temperature, $T/T_m$ [7, 18, 22]. Generally, $Q_c$ increases with an increase in $T/T_m$. For instance, it has been reported that the ratio $Q/Q_c = 1$ when $T/T_m \geq 0.7$, whereas $Q_c/Q_c = 0.6$ to 1 when $T/T_m = 0.5$ to 0.7 [60]. The test temperatures employed by us for pure magnesium were in the range $T/T_m = 0.46$ to 0.57 and the measured $Q_c$ value ($Q_c/Q_c = 0.79$) is consistent with previous experimental results, and remains essentially constant over the temperature range tested.

3.3. Stress Relaxation Test Results

The stress relaxation test results are given in Figs. 15(a) and (b) for 200 °C under different initial creep stresses (20 MPa to 50 MPa), and at 35 MPa at different test temperatures (150 °C to 250 °C), respectively. The temperature and applied stress values shown in Figs. 15(a) and (b) were for the steady-state creep tests. The current stress for the stress relaxation tests were normalized by the initial creep stress, and are plotted as a function of relaxation time. Figs. 15(a) and (b) clearly show that stress relaxation rate
Fig. 15 Typical stress relaxation test results are shown as load ratio versus relaxation time curves for pure magnesium. The load ratio is equal to the current load during stress relaxation test normalized by the corresponding initial load for steady-state creep. (a) $T = 200 \, ^\circ$C and $\sigma = (1) \, 20 \, \text{MPa}, (2) \, 25 \, \text{MPa}, (3) \, 35 \, \text{MPa}, (4) \, 40 \, \text{MPa}, (5) \, 50 \, \text{MPa}$.
Fig. 15  Typical stress relaxation test results are shown as load ratio versus relaxation time curves for pure magnesium. The load ratio is equal to the current load during stress relaxation test normalized by the corresponding initial load for steady-state creep. (b) \( \sigma = 35 \) MPa and \( T = (1) 150 ^\circ \text{C}, (2) 175 ^\circ \text{C}, (3) 200 ^\circ \text{C}, (4) 225 ^\circ \text{C}, (5) 250 ^\circ \text{C}. \)
process is accelerated by increasing either the initial (creep) applied stress or the test temperature. That is to say that an increase in steady-state creep rate (obtained by an increase in either stress or temperature) will result in an increase in the stress relaxation rate. It should be pointed out that the present stress relaxation tests were carried out following steady-state creep of the material. Therefore, the stress relaxation behaviour at the relaxation time \( t = 0 \) gives a measure of the strain-hardening and recovery processes for the corresponding steady-state creep. The dynamic recovery rate values, \( R \), were obtained from the stress relaxation data by taking the negative slope of stress versus relaxation time curves at the onset of the stress relaxation tests as given by the definition of the dynamic recovery rate \([1, 75, 184]\), i.e. \( R = - (\partial \sigma / \partial t)_{\text{def}} \). The dynamic recovery rate values, \( R \), are given in Figs. 16(a) and (b). The stress dependence of the recovery rate is similar to that of the steady-state creep rate, c.f. Fig. 14(a) or 11(b) and Fig. 16(a). A linear relationship between dynamic recovery rate and applied stress when plotted on a log-log scale gives a stress exponent for recovery rate of \( n_r = 5.8 \) with \( n_{LR} = 0.989 \) in \( R = R_0 (\sigma / \mu)^n \exp(-Q_r / kT) \) where \( R_0 \) is a constant and \( Q_r \) is activation energy for recovery, for pure magnesium at 200 °C. This \( n_r \) value is approximately equal to the stress exponent for steady-state creep rate, \( n = 5.9 \). Fig. 16(b) is a plot of the natural logarithm of the dynamic recovery rate, \( \ln R \), versus the reciprocal of the absolute test temperature, \( 1/T \), which gives an activation energy for dynamic recovery, \( Q_r = 88.5 \) kJ/mole with \( n_{LR} = 0.988 \). This low value of \( Q_r \) can be rationalized as follows. In terms of temperature dependence, the steady-state creep rate, \( \dot{\varepsilon}_s \), the dynamic recovery rate, \( R \), and the strain-hardening coefficient, \( H \), can be expressed conveniently by an exponential term
Fig. 16  (a) Applied stress and temperature dependencies of the dynamic recovery rate, $R$, measured by the stress relaxation tests for pure magnesium: log$R$ versus log$\sigma$ gives a stress exponent $n_r = 5.8$ for $T = 200 \degree C$ and $\sigma = 20 \text{ MPa}$ to 50 MPa;
Fig. 16  (b) Applied stress and temperature dependencies of the dynamic recovery rate, 
R, measured by the stress relaxation tests for pure magnesium: \( \ln R \) versus \( 1/T \) 
gives an apparent activation energy for dynamic recovery, \( Q_r = 88.5 \text{ kJ/mole} \) for 
\( \sigma = 35 \text{ MPa} \) and \( T = 150 ^\circ \text{C} \) to \( 250 ^\circ \text{C} \).
(Boltzmann factor), \( \exp(-Q/kT) \), where \( Q_i \) stands for \( Q_e \), \( Q_r \), and \( Q_h \), respectively. According to the Bailey-Orowan equation [75, 157], where \( \varepsilon_a = R/H \), then one has \( Q_e = Q_r - Q_h \). It will be shown in the next section, 3.4, that the strain-hardening coefficient, \( H \), is a decreasing function of the absolute temperature, which will give a negative activation energy for strain-hardening process, \( Q_h = -18 \text{ kJ/mol} \). This negative \( Q_h \) shows that strain-hardening process is not a real thermally activated process, but rather it is a stress activated process in which work is done by the applied stress. Then \( Q_h \) is related to the quantity, \( -bA^-\tau \), where \( \tau \) is the shear stress in slip direction, \( b \) is the Burger's vector, and \( A^- \) is the area swept out by the dislocation segment during an activation event under \( \tau \) (activation area). Or, in other words, \( Q_h \) is equal to the work done by the shear stress \( \tau \) during an activation event for dislocation glide in the strain-hardening. Experimental measurements on activation area \( A^- \) for the creep of several monocrystalline and polycrystalline materials have shown that \( A^- \) is a decreasing function of \( \tau \) and the term \( bA^-\tau \) is essentially a constant [57]. The activation area \( A^- \) of the "constant structure creep" is proportional to the reciprocal effective stress [62].

3.4. Strain-Hardening Rate

The values of strain-hardening, \( H \), were obtained from the steady-state creep rate, \( \varepsilon_a \), and dynamic recovery rate, \( R \), by using the Bailey-Orowan equation, viz. \( \varepsilon_a = R/H \) [75, 157]. The results are given in Figs. 17(a) and (b). In Fig. 17(a) it is seen that the strain-hardening coefficient, \( H \), is almost a constant \( (H = 10.9 \pm 1.5 \text{ GPa} = 0.27E) \) at 200 °C for different initial applied creep stress (20 MPa to 50 MPa). It is in the range given by Evans \( (H = 0.16E \text{ to } 0.36E) \) based on a dislocation network model for strain-hardening.
Fig. 17 Strain-hardening rate, $H$, values were calculated from creep and stress relaxation test data for pure magnesium: (a) $H$ versus $\sigma$ data show that $H$ is a constant ($H = 0.27E$) and is essentially independent of applied stress, $\sigma$;
Fig. 17 Strain-hardening rate, $H$, values were calculated from creep and stress relaxation test data for pure magnesium: (b) $H$ and $E/H$ versus $T$ curves show that $H$ is a decreasing function of temperature, and that $H$ decreases faster than $E$ (or $E/H$ ratio increases) when $T$ increases.
of high temperature recovery creep deformation [185], and the range from about 0.1E to 1.0E measured during steady-state creep by using the stress increment test for many crystalline materials [145, 186]. This constant value for the strain-hardening coefficient will be discussed further in the section 3.5, and modelled by using a dislocation link length statistical model. The temperature dependence of the strain-hardening coefficient is given in Fig. 17(b). It is seen that H is a decreasing function of test temperature. H values decrease from 20.8 GPa to 7.4 GPa when the test temperature is increased from 150 °C to 250 °C. One may expect that the elastic modulus, E, is also a decreasing function of temperature. The E/H ratio was also calculated and included in Fig. 17(b), which shows that the E/H ratio increases with increasing temperature. In Fig. 17(b), the E values were calculated from data given in reference [22] for pure magnesium. From Fig. 17(b), it is seen that the H values decrease from 0.50E to 0.19E when the test temperature increases from 150 °C to 250 °C for the same initial value of creep stress (35 MPa).

3.5. Discussion of Results for Strain-Hardening and Recovery Processes

The steady-state creep rate, \( \dot{\varepsilon}_s \), for recovery creep deformation in dislocation creep regime at elevated temperature of pure metals and Class II alloys is commonly described by following equations:

(i) The Bailey-Orowan equation [75, 157]:

\[
\dot{\varepsilon}_s = \frac{R}{H} \tag{22}
\]

where \( R = - (\partial\sigma/\partial t)_c \) and \( H = (\partial\sigma/\partial e)_c \). This is the fundamental equation for the traditional
theory of recovery creep. It states that the steady-state creep is an outcome of the dynamic balance between strain-hardening and recovery processes. As was pointed out by Orowan himself [75], an assumption is made in the theory that R depends only on the temperature and the stress. If this assumption is made, equation (22) demands, at constant temperature and stress, a constant rate of creep, and this cannot be reconciled with the deceleration and the extremely high rate of flow at the beginning of creep. In order to overcome this difficulty, it can be assumed that R depends also on the entire stress-strain history of the specimen. Equation (22) can only be properly applied to the steady-state deformation stage in which the strain rate is a constant. It can not be applied directly to transient creep in which the creep rate is a function of time. This aspect of the Bailey-Orowan equation has been subsequently demonstrated by introducing the internal stress concept on both experimental and theoretical grounds [187, 188] and the dislocation network model [147] to high temperature recovery creep deformation.

(ii) The Taylor-Orowan equation [172, 173]:

\[
\dot{\varepsilon}_s = abp_m v
\]  

(15)

This basic equation, defines the strain rate in terms of mobile dislocation motion, and as such provides a basis for the description of dislocation plasticity of crystalline materials. It is a general equation that can be applied to the entire dislocation plasticity of crystalline materials regardless of the experimental test conditions (constant stress creep test as well as constant strain rate tensile test). However, this equation describes only the average kinetic properties of dislocation plasticity. The characteristic behaviour of dislocation
motion is not specifically concerned with that for a particular dislocation segment.

(iii) The power law creep equation (Dom equation) [59, 66, 189]:

\[
\dot{\varepsilon}_s = A \frac{D_\nu b}{kT} \left( \frac{\sigma}{\mu} \right)^n
\]  

(23)

which is based upon empirical studies on creep of many crystalline materials. Theoretical development of this equation is based upon dislocation network models for recovery creep, such as by Gittus [190] and by Evans and Knowles [60]. These theoretical considerations of the power law creep equation (23) have exercised some success when compared to experimental creep measurements on a variety of crystalline materials, except for a few instances. Data for parameters in the above equation have been compiled so conveniently for various crystalline materials (metals and ceramics) in the Deformation-Mechanism Maps by Ashby [22, 191].

The stress dependencies of the quantities in the above three equations such as \(\dot{\varepsilon}_s\), \(\rho\), v, \(R\), and \(H\) were studied for pure magnesium. The experimental results show that \(n = 5.9\) for \(\dot{\varepsilon}_s\): Fig. 11(b); and \(n_r = 5.8\) for \(R\): Fig. 16(a). From equations (22) and (23) it is clear that \(H\) is almost independent of the applied stress, since both \(\dot{\varepsilon}_s\) and \(R\) are proportional to \(\sigma^0\) and \(H = R/\dot{\varepsilon}_s\), which is consistent with the experimental results in Fig. 17(a). Then the steady-state creep rate, \(\dot{\varepsilon}_s\), for high temperature recovery creep is primarily determined by the recovery rate, \(R\). The present experimental results support dislocation network models for recovery creep deformation [185, 192] which predict that the strain-hardening coefficient is independent of the applied stress during steady-state recovery
creep deformation.

In a dislocation network model of recovery-controlled creep, Ajaja [193] has shown that the average effective dislocation velocity in steady-state creep is linearly proportional to the annihilation rate, \( \dot{\rho}_a = - (\partial \rho / \partial t) \), through the equation:

\[
v = \psi_s \rho^{-3/2} \dot{\rho}_a
\]  

(24)

where \( \psi_s \) is a constant related to the distribution of dislocations. Combining equations (15) to (17), (23), and (24) yields the stress dependence of the annihilation rate, \( \dot{\rho}_a \), namely:

\[
\dot{\rho}_a = \frac{AD_s \mu b}{a_2 \Sigma M \psi_s b^2 kT} \left( \frac{\sigma}{\mu} \right)^{n+1}
\]  

(25)

It is clear from equation (25) that the annihilation rate, \( \dot{\rho}_a \), has a stronger applied stress dependence than that of steady-state creep rate, \( \dot{e}_s \), c.f. equations (23) and (25). Substituting equations (16) and (25) into equation (23) one finds the average effective dislocation velocity during recovery creep in steady-state stage is related to the applied stress by the equation:

\[
v = \frac{a_2 \Sigma M b}{aZ} \frac{AD_s \mu b}{kT} \left( \frac{\sigma}{\mu} \right)^{n-2}
\]  

(26)

which shows that during steady-state creep in magnesium, the average effective dislocation velocity is proportional to the third power of applied stress, since in equation
n = 5 for recovery creep. This results predicted from network models are similar to
the experimental measurements on the 310 stainless steel and pure magnesium reported
in section §2 of this chapter. In that section, it is seen that the stress dependence of the
initial creep rate gives an indirect measurement to the stress dependence of the average
effective dislocation velocity. The experimental results give \( n_i = 2.8 \) for pure magnesium
and \( n_i = 2.1 \) for the 310 stainless steel, which are close to the results from equation (26).

In a study of the Bailey-Orowan equation, a dislocation network model of recovery
creep [193] has been employed to obtain the strain-hardening coefficient, \( H \), and recovery
rate, \( R \) [147]. The results are given by the following equations:

\[
R = \frac{\alpha_i \mu b}{2 \rho^{3/2}} \dot{\rho}_e
\]  

(27)

and

\[
H = \frac{\alpha_i \mu}{2 \alpha_2 \rho^{3/2} L} \mu
\]  

(28)

respectively. Where \( \alpha_2 \) is a numerical constant, and \( L \) is the obstacle spacing during
dislocation glide. The obstacles for single phase materials are the network dislocations.
The relation between \( L \) and \( \rho \) has been found to follow \( L = \beta \rho^{-1/2} \), where \( \beta \) is a constant
about unity depending on the geometry of the three-dimensional dislocation network
structure in which dislocation link lengths take a typical statistical distribution [194-196].
By combining equations (27) and (28) with equations (16) and (25), the present author
finds that the strain-hardening coefficient, \( H \), and recovery rate, \( R \), are given by the
following two equations:

\[ R = \frac{\alpha_1 M \mu}{2 \alpha Z \psi_s} \frac{A D_p \mu \beta}{k T} \left( \frac{\sigma}{\mu} \right)^n = \frac{\alpha_1 M \mu}{2 \alpha Z \psi_s} \dot{\varepsilon}_s \]  \hspace{1cm} (29)

and

\[ H = \frac{\alpha_1 M}{2 \alpha_2 \beta} \mu \]  \hspace{1cm} (30)

respectively. According to the Bailey-Orowan equation, \( \dot{\varepsilon}_s = R/H \). The constants in the above equations have the following relationship, namely \( \alpha Z \psi_s/(\alpha_2 \beta) = 1 \). From equations (29) and (30) it can be seen clearly that during steady-state recovery creep deformation at elevated temperatures: (i) the steady-state creep rate is directly proportional to the recovery rate (applied stress \( \sigma \) equals the internal stress \( \sigma_i \)); and (ii) the strain-hardening coefficient is a constant independent of the applied stress for the corresponding steady-state creep, and is of the order of shear modulus. These relationships have been observed in the present experimental results measured for pure magnesium. Furthermore, a constant strain-hardening coefficient of the order of shear modulus during high temperature plastic deformation has also been predicted by other strain-hardening models [197-200]. It will be further discussed that equations (29) and (30) are similar to results from the dislocation link length statistical treatment in a micromechanical modelling section 2.3 of Chapter V.

The above equations, namely equations (23), (25), (26), (29) and (30), clearly match the experimental applied stress dependencies of the steady-state creep rate, \( \dot{\varepsilon}_s \), the dislocation annihilation rate, \( \rho_s \), the average effective dislocation velocity, \( v \), the recovery rate, \( R \), and
the strain-hardening coefficient, H, respectively, during elevated temperature recovery
creep deformation. The difference between Class I creep (n = 3) and Class II creep (n =
5) is that the average effective dislocation velocity is linearly proportional to the applied
stress in Class I creep (viscous glide), and is proportional to the third power of applied
stress in Class II creep (jerky glide). The jerky glide is composed of two consecutive
processes, namely thermally activated diffusional climb (slow motion) and subsequent
athermal jerky glide (fast motion).
CHAPTER V  MICROMECHANICAL MODELS

§1. Öström-Lagneborg Creep Model

Many direct observations have shown that the dislocation microstructure is in the form of a three-dimensional network during recovery creep. Such a network has been found for instance in pure α-Fe crept at 823 K [201], Cu-10 at% Ni alloy crept at 873 K [202], an austenitic stainless steel crept at 973 K [203], high purity Al [204], creep deformed polycrystalline MgO [195], monocrystalline Al and NaCl [205], and pure polycrystalline Ni [148], by means of transmission electron microscopy, slip lines on polished surfaces, and etch pit techniques. It is found that the link lengths in the network have a typical statistical distribution. This information has been used to describe creep deformation process by link length distribution (network) models [45].

Among dislocation network models for creep, the Öström-Lagneborg (Ö-L) creep model [145] provides a clear physical picture of the three-dimensional dislocation network and its dynamic evolution during recovery creep. Apart from its limitation on dislocation network coarsening kinetics [45, 146], the model has a considerable predictive capability. It has been shown that the model is able to simulate a large number of the characteristics of both transient and steady-state creep as demonstrated by the model results [145]. In this section, the Ö-L creep model is applied to simulate the creep results of the 310 stainless steel. An extension of the model is attempted in order to describe the subgrain formation during creep deformation.

1.1. The Model and Its Formulations

In the Ö-L creep model [145], the recovery creep process is considered to consist
of the following three subprocesses which take place in the dislocation network: (1) By climb controlled shrinkage of small meshes and growth of large meshes, the average mesh size will increase (recovery process); (2) Under the action of the applied stress, the growing dislocation links will be released by the breakage of junctions in the network, when they have attained an appropriate length \( \lambda \geq \lambda_n \), where \( \lambda_n \) is the threshold link length. The released links expand to loops by glide and thereby generate creep strain (release process); and (3) The expanding loops will be blocked and partitioned into shorter links by adjacent parts of the network, which become immobile again. New shorter links are then supplied (supply process). The above three subprocesses renew themselves continuously and at the same time produce a creep strain rate.

In the dislocation network structure there exists a threshold link length \( \lambda_n = 2 \mu b / \sigma \) [145]. This result for the threshold link length \( \lambda_n \) is obtained by considering slip geometry and crystallographic orientation for slip systems of polycrystalline materials. The three-dimensional dislocation network during creep is characterized by a distribution frequency function, \( \phi(\lambda, t) \), which is defined such that the number of links per unit volume with length from 0 to \( \lambda \) is \( N(\lambda, t) \) and the total dislocation density is \( \rho(t) \) at time \( t \):

\[
N(\lambda, t) = \int_0^\lambda \phi(\lambda, t) \, d\lambda
\]

(31)

and

\[
\rho(t) = \int_0^\infty \lambda \phi(\lambda, t) \, d\lambda
\]

(32)
The first two processes, namely shrinkage and growth, of the individual links were assumed to follow the network coarsening kinetic equation analogous to the grain growth theory [206-208]:

$$\frac{d\lambda}{dt} = M^* \Gamma \left( \frac{1}{\lambda_{cr}} - \frac{1}{\lambda} \right)$$

(33)

where $M^*$ is the mobility of the climb dislocations, $\Gamma$ is the line tension of the dislocations, $\lambda_{cr}$ is a critical link length where the shorter links ($\lambda < \lambda_{cr}$) will shrink ($d\lambda/dt < 0$) and longer links ($\lambda_{cr} < \lambda < \lambda_{mb}$) will grow ($d\lambda/dt > 0$) as well as the links ($\lambda \geq \lambda_{mb}$) will glide. The model describes the development of the distribution frequency function $\phi$ with time during creep and derives all other relevant outputs (the creep strain rate, the creep strain, the mobile and immobile dislocation densities, etc.) from the evolution of this link length distribution. The model equations include (1) the network structure equation; (2) the boundary conditions for the network structure equation; and (3) the strain rate and dislocation density kinetic equations [145]. The Öström-Lagneborg creep model equations are given as follows:

1. The network structure equation:

$$\frac{\partial \phi}{\partial t} = \left( \frac{\partial \phi}{\partial t} \right)_1 + \left( \frac{\partial \phi}{\partial t} \right)_2 + \left( \frac{\partial \phi}{\partial t} \right)_3$$

(34)

$$\left( \frac{\partial \phi}{\partial t} \right)_1 = - \left\{ \frac{d\lambda}{dt} \frac{\partial \phi}{\partial \lambda} - \frac{M^* \Gamma}{\lambda^2} \phi \right\}$$

(34A)
\begin{equation}
\left( \frac{\partial \phi}{\partial t} \right)_2 = - H(x) \frac{q - 1}{\lambda} \frac{d\lambda}{dt} \phi(\lambda, t) \tag{34B}
\end{equation}

\begin{equation}
\left( \frac{\partial \phi}{\partial t} \right)_3 = C(t) \lambda \left\{ \frac{3}{k^2(t)} \phi \left( \frac{\lambda}{k(t)}, t \right) - \phi(\lambda, t) \right\} \tag{34C}
\end{equation}

where $H(x)$ is the Heaviside unit function defined by $H(x) = 1$ when $x \geq 0$ and $H(x) = 0$ when $x < 0$. $x = \lambda - \max(\lambda_{ct}, \lambda_{th})$. $q$ is a constant. $C(t)$ and $k(t)$ are respectively the probability function and the network geometry function in the supply process. The subscripts 1, 2, 3 refer to the three subprocesses in the model, namely recovery, release, and supply processes.

(2). The boundary conditions for the network structure equation (both recovery and strain-hardening processes were assumed to satisfy the volume conservation condition):

\begin{equation}
\int_0^\infty \lambda^3 \left( \frac{\partial \phi}{\partial t} \right)_1 d\lambda = \int_0^\infty \lambda^3 \left\{ \left( \frac{\partial \phi}{\partial t} \right)_2 + \left( \frac{\partial \phi}{\partial t} \right)_3 \right\} d\lambda = 0 \tag{35}
\end{equation}

\begin{equation}
\lambda_{ct}(t) = \int_0^\infty \lambda^2 \phi(\lambda, t) d\lambda / \int_0^\infty \lambda \phi(\lambda, t) d\lambda \tag{36}
\end{equation}

(3). The strain rate and dislocation density kinetic equations during creep (the expanding dislocation loops are considered as circular in the model):

\begin{equation}
\frac{d\varepsilon}{dt} = - \frac{\pi r^2 b}{M} \int_0^\infty \left( \frac{\partial \phi}{\partial t} \right)_2 d\lambda \tag{37}
\end{equation}
\[
\frac{d\rho}{dt} = -(2\pi r) \int_0^\infty \left( \frac{\partial f}{\partial \tau} \right) d\lambda
\]  \hspace{1cm} (38)

where \( r(\sigma, t) = L(\sigma) \rho^{1/2} \) is the average radius of circular dislocation loops, \( M \) is the Taylor factor, and \( L \) is a stress dependent factor.

The Ő-L model will now be applied to simulate the creep behaviour of the 310 stainless steel. It is shown that this model is able to simulate the creep behaviour of the material in that the calculated results agree well with the experimental results and with other independent analyses made by other investigators. The model will be also evaluated in the light of recent elastic theories of the subgrain boundaries and the recent experimental creep results. The limitations of the model are identified for dislocation network recovery coarsening kinetics.

1.2. Simulation of the Experimental Results

The results for the 310 stress dependence of creep in the stainless steel are given in Fig. 12. The temperature dependence of the steady-state creep rate for the 310 stainless steel (200 MPa, 650 °C to 800 °C) is given in Fig. 8(b) at several creep strains. Here the apparent activation is measured for another two applied stresses (170 MPa and 230 MPa). The results are given in Fig. 18, together with data for 200 MPa. From Fig. 18 it is seen that the activation energy for steady-state creep at the three applied stresses is equal to 341 kJ/mole and is independent of the applied stress for present set of creep tests (170 MPa to 230 MPa, 650 °C to 800 °C).
Fig. 18  A plot of \( \ln \dot{\varepsilon} \) versus \( 1/T \) for the 310 stainless steel crept at three applied stresses, \( \sigma = (1) 170 \text{ MPa}, (2) 200 \text{ MPa}, (3) 230 \text{ MPa} \) over a temperature range of 650 °C to 800 °C. The results show that the apparent activation energy for creep, \( Q_c \), is essentially independent of the applied stress, \( \sigma \), for the present test conditions.
The parameters for the numerical calculation are as follows (for σ = 110 MPa and T = 973 K): b = 2.5 \times 10^{-10} \text{ m}, \mu = 6.1 \times 10^{10} \text{ Pa}, \rho_o = 7.9 \times 10^{12} \text{ m}^2, \text{ and } I_o = 0.7, \text{ where } \mu \text{ was measured for the 310 stainless steel using a tensile test at 700 °C, and } I_o \text{ is the volume integral constant. Since the steady-state creep is independent of the initial state [145], the initial dislocation density } \rho_o \text{ and the initial distribution frequency function } \phi_o \text{ were taken to be the same as given in the earlier work [145]. The finite difference method [158] was used to the numerical integration of the model equations (34) to (38). An equal space step in } \lambda \text{ and an increasing time step in } t \text{ were used in order to reduce the computation time. This change in time step is in accordance with the finding that the variation in } \phi(\lambda, t) \text{ is larger at early stage of creep and gradually becomes smaller. The fit between experimental and calculated results is obtained for } L = 4.5 \text{ and } M^* = 3.2 \times 10^8 \text{ Pa}^{-1} \text{s}^{-1}. \text{ The initial and steady-state values of the model functions } k(t), C(t) \text{ and } \lambda_{cr}(t) \text{ are: } k_i = 0.715, C_i = 2.4 \times 10^5 \text{ m}^{-1} \text{s}^{-1}, \lambda_i = 3.96 \times 10^{-7} \text{ m}; \text{ and } k_o = 0.762, C_o = 2.1 \times 10^4 \text{ m}^{-1} \text{s}^{-1}, \lambda_o = 1.4 \times 10^{-7} \text{ m}. \text{ The outputs of the calculation include the change of } \phi(\lambda, t) \text{ with time, the creep strain } \varepsilon(t), \text{ the total dislocation density } \rho(t), \text{ the mobile and the immobile dislocation densities } \rho_m(t) \text{ and } \rho_i(t), \text{ respectively. They are presented in Figs. 19 to 21. As is shown in Fig. 19 the agreement between the calculated and experimental creep curves is good. Also, the calculated values of } \phi(\lambda, t) \text{ and } \rho(t) \text{ are in fair agreement with earlier results [145, 208]. From Fig. 21 it is seen that } (\rho_m/\rho)_o = 0.2 \text{ for steady-state creep. This value is in the range 0.15 to 0.25 as given by Ajaja [180] in his analysis which is also based on the link length distribution. It is also close to the value estimated by Kassner [177] on the basis of the Taylor-Orowan dislocation dynamics [172, 173] whereby during}
Fig. 19  Calculated (dotted curve) and experimental creep data (symbol) of creep strain versus time for the 310 stainless steel crept at an applied stress of $\sigma = 110$ MPa and a test temperature of $T = 700$ °C.
Fig. 20  The calculated distribution function, $\phi(\lambda, t)$: (1) initial state, (2) at time $= 2.5$ minutes, and (3) steady-state creep.
Fig. 21  Variation of (a) total dislocation density, $\rho_t(t)$, (b) the immobile dislocation density, $\rho_i(t)$, and (c) the mobile dislocation density, $\rho_m(t)$, with creep time.
steady-state creep only about 1/3 of the total dislocations are mobile. This will also be
calculated quantitively by using a dislocation link length statistical model, in the section
2.2 of this chapter where it will be shown that \( \frac{\rho_m}{\rho_s} = 0.218 \).

For varying applied stresses and temperatures, the two sets of creep curves were
calculated, one at 700 °C and stresses of 110, 230, 260 and 290 MPa, and another at 200
MPa and temperatures of 700, 750 and 775 °C. In the calculation, \( \rho_s \) and \( L \) were assumed
to be applied stress dependent via \( \rho_s \propto \sigma^2 \) and \( L \propto \sigma^{-1} \), and \( M^* \) is assumed to be
temperature dependent [145]. In order to obtain a good fit between the experimental data
and the calculated results, the release term was multiplied by a subgrain function \( S_g(t) \),
which is equals unity at the onset of creep and increases during transient creep to a value
of about 2 in steady-state creep. The results are presented in Figs. 22(a) and (b). In
general, agreement between the calculated and experimental curves is reasonably good,
and the fit is better for lower applied stresses and temperatures. From the calculations, the
following observations were made. Temperature affects creep primarily through increasing
the mobility of the climb dislocations, \( M^* \), which is in accordance with the Arrhenius
equation. Applied stress affects creep behaviour through various parameters related to the
dislocation network structure. At 700 °C, when \( \sigma \) increases from 100 MPa to 290 MPa:
(i) steady-state \( S_s \) increases from 1 to 1.85; \( C(t) \) increases from \( 2.4 \times 10^5 \) to \( 6.2 \times 10^5 \) m
\( ^1 \)s\(^{-1} \) (initial values) and from \( 2.1 \times 10^4 \) to \( 5.3 \times 10^4 \) m\(^1 \)s\(^{-1} \) (steady-state values); \( \lambda_{cr}(t) \)
decreases from \( 3.96 \times 10^{-7} \) m to \( 1.54 \times 10^{-7} \) m (initial values) and from \( 1.4 \times 10^{-7} \) m to
\( 0.55 \times 10^{-7} \) m (steady-state values); (ii) the steady-state total dislocation density, \( \rho_s \), is
related to the applied stress \( \sigma \) by \( \rho_s \propto \sigma^2 \), which shows that the creep (flow) stress is
Fig. 22 Experimental creep data (symbols) for the 310 stainless steel and the calculated results (dotted curves): (a) T = 700 °C for three applied stresses, σ = (1) 110 MPa, (2) 260 MPa, and (3) 290 MPa;
Fig. 22  Experimental creep data (symbols) for the 310 stainless steel and the calculated results (dotted curves): (b) $\sigma = 200$ MPa for three test temperatures, $T = (1) 700 ^\circ C$, (2) $750 ^\circ C$, and (3) $775 ^\circ C$. 
primarily determined by the network dislocations: the ratio of the mobile dislocation density to the total dislocation density in steady-state, \( (\rho_m/\rho_i) \), is in a range from 0.18 to 0.23, and is essentially independent of \( \sigma \) and \( T \); and (iii) \( k(t) \) is in the range from 0.701 to 0.783 for all the applied stresses. This shows that the network dislocation structure can be visualized as a homogeneous structure for the 310 stainless steel under the present creep testing conditions.

The dislocation mobility \( M' \) value can be used to check the validity of dislocation network coarsening kinetics, equation (33). This is made possible by an estimation of the incubation time (the time interval with zero strain rate) after a small stress reduction at steady-state stage. From equation (33), it can be shown that this incubation time \( \Delta t \) is given by [145]:

\[
\Delta t = \frac{\Delta \lambda}{(d\lambda/dt)_{\lambda_{th}}} = \frac{\lambda_{ch}(-\Delta \sigma/\sigma)}{M'T(1/\lambda_{cr} - 1/\lambda_{ch})}
\]  

(39)

For a stress reduction of 1% \((-\Delta \sigma/\sigma = 10^{-2})\), the value of \( \Delta t \) = 13.0 s. This value is substantially larger than the experimental measured value which is about 1.5 seconds [145, 208]. This may be due to the fact that the network coarsening kinetics equation (33) underestimates the dislocation mobility \( M' \) value.

1.3. Discussion and Summary

As has been pointed out by Lagneborg [209], there are a number of widely accepted theories for recovery creep deformation. These include Weertman's dislocation climb models [167, 210], models based on thermally activated motion of jogged screw
dislocations [167, 211-214], and models based on the climb process of extended dislocations [30-32, 215, 216]. These theories have cast light upon the climb recovery, and dislocation dynamics in dislocation creep, and are consistent with the experimental results for the activation energy, the stress exponent of steady-state creep rate, and the stacking fault energy effect [7]. However, the drawbacks of these models are also well-known, e.g. whether dislocations on parallel slip planes and of opposite sign will be held up by mutual interaction and form pile-ups as in the climb model [18], and that the uncertainty of the mobile dislocation density $\rho_m$ is great, with respect to both its magnitude and stress dependence in the dislocation-jog models. Furthermore, the stress dependence of the spacing between the jogs is not considered. On the other hand, the network models, especially the recent ones, are based upon a detailed description of the evolution of the distribution of link lengths, and are far more appealing on physical grounds. One of the principal advantages of such models is that out of all the existing dislocation links, it automatically and in a physically-sound fashion "selects" the few mobile links when it comes to deriving the creep strain rate.

However, the link length distribution models are not complete and final. There are one or two approximations made in the models, which relate to the subgrain formation and the network recovery coarsening kinetics. The first assumption is that the distribution frequency function $\phi(\lambda, t)$ only takes into account those free dislocation links not incorporated in subgrain boundaries. It now seems that this is a justifiable approximation from the point of view of Li's elastic theories of subgrain boundaries [217-221] and recent experimental observations on high temperature deformation [177, 222-227].
uniform array of dislocations is inherently energetically unstable and tends to form a modulated cell structure according to both experimental and theoretical observations [228-231]. High temperature deformation of an Al-Mg-Mn alloy has been carried out [227] in plain-strain compression at constant strain rate and temperature, with abrupt changes in the Zener-Hollomon parameter, \( Z = \varepsilon \exp(\Delta H/RT) \), where \( \Delta H \) is activation enthalpy, \( R \) is the gas constant. It was found that the proof strength depends primarily on the dislocation density within the subgrains (free dislocations) [227]. The subgrain size played a lesser role. This is interpreted in terms of the fact that dislocations within subgrain boundaries have a lower strengthening efficiency per dislocation. The lesser role of the subgrain size can be considered in terms of a Hall-Petch contribution, with the subgrain-boundary strength being much less than the strength of grain boundaries [227]. In fact, experiments show that characteristic networks of dislocations tend to form in creep. The networks outline cells within which there are relatively few free dislocations. As the networks do not exert significant long-range stresses, most of the free dislocations within the cells are unaffected by the stresses produced by dislocations in the cell walls [206]. They are, however, subject to the internal stress produced by the line tension of the dislocation link itself [232]. Since the effects of the array are all short-range in nature, it can be said that an infinite edge dislocation array is a weaker barrier for the penetration of parallel edge dislocations than the same set of edge dislocations which composed the array but are distributed randomly [220]. The numerical analysis of the interaction of a dislocation wall (tilt substructure boundary) with parallel approaching dislocations where slip vectors are the same as those of the dislocations in the wall shows that such a wall,
whether fully or partially pinned, is a less effective barrier for the penetration of dislocations than a single pinned dislocation. Free dislocations in the wall usually reduce the resistance of the wall to the penetration of approaching dislocations [217]. Another series of calculations have also been made of the short-range stresses of the hexagonal screw dislocation boundary, of the straight tilt boundary, and of a zig-zag tilt boundary [221]. For all slip systems, the stresses with which the boundaries resist the passage of glide dislocations are found to be smaller than the stresses required to bow out the glide dislocations on the primary plane against the resistance of their own line tension through the gaps in the boundaries. In spite of its generally larger stresses, the zig-zag wall is somewhat more easily penetrated than the straight tilt wall [221]. The interaction between a wall and a solute atom is found to be less than that between the single nearest dislocation with the same solute atom [220]. A short-range obstacle of single dislocation will easily be surmounted by thermal fluctuation. Also, since the flow stress stays nearly constant, the dramatic changes in the character of the subgrain boundaries that are observed during steady-state deformation suggest that the details of the subgrain boundaries are not an important consideration in the rate controlling process for creep, and as such any description of the rate-controlling process for creep should consider the density of dislocations not associated with subgrain boundaries [177, 222-226].

The second assumption relates to the network coarsening kinetics. equation (33). This assumption must be improved for all link length distribution models in order that they can accurately describe creep deformation. It has been pointed out that this equation in its present form cannot fully describe the network coarsening process [145, 205, 208,
It is shown that equation (33) gives a large value for the incubation time, as compared to experimental results and may be due to its underestimation of the dislocation mobility. It was also found experimentally that [205, 233] that this kinetic equation is inadequate to account for the dislocation network coarsening during creep deformation. The measurements on aluminium (Al) and sodium chloride (NaCl) creep samples [205] suggested that when \( \lambda \to 0, \frac{d\lambda}{dt} \approx \frac{1}{\lambda^n} \), where \( 1 < m < 2 \) (\( m = 1.33 \) for Al and \( m = 1.48 \) for NaCl). Ardell and Lee [233] showed analytically that equation (33) is incompatible with the linear behaviour of the function \( \phi(\lambda, t) \) at small \( \lambda \). A linear behaviour of \( \phi(\lambda, t) \) with \( \lambda \) has been observed by Ardell and co-workers [205, 233] for Al under both the Harper-Dorn and power law creep conditions and for creep of NaCl. Therefore, in view of the inaccuracy of equation (33), a new theory for dynamic network coarsening kinetics in recovery creep is thus needed to match the experimental and theoretical findings. At steady-state, this diffusion-controlled network coarsening kinetics seems to be a solution to the Laplace equation [53, 234, 235], \( \nabla^2 c = 0 \), with a proper set of boundary conditions, where \( \nabla^2 \) is a Laplacian, and \( c \) is the local equilibrium vacancy concentration respectively. Therefore, as an approximation, equation (33) can describe the basic physical nature of the dislocation network coarsening process during creep deformation. This has been demonstrated by many applications of this kinetics to creep of different materials [145, 205, 208, 233]. On the other hand, the kinetics is only sufficient for a first approximation at the present time before an improved equation is obtained. Further work is required to improve the formulation of the network coarsening kinetics for recovery creep.
§2. The Dislocation Link Length Statistics

Like photons and electrons are carriers of light and electrical current, dislocations are carriers of plastic deformation strain in crystalline materials. Deformed crystals contain very large number of dislocations, typically $10^9$ to $10^{12}$ cm/cm$^3$ (in well-annealed monocrystals, the dislocation density is typically $10^6$ cm/cm$^3$). These dislocations are arranged in complex, tangled, curved arrays. Moreover, the dislocation link lengths take typical statistical distributions as has been shown from many direct experimental observations. The modelling of the behaviour of dislocations and the plastic deformation of crystals requires a statistical treatment. The macroscopical plastic deformation behaviour of crystalline solids is an integral of all microscopical actions of individual dislocation links in the network.

The dislocation link length statistics for the plastic deformation of crystals contains a distribution frequency function for the three-dimensional dislocation network structure. The mechanical behaviour of the crystalline materials is described by the dynamic evolution of this distribution function. Both strain-hardening and recovery during plastic deformation are analyzed in terms of the evolution of the dislocation link length distribution function for the three-dimensional dislocation network structure. The results from such dislocation link length distribution models are in agreement with the experimental results of direct dislocation microstructure observation and mechanical measurements, such as tensile, creep, and stress change (increment or decrement) tests. It has been recently pointed out clearly that the distribution of dislocation links plays a fundamental role in the physics of plastic deformation and that a knowledge of the
behaviour of such a distribution function, \( \phi(\lambda) \), can provide significant insight into the nature of elevated temperature plastic deformation [145, 205].

Although several experimental measurements and empirical expressions exist in the literature on the dislocation link length distribution, \( \phi(\lambda) \), see for example [195, 203, 205, 226, 236], it was only recently that an analytical expression for such a dislocation link length distribution frequency function has been obtained mathematically by Wang et al [148], see Appendix A at end of dissertation. It has been shown that the statistics model equation (A1) agrees with experimental results for pure polycrystalline Ni and a polycrystalline 1Cr18Ni9Ti stainless steel [148]. However, the model has never been studied for other experimental conditions and its applicabilities are not generally known. In this section, this model is applied to the study of the dislocation network microstructure, the strain-hardening and recovery behaviour during recovery creep, and the strain rate change during constant strain rate tensile tests. The following sections present all original results obtained by the present author.

2.1. The Relationship between Average Link Length and Dislocation Density

By definition, the dislocation density, \( \rho \), is equal to the product of the average dislocation link length, \( \langle \lambda \rangle \), and the total dislocation links per unit volume, \( N \), namely \( \rho = N \langle \lambda \rangle \). In reference [148], it has been shown that \( \rho = 2\pi^{1/2}N\lambda_m \), where \( \lambda_m \) is the link length at which \( \phi(\lambda) \) attains its maximum value. Thus, a relation is obtained between \( \langle \lambda \rangle \) and \( \lambda_m \), namely:
\[ \langle \lambda \rangle = \frac{2}{\sqrt{\pi}} \lambda_m \]  

(40)

Now, we will consider the relationship between the link length distribution and the volume of the crystal. The three-dimensional network divides the crystal into space filling polyhedra. During plastic deformation, since the change in volume of the crystal is insignificant, the change in the total volume of the polyhedra must likewise be insignificant. Since the volume fraction of polyhedra is unity, constancy of volume demands that:

\[ \int_0^\infty c\lambda^3 \Phi(\lambda) d\lambda = 1 \]  

(41)

where \( c \) is the dislocation network geometry factor relating the volume of a polyhedron to its edge length which is the dislocation link length, \( \lambda \), in the case under consideration. Integration of equation (41) using equation (A1) results in the relation \( 2c\rho \lambda_m^2 = 1 \). Combining this result with equation (40) one finds that the average dislocation link length \( \langle \lambda \rangle \) is related to the total dislocation density \( \rho \) by the following equation:

\[ \langle \lambda \rangle = \left( \frac{2}{\pi c} \right)^{1/2} \rho^{-1/2} \]  

(42)

Imagine, that the plastic deformation is produced by the operation of Frank-Read sources of pinned links longer than the threshold link length, \( \lambda_{th} \), then the applied shear
stress \( \tau \) is related to the average link length \( <\lambda> \) by the Orowan relationship, \( \tau = \mu b <\lambda> \).

Therefore, equation (42) gives a direct proportionality between \( \tau \) and \( \rho^{1/2} \) as is demanded by strain-hardening models in which \( \tau = \alpha \mu b \rho^{1/2} \), where \( \alpha = (\pi c/2)^{1/2} \) is a constant, see equation (16). Equation (42) is also identical to the often-quoted equation obtained by Lin and McLean [194], namely \( <\lambda> = \beta \rho^{1/2} \), where \( \beta \) is a constant. Here for equation (42), \( \beta = (2/\pi c)^{1/2} \). From equation (42), the total number of dislocation links per unit volume is given by \( N = \beta^2 \rho^{3/2} \). For a tight, uniform network of dislocations, \( \beta = 1 \) (\( c = 0.64 \)). For a locally tight, non-uniform dislocation network, \( \beta < 1 \) (\( c > 0.64 \)). Equation (42) clearly shows that the constant \( \beta \) is related to the three-dimensional dislocation network geometry factor, \( c \). This geometrical dependence of the \( \beta \) value has been also pointed out by others when considering either the non-uniformity in dislocation density [195] or dislocation structure and internal stress in creep [196].

2.2. The Mobile Dislocations

To obtain the mobile dislocation density, \( \rho_m \), from the distribution frequency function, \( \phi(\lambda) \) of equation (A1) in Appendix, it is convenient to relate \( \phi(\lambda) \) with the average link length, \( <\lambda> \), and the threshold link size, \( \lambda_{th} = 2\mu b/\sigma \) [145]. see Fig. 23. It is to be expected that in a three-dimensional network of dislocation links, only some of the links (say a fraction of \( f_\rho \)) will lie on crystallographic orientations (crystal planes and directions) which are favourable for glide. These are the potentially mobile links. Also only the links that attain a length longer than \( \lambda_{th} \) through recovery coarsening can glide under an externally applied stress, \( \sigma \). They are the mobile dislocations. Other links which are unfavourably orientated or are shorter than \( \lambda_{th} \) will be immobile (immobile
Fig. 23  Schematic illustration of the relationship between distribution function $\phi(\lambda)$ and the threshold link size $\lambda_{th}$ and the average link length $\langle \lambda \rangle$. In a stress increment test, the threshold link length $\lambda_{th}$ decreases by an amount of $\Delta \lambda$ from $\lambda_{th}$ to $(\lambda_{th} - \Delta \lambda)$, whereas it increases $\Delta \lambda$ from $\lambda_{th}$ to $(\lambda_{th} + \Delta \lambda)$ upon a stress decrement.
dislocations). Thus the mobile dislocation density, \( \rho_m \), can be expressed as follows:

\[
\rho_m = \int_{\lambda_{th}}^{\infty} f_p \lambda \phi(\lambda) \, d\lambda
\]

where \( f_p \) is a crystallographic orientation factor, and is in the range from 0 to 1. Using \( \phi(\lambda) \) as given by equation (A1) in Appendix and integrating equation (43), one obtains the mobile dislocation density, \( \rho_m \), which is given by:

\[
\rho_m = \Bigg\{ f_p \left( 1 + \frac{\lambda_{th}^2}{\lambda_m^2} \right) \exp \left( -\frac{\lambda_{th}^2}{\lambda_m^2} \right) \Bigg\} \rho
\]

Now \( \lambda_m \) can be obtained by combining equations (16), (40) and (42). The result is \( \lambda_m = (2c)^{1/2} \alpha M \mu b / \sigma \). Thus the ratio between \( \lambda_{th} \) and \( \lambda_m \) can be expressed as \( \lambda_{th} / \lambda_m = (8c)^{1/2} / (\alpha M) \), and equation (44) gives the mobile dislocation density, \( \rho_m \), as follows:

\[
\rho_m = \Bigg\{ f_p \left( 1 + \frac{8c}{\alpha^2 M^2} \right) \exp \left( -\frac{8c}{\alpha^2 M^2} \right) \Bigg\} \rho
\]

This result shows that the mobile dislocation density, \( \rho_m \), is directly proportional to the total dislocation density, \( \rho \). This is in agreement with a recent analysis made by Ajaja [180]. An estimate can be made of the proportionality constant in brackets on the right hand side of equation (45), by taking the following values: \( \alpha = 0.45 \), \( M = 3.1 \), \( c = 0.7 \) [145], and \( (f_p)_{\text{max}} = 1.0 \) at elevated temperatures. The resultant proportionality
constant has a maximum value of about 0.218. That is to say that only about 0.218 of
total dislocations (or less) in the three-dimensional dislocation network are mobile
dislocations. The majority of dislocations are immobile in the three-dimensional
dislocation network structure developed during plastic deformation.

Also, the normalized threshold link length, \( u_{th} = \lambda_{th}/\langle \lambda \rangle \), can be obtained from
equations (16) and (40). This normalized threshold link length \( u_{th} \) is given by:

\[
\frac{u_{th}}{\alpha_s M} = \frac{\sqrt{2\pi C}}{\alpha_s M}
\]  

(46)

Equation (46) shows that the normalized threshold link length, \( u_{th} \), is determined by the
gometry factor, \( \alpha_i \), the constant, \( \alpha_i \), and the Taylor factor, \( M \), and is essentially
independent of the applied stress. Using the previously obtained numerical values for \( \alpha_i \),
\( M \), and \( \alpha_i \), and \( M \), equation (46) gives \( u_{th} = 1.5 \), which is in reasonable agreement with Ajaja's
analysis [180], where \( u_{th} \) values are estimated to be in a range from about 1.5 to 2.0.

2.3. Strain-Hardening and Recovery Processes of Recovery Creep Deformation

Now we apply the results from the previous sections to the strain-hardening and
recovery processes for plastic deformation, in this instance during the steady-state creep
of crystalline materials. The strain-hardening coefficient, \( H \), and recovery rate, \( R \),
measured from a stress change (increment or decrement) test are defined by the following
formulas:
\[ H = \left( \frac{\partial \sigma}{\partial \varepsilon} \right)_{\varepsilon} = \lim_{\Delta \varepsilon \to 0} \left( \frac{\Delta \sigma}{\Delta \varepsilon} \right)_{\varepsilon} \] \tag{47}

and

\[ R = -\left( \frac{\partial \sigma}{\partial t} \right)_{\varepsilon} = -\lim_{\Delta \varepsilon \to 0} \left( \frac{\Delta \sigma}{\Delta \varepsilon} \right)_{\varepsilon} \] \tag{48}

First, we calculate \( H \) from the statistical model. In order to obtain \( H \), we first consider \( \Delta \varepsilon \), which is an instantaneous plastic strain generated upon a stress increment from \( \sigma \) to \( (\sigma + \Delta \sigma) \). If the applied stress \( \sigma \) is increased by an amount \( \Delta \sigma \), the threshold link size \( \lambda_{th} \) will decrease by an amount, \( \Delta \lambda = \lambda_{th}(\Delta \sigma/\sigma) \), for a small stress increment \( \Delta \sigma \). Fig. 20. According to equation (32), then the total length of dislocations per unit volume moved instantaneously by this stress increment \( \Delta \sigma \) is given by:

\[ \Delta \rho_m = \mathcal{F}_p \lambda_{th} \phi(\lambda_{th}) \Delta \lambda \] \tag{49}

Assuming that the dislocation links activated (set in motion) by the stress increment glide a distance which is approximately twice the average link length, \( \langle \lambda \rangle \), (as see Evans' strain-hardening model [185]), the instantaneous plastic strain increment \( \Delta \varepsilon \) generated by this stress increase is then given by the Taylor-Orowan equation [172, 173, 237]:

\[ \Delta \varepsilon = \frac{b \sum_{i=1}^{m} A_i}{M V} = \frac{2b \Delta \rho_m \langle \lambda \rangle}{M} \] \tag{50}
where \( V \) is the volume of the deforming crystal, and \( A_i \) is the area swept out by the \( i \)-th gliding dislocation link. Using equations (16), (42), (49) and (A1), and \( \lambda_m / \lambda_c = (8c)^{1/2}/(\alpha_1 M) \), equation (50) can be rearranged into the following form:

\[
\Delta \varepsilon = \left\{ \frac{256 \sqrt{2} F_p c^{3/2}}{\sqrt{\pi} \alpha_1^5 M^6 \mu} \exp \left( -\frac{8c}{\alpha_1^2 M^2} \right) \right\} \Delta \sigma \tag{51}
\]

which gives a strain-hardening coefficient, \( H \), via equation (47) as follows:

\[
H = \left\{ \frac{\sqrt{\pi} \alpha_1^5 M^6}{256 \sqrt{2} F_p c^{3/2}} \exp \left( -\frac{8c}{\alpha_1^2 M^2} \right) \right\} \mu \tag{52}
\]

Therefore, it is evident that the strain-hardening coefficient, \( H \), is directly proportional to the shear modulus, \( \mu \), which essentially agrees with strain-hardening theory [185, 200]. This is also observed for our pure magnesium samples during steady-state creep reported in section 3.4 of Chapter IV, where \( H = 0.27E \) at 200 °C and 20 MPa to 50 MPa, Fig. 14(a). Using the following numerical values for the factors in equation (52): \( \alpha_1 = 0.45 \), \( M = 3.1 \), \( c = 0.7 \), and \( F_p = 1 \) at elevated temperatures, one obtains the proportionality constant for equation (52), i.e. \( H = 2.4\mu \). By using Young's modulus, \( E = 2(1 + v)\mu \) and taking Poisson's ratio \( v = 1/3 \) [237], the strain-hardening coefficient, \( H \), is shown to be related to Young's modulus \( E \) by \( H = 0.9E \). This estimate is in the range from about 0.1E to 1.0E measured during steady-state creep by using stress change (increment) test for many crystalline materials [145, 186].

Secondly, the recovery rate, \( R \), will be considered. During high temperature
deformation (creep), while the strain-hardening process refines the dislocation network, the dislocation network is also undergoing recovery coarsening, in that shorter links shrink and longer links grow. This recovery coarsening process increases the average link length and decreases the total dislocation density and the total free energy of the deforming crystal. In a stress dip test \((-Δσ)\) during steady-state creep, the threshold link length \(\lambda_{th}\) will increase by an amount \(Δλ = λ_{th}(-Δσ/σ)\). Since after the stress decrement, there are no links whose lengths are longer than \(λ_{th}\), creep deformation will cease for a period of time \(Δt\) during which period the network links coarsen to produce longer links. This is called the incubation time. Equation (39) can be used to give a quantitative estimate of this incubation time. By approximating the critical link length \(λ_{cr}\) to the average link length, \(⟨λ⟩[145, 208]\) (where links longer than \(⟨λ⟩\) will grow and those shorter than \(⟨λ⟩\) will shrink), the incubation time is then given by:

\[
Δt = \frac{λ_{th}(-Δσ/σ)}{M′τ(1⟨λ⟩ - 1/λ_{th})}
\]  

(53)

Using equations (16), and (42) for \(⟨λ⟩\) and the relation \(λ_{th} = 2μb/σ\), the equation (53) can be rearranged into following form:

\[
Δt = \left(\frac{M′τ}{σ_2M} \left(\frac{\sqrt{2πe}}{σ} - 1\right)\right)^{-2} \frac{4μ^2b^2}{σ^3} (-Δσ)
\]  

(54)

When \(Δσ\) is sufficiently small, one can obtain the recovery rate, \(R\), from equations (48) and (54), giving:
\[ R = \frac{\mu M^* T}{4b^3} \left( \frac{\sqrt{2\pi C}}{\alpha_i^2 M} - 1 \right) \left( \frac{\sigma}{\mu} \right)^3 \]  

(55)

Applying the Bailey-Orowan equation [75, 157], i.e. equation (22), one obtains a creep rate equation from equations (52) and (55) as follows:

\[ \dot{\varepsilon}_s = \left\{ \frac{128 M^* T f \rho C^{3/2}}{\sqrt{2\pi b^2 \alpha_i^5 M^6}} \left( \frac{\sqrt{2\pi C}}{\alpha_i^2 M} - 1 \right) \exp \left( -\frac{8\alpha_i^2 M^2}{\alpha_i^2 M^2} \right) \right\} \left( \frac{\sigma}{\mu} \right)^3 \]  

(56)

This equation is consistent with the so-called "natural law" [167, 238-240]. However, stress exponents greater than 3 can result from the potential stress dependence of the dislocation network geometry and its link length distribution through the parameters in equation (56). From the foregoing analysis, it is seen that creep rate, \( \dot{\varepsilon}_s \), is essentially determined by the recovery rate, \( R \), since the strain-hardening coefficient, \( H \), is a constant; thus the term "recovery controlled creep deformation". The temperature dependence of creep rate is determined by the dislocation climb mobility, \( M^* \), which is related to the atomic self-diffusion (or vacancy diffusion) during high temperature plastic deformation of crystalline materials.

2.4. Strain Rate Change During Constant Strain Rate Deformation

In this section, an analysis will be made of the strain rate change test performed during a constant strain rate tensile test of crystalline materials. In practice, the strain rate change technique is a very useful method for strain rate sensitivity measurements and microstructural characterization.
During steady-state constant strain rate deformation, if the imposed strain rate, $\dot{\varepsilon}$, is increased instantaneously by an amount $\Delta \dot{\varepsilon}$, the flow stress, $\sigma$, will increase spontaneously by an amount $\Delta \sigma$. This will result in an increase in both $\rho_m$ and $v$ values. According to equation (15), the result of this strain rate increment can be expressed by:

$$\Delta \dot{\varepsilon} = a b \Delta \rho_m v + a b \rho_m \Delta v$$

(57)

Where $\Delta v$ and $\Delta \rho_m$ can be calculated from equations (26) and (49), respectively, and are given as follows:

$$\Delta \rho_m = \rho_m \left\{ \frac{128 f_p c^2}{Za_1^2 M^4} \exp \left( - \frac{8 c}{a_1^2 M^2} \right) \right\} \left( \frac{\Delta \sigma}{\sigma} \right)$$

(58)

and

$$\Delta v = \frac{(n - 2) a_1^2 M^2 b}{\alpha Z} A \frac{D_v b}{k T} \left( \frac{\sigma}{\mu} \right)^{n-2} \left( \frac{\Delta \sigma}{\sigma} \right)$$

(59)

Combining equation (57) with equations (58) and (59), and using equations (15) and (23), one obtains the relationship between the instantaneous increment in the otherwise constant strain rate, $\Delta \dot{\varepsilon}$, and the corresponding increment in the flow stress, $\Delta \sigma$, during a strain rate change:

$$\left( \frac{\Delta \sigma}{\Delta \ln \dot{\varepsilon}} \right)_T = \left\{ \frac{128 f_p c^2}{Za_1^2 M^4} \exp \left( - \frac{8 c}{a_1^2 M^2} \right) + (n - 2) \right\}^{-1} \sigma$$

(60)

where $\Delta \ln \dot{\varepsilon} = \Delta \dot{\varepsilon}/\dot{\varepsilon}$ is applied for a small strain rate increment. This equation clearly
shows that: (i) the increase in the flow stress, $\Delta \sigma$, due to an instantaneous increase in strain rate, $\Delta \dot{\varepsilon}$, during constant strain rate deformation has a linear relationship between $(\Delta \sigma/\Delta \ln \varepsilon)_i$ and $\sigma$ (the Haasen plot [241]); and (ii) the slope is equal to the strain rate sensitivity, $m = (\partial \ln \sigma/\partial \ln \varepsilon)_i$ (in the relation $\sigma = K \dot{\varepsilon}^m$, where $K$ is a material constant) for deforming crystalline materials.

In order to investigate the microstructural dependence and the properties of the strain rate sensitivity, $m$, let:

$$x = \frac{C}{a_1^2}$$

(61)

and

$$y = \frac{128c}{ZM^4} x^2 \exp \left( -\frac{8}{M^2} x \right)$$

(62)

Hence by rearranging equation (60), the strain rate sensitivity, $\tilde{m} = (\partial \ln \sigma/\partial \ln \varepsilon)_i$, can be written as follows:

$$m = \frac{1}{y + (n - 2)}$$

(63)

The behaviour of the function $y$ and the strain rate sensitivity $m$ can now be studied. Taking $c = 0.7$, $f_p = 1$, $Z = 0.218$ [145], $M = 3.1$ and $n = 5$, the function $y$ and the strain rate sensitivity $m$ have been plotted as a function of the variable $x$ in Figs. 24 and 25, respectively. Function $y$ takes a typical distribution behaviour with respect to the
Fig. 24  Variation of the function $y$ with the variable $x$. 
Fig. 25 Variation of the strain rate sensitivity $m$ with the variable $x$. 
variable \( x \), which has a maximum value \( y_{\text{max}} = 4.97 \) at \( x_m = 2.40 \). Fig. 24. The corresponding parameter \( \alpha_{\text{in}} = 0.54 \) at \( x_m \). This is very reasonable in terms of both experimental measurements and theoretical calculations \[178, 179\], where the parameter \( \alpha_4 \) takes values within the range from about 0.1 to 1.0, see section §2 of Chapter IV. The variation of the strain rate sensitivity, \( m \), with the variable \( x \) is in the range from 0.13 to 0.33, Fig. 25. This is reasonable when considering that for \( n = 5 \) used in this analysis, then \( m = 1/n = 0.2 \). Experimental creep measurements have shown that the stress exponent \( n = 5.86 \) for pure magnesium at 200 °C and 20 MPa to 50 MPa, and \( n = 2.8 \) to 16 for the 310 stainless steel at 700 °C and 80 MPa to 320 MPa, during steady-state creep deformation. According to this analysis, for pure magnesium \( m = 0.17 \), and for the 310 stainless steel \( m = 0.06 \) to 0.36. These results further demonstrate that the model based on the dislocation link length statistics is in good agreement with experimental measurements and other theoretical calculations. The model gives reasonable estimations of the strain rate sensitivity, \( m \), and physically meaningful parameter, \( \alpha_4 \), for the strain rate change test during high temperature constant strain rate deformation of crystalline materials. From this analysis, it is also seen that the strain rate sensitivity, \( m \), changes with dislocation distribution. However, the \( m \) values are confined to the range 0.13 to 0.33. It is therefore evident that the larger \( m \) values (\( \geq 0.5 \)) can not result solely from the variation of dislocation distribution. Grain boundary mechanisms must be operative in order that a larger \( m \) value is obtained and superplastic deformation is possible. The movement of intragranular dislocations (power law creep) saves as an accommodation process to the grain boundary mechanisms and has an important influence on the flow
mechanism. This connection between creep and the superplastic deformation of polycrystalline materials will be discussed in the next section §3 of this chapter.

From the above analysis, it has been shown that the increase in the flow stress, \( \Delta \sigma \), upon an instantaneous strain rate increase, \( \Delta \dot{\varepsilon} \), follows the Haasen relationship [241], in that the \( (\Delta \sigma/\Delta \ln \dot{\varepsilon})_r \) versus \( \sigma \) plot is linear, and the slope is equal to the strain rate sensitivity, \( m \), in accordance with Cottrell-Stokes law [242, 243]. The material parameters in the model are also examined in the light of this new analysis and experimental data. The strain rate sensitivity, \( m \), values predicted by the model are in good agreement with deformation behaviour of crystalline materials, and the parameter, \( \alpha_r \), agrees well with experimental measurements.

In obtaining equation (58), the ratio between the mobile dislocation density and the total dislocation density \( Z = \rho_m/\rho \) is considered to be a constant. Otherwise its dependencies on \( \sigma \) or \( \varepsilon \) would have to be taken into account in the derivatives and this would lead to more complex results. However, if one takes the derivative of equation (16) for a constant \( Z \) value, then the following relationship is obtained: \( \Delta \ln \sigma = (1/2) \Delta \ln \rho = (1/2) \Delta \ln \rho_m \) or \( \Delta \rho_m = 2 \rho_m (\Delta \sigma/\sigma) \). Therefore the bracketed section in equation (58) will have the value of 2. If one takes the following values for the parameters \( c = 0.7, M = 3.1, \ Z = 0.218, \alpha_i = 0.45 \) and \( f_p = 1 \) for elevated temperature plastic deformation of crystalline materials [145, 178, 179], then the bracketed section in equation (52) will have a value of about 4. Given that a degree of uncertainty exists in the values of the parameters \( c, Z, \alpha_i \), this factor 2 can be regarded as a reasonable estimation. Furthermore, if taking the value 2 given by the bracketed section in equation (58), then one has the following
which clearly shows that the parameters $c$, $Z$ and $\alpha_i$ are not independent of each other but rather they are interrelated as given in equation (64). The above three parameters are determined by the geometry of dislocation network structure and its link length distribution during plastic deformation. By taking $c = 0.7$, $f_p = 1$, $M = 3.1$ and $Z = 0.218$, the parameter $\alpha_i = 0.36$ or 0.94. These $\alpha_i$ values are in reasonably good agreement with numerous experimental measurements and theoretical calculations for various crystalline materials which show that the parameter $\alpha_i$ takes values within a range from about 0.1 to 1.0 [178, 179]. It should also pointed out that by applying equation (23), equation (60) should be applicable to the steady-state plastic deformation of crystalline materials at elevated temperatures.

Also, by taking derivatives on both sides of equation (23), one obtains $\Delta \ln \dot{\epsilon} = n \Delta \ln \sigma$. Then by comparison with equation (60), the bracketed section in equation (60) is equal to $n$. That is:

$$m = 1/n$$

(65)

The above equation can also be obtained by substituting equation (64) into equation (60). Equation (65) is a general result which is valid for both constant strain rate deformation and constant stress creep experiments. This shows that equation (60) can be applied to
plastic deformation of crystalline materials, where a strain rate change is applied to the deforming specimen undergoing constant strain rate plastic deformation. However, it should be pointed out that equation (60) is valid under the condition that the dislocation microstructure of the plastic deforming specimen is in the form of a three-dimensional network. This is related to the assumption made in its derivation that the dislocation link length of the deforming material takes a statistical distribution given by equation (A1). This is a reasonable assumption for polycrystalline materials [145], especially for their plastic deformation at elevated temperatures (such as recovery creep and hot working).

2.5. Brief Summary

In summary, the results coming from a model based on the dislocation link length statistics [148] are in fairly good agreement as compared to both experimental measurements and other independent theoretical results. In particular, analyses of the three-dimensional dislocation network structure, the strain-hardening and recovery processes during steady-state creep deformations, and the strain rate change during constant strain rate tensile test, have shown the following main results: (i) The average dislocation link length of a three-dimensional dislocation network is related to the total dislocation density by the equation $<\lambda> = (2/\pi c)^{1/2} \rho^{1/2}$, where $c$ is the dislocation network geometry factor; (ii) The strain-hardening coefficient $H$ is proportional to, and is the order of, Young's modulus of the materials for a stress change test during high temperature deformation. The creep rate is primarily determined by the recovery rate $R$ through the mobility of diffusion controlled dislocation climb; (iii) A creep strain rate equation is also obtained which is consistent with the "natural law" creep equation; and (iv) The increase
in the flow stress, $\Delta \sigma$, associated with an instantaneous strain rate increase, $\Delta \dot{\varepsilon}$, during constant strain rate deformation at a given temperature follows the Haasen plot in that $(\Delta \sigma/\Delta \ln \dot{\varepsilon})_T$ versus $\sigma$ is linear with a slope equal to the strain rate sensitivity, $m = (\partial \ln \sigma / \partial \ln \dot{\varepsilon})_T$. 
§3. Superplasticity

In this section, the grain size effect in high temperature creep and superplastic deformation of polycrystalline materials is discussed and modelled using constitutive creep equations. A model is presented for high temperature creep of polycrystalline materials. Two deformation modes are considered in the model, namely diffusional flow and power-law creep. The grain size distribution is also taken into account to describe the overall steady-state creep deformation properties. A critical grain size, \( D_c \), is defined in the grain size distribution, which is determined by such factors as applied stress, test temperature, and the material properties (diffusion coefficient, shear modulus, melting temperature, etc.). At a certain temperature and applied stress, grains with a size smaller than \( D_c \) will deform by diffusional flow, and grains with a size larger than \( D_c \) will deform by power-law creep. This model predicts that within certain steady-state creep rate ranges, a polycrystalline material will undergo superplastic deformation.

The grain size plays an important role during the plastic deformation of polycrystalline materials. When the test temperature is higher than about one half of the melting point of the material, the grain boundary is an active diffusion path [23-25]. It then follows naturally that a material with a smaller grain size will creep at a higher rate than one with a larger grain size. That is to say that materials with smaller grains are less creep resistant at elevated temperatures, although grain boundaries are a source of strength for low temperature deformation. It is usual to use an average grain size (diameter) to characterize the creep properties. However, a polycrystalline material rarely has a uniform grain size, but rather has a spectrum of grain sizes. Thus, this grain size distribution must
be taken into account to better characterize the high temperature deformation.

In our model, the grain size distribution is represented by a frequency function, \( f(D) \), where \( D \) is the grain diameter. The frequency function, \( f(D) \), is defined such that the number of grains with a diameter between \( D \) and \( (D + dD) \) is \( f(D)dD \). To illustrate the model Colbeck’s [244] normal distribution of the logarithm was applied in the calculation of grain volume fraction. Two deformation modes will be considered, namely diffusional flow for grains with diameter \( D < D_c \), and power law creep for grains with diameter \( D > D_c \). By means of this model, some unique deformation characteristics of polycrystalline materials can be understood, in particular superplasticity. Some typical results for pure polycrystalline aluminium, magnesium, cadmium, and stoichiometric polycrystalline \( \text{UO}_2 \) are presented to illustrate the model.

In this model the distribution functions for grain sizes are the Colbeck’s [244] normal distribution of the logarithm. In the numerical calculations Colbeck’s [244] normal distribution of the logarithm was applied. The distribution says the probability of finding grains with size between \( \tilde{D} \) and \( (\tilde{D} + d\tilde{D}) \) is \( f(\tilde{D})d\tilde{D} \), where \( f(\tilde{D}) \) is the frequency function that in logarithmic scale has the form:

\[
f(\ln\tilde{D}) = \frac{1}{\sqrt{2\pi} \ln\sigma_g} \exp\left\{-\frac{1}{2} \left( \frac{\ln\tilde{D}/\tilde{D}_g}{\ln\sigma_g} \right)^2 \right\}
\]

(66)

The parameters in this equation are \( \tilde{D} \) = the normalized grain size (\( \tilde{D} = D/\bar{D} \)), where \( \bar{D} \) is the mean diameter; \( \sigma_g \) = the geometric standard deviation (\( = 1.460 \)); \( \tilde{D}_g \) = the normalized geometric mean (\( = 0.931 \)).
Using this distribution frequency function, the following four calculations were made, namely:

1) The number frequency function itself, \( f(\bar{D}) \):

2) The number fraction distribution \( F(\bar{D}) \) or the probability of finding grains with size from 0 to \( \bar{D} \):

\[
F = \int_0^{\bar{D}} f(D) dD = \int_{-\infty}^{\ln \bar{D}} f(lnD) dlnD \tag{67}
\]

3) The volume frequency function \( v(\bar{D}) = \bar{D}^3 f(\bar{D}) \). In this calculation the logarithmic scale was used:

\[
v(ln\bar{D}) = [\exp(ln\bar{D})]^3 f(ln\bar{D}) \tag{68}
\]

4) The volume fraction distribution \( V \) of grains with size from 0 to \( \bar{D} \):

\[
V = \int_0^{\bar{D}} v(D) dD = \int_{-\infty}^{\ln \bar{D}} v(lnD) d(lnD) \tag{69}
\]

All above four distributions were calculated in both normal scale \( \bar{D} \) and logarithmic scale \( \ln\bar{D} \).

3.1. The Model

A single phase polycrystalline material usually has a typical statistical grain size distribution, such as schematically illustrated in Fig. 26. At an elevated temperature and
Fig. 26  Schematic diagram showing the distribution frequency function, \( f(D) \), and the critical grain diameter, \( D_c \).
a given applied stress, the smaller grains \((D < D_c)\) will deform at a faster rate by diffusional (linear) flow due to grain boundary and lattice diffusion, while the other portion of larger grains \((D > D_c)\) will deform at a slower rate by power law creep due to consecutive movement of dislocation glide and climb. This critical grain size, \(D_c\), is determined by the applied stress for a material deforming at a given temperature. When the test temperature is held constant, the smaller the value of the applied stress, the larger is the \(D_c\) value (more grains and/or volume fraction will deform by diffusional flow at a small applied stress level). This single phase polycrystalline material can be pictured to be a composite with two "phases", namely "phase 1" of smaller grains and "phase 2" of larger grains in terms of the deformation modes. These two "phases" tend to deform at different strain rates for a constant applied stress and test temperature. On the other hand, compatibility demands that all grains within a deforming material deform at a uniform strain rate. The requirement of compatibility and the heterogeneity in strain rate due to grain size distribution will then result in an internal stress among grains with different sizes. The larger grains will support a larger stress (larger than the applied stress), in order to deform at a faster strain rate to match the deformation rate in smaller grains, and the smaller grains will carry a smaller stress (less than the applied stress) to reduce their deformation rate. The volume average of this distributed internal stress is equal to the applied stress. This stress redistribution among grains can be described by a stress enhancement factor, \(\chi\), which is equal to the ratio between the stress in coarse grains and the applied stress and is in the range 1 to 3 [245], Fig. 27. This stress enhancement factor is determined by the grain size distribution and the creep test conditions (applied stress
Fig. 27 The stress enhancement factor, $\chi$, versus the volume fraction of the fine grains $(D \leq D_o)$, $V_f$ (solid curve). The upper bound values $\chi_{\text{max}} = 1/(1 - V_f)$, and the lower bound values $\chi_{\text{min}} = 1$, are given by dot-dashed curve and dashed line, respectively.
and temperature) as well as the material properties. When the material deforms by only one of the two deformation modes (power law creep for very high stress or diffusional flow for very low stress), the stress enhancement factor, $\chi$, is equal to 1, while $\chi$ is larger than 1 when the two deformation modes are operating simultaneously.

3.2. Application of the Model

(A) Deformation of Pure Polycrystalline Aluminium

Creep data were calculated for pure polycrystalline aluminium with an average grain size of 15 $\mu$m and a maximum grain size of 150 $\mu$m, at a temperature of 573 K. The rate equation used is as follows:

$$\dot{\epsilon}_s = K_p \bar{\sigma}^n + \bar{\sigma}/\eta$$

(70)

Where $\dot{\epsilon}_s$ is the overall steady-state creep rate, $K_p$ is the power law creep constant, $\bar{\sigma}$ is the shear modulus $\mu$ normalized applied stress $\sigma$ ($\bar{\sigma} = \sigma/\mu$), $n$ is the stress exponent, and $\eta$ is the viscosity [16, 132, 135]. The power law creep constant $K_p$ and the viscosity $\eta$ in equation (70) are given by:

$$K_p = A \frac{D_0 \mu b}{kT}$$

(70A)

and

$$\eta = \frac{kTd^3}{14\mu\Omega D8 (\pi + D_c d/D8)}$$

(70B)

respectively. Where $A$ is a dimensionless material constant for power law creep; $d$ is the
Table 1. Material parameters for creep of pure polycrystalline aluminium [191].

<table>
<thead>
<tr>
<th>Ω, cm³</th>
<th>b, cm</th>
<th>μ, N/m²</th>
<th>(1/μ)dμ/dT, K⁻¹</th>
<th>(D,λ), cm²/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.66 \cdot 10^{21}</td>
<td>2.86 \cdot 10⁻⁶</td>
<td>2.54 \cdot 10^{10}</td>
<td>5.4 \cdot 10⁻⁴</td>
<td>0.035</td>
</tr>
<tr>
<td>Qₜ, kJ/mole</td>
<td>(D₀)ₜ, cm²/s</td>
<td>Qₜ, kJ/mole</td>
<td>A</td>
<td>n</td>
</tr>
<tr>
<td>120.44</td>
<td>0.1</td>
<td>60.22</td>
<td>3.40 \cdot 10⁶</td>
<td>4.4</td>
</tr>
</tbody>
</table>
Fig. 28 Creep data calculated for pure polycrystalline aluminium with an average grain size of 15 µm and a maximum grain size of 150 µm, at a temperature of 573 K.

(a) The shear modulus normalized applied stress, \( \sigma \), versus the steady-state strain rate, \( \dot{\varepsilon}_r \). The numbers in the figure refer to the individual grain size (in µm), in which the power law creep and the diffusional flow have the same strain rate at each of the indicated applied stresses:
Fig. 28  Creep data calculated for pure polycrystalline aluminium with an average grain size of 15 \( \mu \text{m} \) and a maximum grain size of 150 \( \mu \text{m} \), at a temperature of 573 K.

(b) The strain rate sensitivity, \( m \), versus the steady-state strain rate, \( \varepsilon' \), S-curve.
grain size: $D_\gamma$ is the lattice diffusion coefficient; $\Omega$ is the atomic volume; $D\delta$ is the diffusion parameter pertinent to either the grain boundary, $D_\gamma\delta_\gamma$, or boundary phase, $D_\mu\delta_\mu$, if there is a grain boundary phase present, for instance an amorphous phase in some ceramic materials; and $kT$ has its usual meaning. The material parameters (diffusion coefficient, shear modulus, melting point) used in the calculation were taken from reference [191], see Table 1. The results are presented in Fig. 28. The numbers in Fig. 28(a) refer to the individual grain sizes ($\mu m$), in which the power law creep and the diffusional flow have the same rate at each of the indicated applied stresses. This result indicates that for aluminium ($\bar{D} = 15 \mu m$), the strain rate sensitivity, $m = \frac{\partial \log \varepsilon}{\partial \log \sigma}$, has a larger value within the strain rate range $10^{-6}$ to $10^{-3} \text{ s}^{-1}$. Fig. 28(b). This large value for the strain rate sensitivity, $m$, is generally associated with superplastic deformation in both alloys and ceramic materials [246-251]. If the average grain size increases to 30 $\mu m$ and the maximum grain size increases to 300 $\mu m$, then the occurrence of the larger $m$ values shifts to a smaller strain rate range, namely from $10^{-9}$ to $10^{-6} \text{ s}^{-1}$, which is out of the range generally seen for superplastic deformation. This is consistent with the general superplasticity requirement of a grain size of less than about 10 $\mu m$ for fine structure superplasticity (FSS) [250, 251].

(B) Transition Between Power Law Creep and Diffusional Flow

During elevated temperature deformation, there is a deformation mechanism transition when the applied stress is reduced at a constant temperature. This creep mechanism transition is related to the grain size distribution in the material. The transition is one where there is a change from $n = 1$ (diffusional flow) to $n = 5$ (power law creep)
as the applied stress is increased for a particular grain size. For diffusional flow of "phase 1" (\(n = 1\)), the creep rate increases as the grain size is decreased. On the other hand, the mechanism controlling creep in the power law creep of "phase 2" (\(n = 5\)) is almost independent of changes in grain size. This will cause a creep mechanism transition which is related to the grain size distribution, when the applied stress changes from a small value to a large value at a given temperature. When the two types of deformation modes are in equilibrium, the strain rates due to the two deformation modes are equal. Then the transition stress, \(\sigma_{\text{eq}}\), as well as corresponding internal stress, \(\sigma_i\), can be obtained. The transition stress, \(\sigma_{\text{eq}}\), is related to the grain size in a Hall-Petch-type relationship for those materials where recovery is the rate-controlling process in elevated temperature creep. The results for three materials, namely stoichiometric polycrystalline UO\(_2\), pure polycrystalline magnesium and cadmium, are given in Table 2. The internal stress calculated for these three materials are in good agreement with experimental measurements [252-256]. Also, from Table 2 it is seen that the internal stress is in the range of \(10^{-4}\) \(\mu\) to \(10^{-3}\) \(\mu\), where \(\mu\) is shear modulus of the material at the testing temperature. The magnitude of this internal stress (corresponding to the transition stress) is equal to the Peierls force, \(\sigma_p = -10^{-4}\) \(\mu\) to \(-10^{-2}\) \(\mu\) for dislocation glide, the higher values being associated with covalent crystals and the lower values with closed-packed metals [53]. This Peierls force is the maximum value of a periodic lattice resistance stress to dislocation glide associated with the Peierls energy (a variable lattice displacement potential). The relatively smaller internal stress as compared to the Peierls force may be due to the fact that \(\sigma_p\) should decrease with increasing temperature [53]. Some recent
experimental results have shown that both the dislocation density in Harper-Dorn creep and the transition between power law creep and Harper-Dorn creep are also controlled by the Peierls stress in a number of polycrystalline materials, such as ferrite, beryllia, alumina, calcite and olivine [257, 258]. Dislocation glide is athermal in nature during recovery creep deformation, which is permitted by the dislocation network growth for links to gain enough length to glide athermally under the action of the applied stress [145, 208]. That is to say, when the applied stress is smaller than the transition stress, dislocations can only climb and can not glide athermally, while thermal glide motion of dislocation links may operate at elevated temperatures. This corresponds to the diffusional flow of "phase 1" (n = 1). On the other hand, when the applied stress is larger than the transition stress, dislocations can climb as well as glide, which corresponds to the power law creep of "phase 2" (n = 5).
Table 2. The transition stress, $\sigma_{\text{equiv}}$, the internal stress, $\sigma_i$, for UO$_2$, Mg and Cd.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature (°C)</th>
<th>Grain Size (µm)</th>
<th>$\sigma_{\text{equiv}}$ ($10^3$)</th>
<th>$\sigma_i$ ($10^3$)</th>
<th>($\sigma_i/\sigma_{\text{equiv}}$)$_{\text{calculated}}$</th>
<th>($\sigma_i/\sigma_{\text{equiv}}$)$_{\text{measured}}$ ([245, 246])</th>
</tr>
</thead>
<tbody>
<tr>
<td>UO$_2$</td>
<td>1650</td>
<td>18</td>
<td>4.163</td>
<td>3.209</td>
<td>0.771</td>
<td>---</td>
</tr>
<tr>
<td></td>
<td>1666</td>
<td>13</td>
<td>4.956</td>
<td>3.833</td>
<td>0.773</td>
<td>---</td>
</tr>
<tr>
<td></td>
<td>1666</td>
<td>10</td>
<td>5.452</td>
<td>4.172</td>
<td>0.765</td>
<td>---</td>
</tr>
<tr>
<td>Mg</td>
<td>25</td>
<td>90</td>
<td>7.101</td>
<td>7.083</td>
<td>0.997</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>90</td>
<td>6.079</td>
<td>6.063</td>
<td>0.997</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>90</td>
<td>0.995</td>
<td>0.977</td>
<td>0.982</td>
<td>0.80</td>
</tr>
<tr>
<td>Cd</td>
<td>24</td>
<td>220</td>
<td>7.173</td>
<td>6.840</td>
<td>0.954</td>
<td>0.93-0.98</td>
</tr>
</tbody>
</table>
3.3. Discussion and Summary

For a polycrystalline material with a typical statistical grain size distribution, the critical grain size, $D_e$, is very important. This critical grain size $D_e$ is determined by the applied stress value for a material deforming at a given temperature. A smaller applied stress corresponds to a larger critical grain size $D_e$, and more grains and/or grain volume fraction is "phase 1" ($n = 1$) and will deform by diffusional flow. That is to say, the critical grain size, $D_e$, divides the specimen into two components, one includes smaller grains ($0 < D < D_e$) of "phase 1" and the other includes the rest of larger grains ($D > D_e$) of "phase 2". The smaller grains deform by diffusional flow - "phase 1" with $n = 1$, and the larger grains deform by power law creep - "phase 2" with $n = 5$. At a higher applied stress level, "phase 2" composes most of the deforming polycrystalline material and dominates the overall deformation behaviour. On the other hand, at a small applied stress level, "phase 1" has a larger volume fraction and will control the overall deformation behaviour. Within an intermediate applied stress range, the two deformation modes operate simultaneously. It is in this transition range that the deformation behaviour of a polycrystalline material will continuously transform from power law creep - "phase 2" with $n = 5$ to diffusional flow - "phase 1" with $n = 1$ as the applied stress is decreased. When this grain size distribution is taken into account, the elevated temperature deformation of polycrystalline materials can be better understood. A single phase polycrystalline material can be viewed as a two phase flow mixture. The high value of strain rate sensitivity, m, for potential superplastic deformation can also be reached within an applied stress and/or strain rate range, which is consistent with numerous experimental
observations made for both alloys and ceramic materials [246-251]. The transition between the two deformation models is linked to the grain size distribution and to the creep testing conditions. For a particular grain size in the distribution, the internal stress corresponding to the transition stress is of the order of the Periels stress for dislocation athermal glide at a given temperature. When the transition stress is smaller than the Periels stress, only diffusional flow takes place within grains and along grain boundary regions, and the creep rate is linearly proportional to the applied stress.

The model is also in agreement with a recent analysis on the role of intragranular dislocation strain (1S) in the superplastic Pb-62% Sn eutectic alloy and the proposed model of dislocation mechanism for superplastic deformation [248]. The intragranular dislocation strain is nonuniform in nature in both the Sn and Pb phases, and tends to occur in an oscillatory manner with changes from positive to negative contributions to the total strain as deformation continues. The net contribution to the total strain is close to zero so that the grains remain essentially equi-axed even at high elongations. On one hand, intragranular dislocation interactions are not important with respect to the total strain since only about three dislocations move through every grain each second. On the other hand, the absorption of lattice dislocations in the boundaries serves as a source for mobile extrinsic grain boundary dislocations. Therefore, the accommodation process has an important influence on superplastic deformation as a whole. In this dislocation model for superplastic deformation [248], the two deformation mechanisms are operating at the same time, namely intragranular dislocation movement and the grain boundary sliding and grain re-arrangement. In non-superplastic materials under conditions where grain boundary
sliding and cavitation are unimportant, the strain of each individual grain correlates, on average, with the macroscopic elongation of the specimen as a whole. In the superplastic Pb-62\%Sn alloy, however, the specimen elongation is very high and yet the movement of intragranular dislocations makes no significant contribution to the macroscopic strain [248]. Although intragranular dislocation movement does not contribute directly to the total strain, it does accommodate to the grain re-arrangement (the relative translation of individual grains) and grain boundary sliding (GBS) by providing mobile extrinsic grain boundary dislocations. Thus, there is a physical interconnection between the dominant dislocation processes occurring at the grain boundaries and the less important (few in number and little in strain) dislocation movement with the grains.

A "phase" approach incorporating a grain size distribution effect is described for high temperature deformation. In this model, a creep mechanism transition can lead to stress redistribution and a stress enhancement in the coarse grains of a polycrystalline material with a continuous grain size distribution within a certain range of applied stress and/or strain rate. On the other hand, heterogeneous deformation gives rise to stress relaxation in the coarse grains. The resultant effect of these two opposing effects is to give a stress enhancement factor, $\chi$, which is less than its upper limit, $1/(1 - V_p)$, but larger than one. Superplastic deformation is then made possible by this stress enhancement factor $\chi$ acting in coarse grains of a deforming crystal. For elevated temperature creep, a grain size factor is suggested for the constitutive simulation, which could allow a better description of elevated temperature deformation of polycrystalline materials.
CHAPTER VI  CONCLUSIONS

Experimental creep and stress relaxation measurements on pure magnesium and the 310 stainless steel and calculations based on dislocation network (dislocation link length distribution) models and the dislocation link length statistics have given the following results:

(1) The creep curves are all normal type (N-type) transient under the present experimental test conditions:

(a) The creep strain rate ratio, $F$, between the initial and steady-state strain rate decreases from a large value to about unity when the test temperature or the applied stress is increased.

(b) Upon loading, the apparent activation energy for creep, $Q_c$, is lower and is closer to the activation energy for pipe diffusion, $Q_p$, than the value at steady-state which is of the order of the activation energy for volume diffusion, $Q_v$.

(2) The results on the applied stress dependencies of the creep rates for both of the materials are as follows:

(a) The initial creep rate is less strongly dependent upon the applied stress than the steady-state creep rate. Expressed in terms of the power law equation, $\dot{\varepsilon} = \dot{\varepsilon}_0 \sigma^n$, it was found that $n_i = 2.8$ and $n_s = 5.9$ for pure magnesium, and $n_i = 2.1$ and $n_s = 4.6$ for the 310 stainless steel.

(b) The difference between the initial and steady-state creep rates on the stress dependence is due to an internal stress increase in the course of normal transient creep of the two materials. The stress dependence of the initial creep rate offers a useful indirect
measurement on the stress dependence of the average effective dislocation velocity during elevated temperature recovery creep deformation.

(3) The main conclusions to come from the experimental study on pure magnesium using a combined creep and stress relaxation test method can be summarized as follows:

(a) Dynamic recovery rate. R. values for the corresponding steady-state creep showed a similar stress dependence to that of steady-state creep rate, \( \dot{\varepsilon}_c \). The stress exponent obtained from logR versus log\( \sigma \) was \( n_c = 5.8 \). The activation energy for dynamic recovery was \( Q_r = 88.5 \text{ kJ/mole} \). The strain-hardening coefficient, H, calculated from the creep and stress relaxation results showed that: (i) H is a constant (\( H = 0.27E \)) and is independent of the applied stress at 200 °C and 20 MPa to 50 MPa; and (ii) H decreases from 0.50E to 0.19E with an increase in test temperature from 150 °C to 250 °C at 35 MPa.

(b) The data generated from the present experiments for pure magnesium can be explained in terms of dislocation network models for recovery creep of pure metals and Class II alloys. The experimental procedure used in this study provides a practical method to investigate the high temperature plastic deformation behaviour of crystalline materials in which a recovery process is the rate determining process. The method consists of a creep test to steady-state stage and followed by a stress relaxation test at the same test temperature, which can be carried out using a normal tensile or creep test machine.

(4) Simulation of the creep of the 310 stainless steel using the Öström-Lagneborg creep model for recovery creep has shown that:
(a) The calculated results of creep strain show a good fit to the experimental results. Calculations of the network distribution function $\phi(\lambda, t)$ and the dislocation density $\rho(t)$ are in fair agreement with results obtained by previous independent investigators. The mobile dislocation density constitutes about 20% of the total dislocation density under the range of creep testing conditions used in the present study.

(b) Based on a detailed examination of the elastic theories of subgrain boundaries and recent experimental findings, it is suggested that the framework of the Öström-Lagneborg creep theory is able to model the creep behaviour of materials where a subgrain structure is formed during creep.

(c) The deficiency of the Öström-Lagneborg creep model is that the recovery coarsening kinetics of the network structure is only an approximation. The creep model underestimates the mobility of climbing dislocations during elevated temperature recovery creep deformation of crystalline materials in terms of the model predictions and experimental measurements on the incubation time for the stress change test carried out in steady-state creep.

(5) Application of a dislocation link length statistical model to creep and constant strain rate tensile tests have produced the following results:

(a) The average dislocation link length of a three-dimensional dislocation network is related to the total dislocation density by the equation $<\lambda> = (2/\pi c)^{1/2} \rho^{1/2}$, where $c$ is the dislocation network geometry factor. This result is consistent with experimental measurements of dislocation density and dislocation link lengths.

(b) The strain-hardening coefficient, $H$, is proportional to, and is the order of,
Young's modulus of the materials for a stress change test during elevated temperature recovery creep. The recovery creep strain rate is shown to be primarily determined by the recovery rate through the mobility of dislocation links. A creep rate equation is also obtained which is consistent with the "natural law" creep equation.

(c) The increase in the flow stress, $\Delta \sigma$, associated with an instantaneous strain rate increase, $\Delta \dot{\varepsilon}$, during constant strain rate deformation of crystalline materials at a given temperature follows the Haasen plot in that $(\Delta \sigma/\Delta \ln \dot{\varepsilon})_T$ versus $\sigma$ is linear, with a slope equal to the strain rate sensitivity, $m = (\partial \ln \sigma/\partial \ln \dot{\varepsilon})_T$. A model based on the dislocation link statistics predicts reasonable strain-rate sensitivity, $m$, and the parameter, $\alpha$, values which agree well with experimental results for many crystalline materials.

(6) A model is proposed for structure superplastic deformation of a polycrystalline material with a continuous grain size distribution, in which two deformation modes are considered, namely power law creep and linear flow. The single phase deforming polycrystalline material is visualized as a two "phase" mixture. The results are as follows:

(a) In this model, a creep mechanism transition can lead to stress redistribution and a stress enhancement in the coarse grains. On the other hand, heterogeneous deformation gives rise to stress relaxation in the coarse grains. The resultant effect is to give a stress enhancement factor, $\chi$, which is less than its upper limits, $1/(1 - V_f)$, but larger than 1. Superplastic deformation is made possible by this stress enhancement factor, $\chi$, acting in coarse grains of a deforming polycrystal.

(b) Application of the model to pure aluminium show that the model results are consistent with superplastic requirement of fine grains less than about 10 $\mu$m. The strain
rate range predicted by the model is well within the range reported for many polycrystalline metallic and ceramic materials.

(c) The internal and transition stresses calculated from this model for three materials $\text{UO}_2$, Mg, and Cd are similar to those experimental measurements. The creep measurement on the transition stress from power law creep to linear flow may provide information on the Peierls stress for dislocation glide.
RECOMMENDATIONS FOR FURTHER WORK

Two specific topics are suggested for further work. These are:

(1) In-situ transmission electron microscopy observations on selected specimens: A device is required to simulate creep conditions within the TEM chamber, where a constant load (or strain rate) and a constant temperature can be maintained during the observation. A high voltage electron microscopy (e.g. 1 MV) would be required. The observation will need to concentrate on the detailed movement of an individual dislocation link during creep. Special attention should be paid to the characteristic movements of dislocation nodes and jogs, the configuration of an entire dislocation link during its motion, and the interactions between moving dislocation links. These observations would provide a dynamic picture for dislocation network models of elevated temperature recovery creep deformation of crystalline materials.

(2) Theoretical studies aimed at improving the modelling the recovery dislocation network coarsening kinetics for elevated temperature creep: This is an important mechanism issue since all dislocation network models make use of such a network coarsening kinetics. This work would be coupled with both the In-situ TEM observations on the characteristics of dislocation motion and calculations of forces, energies, and solid state diffusion within the dislocation network structure where an external stress is applied to a deforming crystalline material.
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APPENDIX A

THE DISLOCATION LINK LENGTH STATISTICS

The dislocation link length statistical treatment [148] is built upon mathematical principles. The model equation is derived from a three-dimensional statistical analysis and the method of Lagrange multipliers. In obtaining the model equation, one physical assumption was made that the distribution of actual links in all orientations of crystalline materials have equal probability of link spatial distribution. That is to say, there is a uniform distribution with respect to crystallographic orientations: different slip systems uniformly orient in a three-dimensional space. It is a reasonable assumption for polycrystalline materials deforming at elevated temperatures, such as plastic deformation of FCC polycrystalline materials under recovery creep or hot working conditions [145].

In a polycrystalline specimen, the total number of slip systems (slip planes and slip directions) is very large, i.e. equal to the number of grains times the number of slip systems in each of the individual grains (one single crystal for a grain). Face-centred cubic (FCC) crystals have the most physically distinct slip systems, in total twelve (\{111\}<110> type) than for body-centred cubic (BCC) crystals, the hexagonal close-packed (HCP) structures, and other crystal structures. At elevated temperatures, typically above 0.5T_m (T_m is the melting temperature of the crystal measured in degrees Kelvin), all possible slip systems including those on crystal planes of higher indices are operative, and dislocations acquire a new degree of freedom: they can climb as well as glide. It is likely therefore that dislocation links will distribute in all possible positions (crystal orientation) within the test specimen during plastic deformation of polycrystalline
materials at elevated temperatures.

This analytical dislocation link length distribution frequency function is given by:

\[ \phi(\lambda) = 2\rho \left( \frac{\lambda^2}{\lambda_m^2} \right) \exp \left( - \frac{\lambda^2}{\lambda_m^2} \right) \]  \hspace{1cm} (A1)

where \( \lambda_m \) is the dislocation link length at which \( \phi(\lambda) \) attains its maximum value.

It can be shown that equation (A1) can be transformed into the following formula to express the relative numbers of dislocation links \( \Delta N/N \) having lengths confined to the interval \( \Delta \lambda \) between \( (\lambda - \Delta \lambda/2) \) and \( (\lambda + \Delta \lambda/2) \):

\[ \frac{\Delta N}{N} = \left\{ \left( \frac{4h^2}{\sqrt{\pi}} \right) \lambda^2 \exp \left( - h^2\lambda^2 \right) \right\} \Delta \lambda \]  \hspace{1cm} (A2)

where \( h = 1/\lambda_m \). This equation shows that equation (A1) for the dislocation link length distribution in solid crystals has the same functional form as Maxwell distribution law expressing the relative numbers of molecules in a gas which have various given speeds or kinetics energies of thermal agitation at any instant. This shows that the dislocation link length distribution in a plastically deforming crystalline solid has the same statistical behaviour as the distribution of the molecules velocity or kinetics energy in an ideal gas.
APPENDIX B

OPERATING PROCEDURES FOR INSTRON 8562 TEST SYSTEM

The following describes the operating procedures for conducting a creep and a stress relaxation tests:

(1) Displacement and load feed back X-Y recorder calibration:

   (A) Displacement: (a) Turn power on for test frame and X-Y recorder, no sample;
   (b) Position control - no suppression, Display 1 - position - track; (c) Position actuator
       midstroke (set point to zero); (d) Output Y channel - position; (e) Mark off 20 cm on
       chart paper (Y); (f) Select 2.5 mV/cm on Y channel; (g) Adjust pen to zero with Y
       position; (h) Set actuator to 0.25 mm and wait a few seconds for system to stabilize; (i)
       Recorder to variable; (j) Adjust variable for full scale, i.e. 20 cm (equal to displacement
       0.25 mm); (k) Do not touch variable again, otherwise the scaling calibration is upset.

   (B) Load: (a) Output X channel - load; (b) Mark off 20 cm on chart paper (X);
       (c) Select 2.5 mV/cm on X channel; (d) Put in standard calibration sample loosely and
       set load to zero; (e) Load control; (f) Adjust pen to zero with X position; (g) Set actuator
       to 500 N and wait a few seconds for system to stabilize; (h) Recorder to variable; (i)
       Adjust variable for full scale, i.e. 20 cm (equal to load 500 N); (j) Do not touch variable
       again, otherwise the scaling calibration is upset.

(2) Creep test: (a) Balance the load of sample - set up key; (b) Put in actual
    sample loosely and set load to zero; (c) Heat up sample; (d) Zero suppression for position
    prior to heating for thermal expansion output; (e) At desired temperature, zero suppress
    again; (f) Set point for load then zero suppress immediately after load settling for elastic
strain value: (g) Set the X-Y recorder to record the creep strain versus time creep curve. The furnace self adaptive temperature control operating procedures: (a) Sample in; (b) Turn on power; (c) Close furnace doors; (d) Check sensor type (R) shows (5); (e) Primary control - thermocouple operational; (f) Set temperature - clear to change - then enter; (g) Run; (h) Toggle clear; (i) Primary control to monitor.

(3) Stress relaxation test: (a) Creep test to steady state stage; (b) Y channel to load; (c) Check the Y channel load on chart is equal to the creep load; (d) Start recorder for load, one will see a constant load; (e) Check furnace and temperature; (f) Turn off the actuator to start stress relaxation test, one will see the load decreases with time.

(4) Some important observations: (a) The Instron Model 8560 test system is a very sensitive machine which must be operated with great care; (b) Adequate practice with the machine and some actual specimens is strongly recommended for all users (either new or qualified); (c) In some cases the emergency stop (red buttons in front of panel and on microprocessor control panel) may be used; (d) The maximum capacity of the system is 100,000 N for load and 50 mm for displacement (corresponding 10 Volts output) must not ever be exceeded; (e) Call Mr. John D. Sollen, or other representatives from Instron Canada, Ltd., Burlington, Ontario, for any technical assistance on the machine.
PUBLICATIONS RESULTING FROM THIS WORK

1. Refereed Journal Publications:


II. Refereed Conference Proceedings:


VITA AUCTORIS

Longquan Shi, born in June 1963, Linchuan, Jiangxi, P.R. China.

B.A.Sc.: July 1983, Department of Materials Science and Engineering, Zhejiang University, Hangzhou, Zhejiang, P.R. China.

M.A.Sc.: July 1986, Institute of Metal Research, Academia Sinica (Chinese Academy of Sciences), Shenyang, Liaoning, P.R. China.
