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**HIGH TEMPERATURE FLOW AND FORGING BEHAVIOUR
OF
SUPERALLOY COMPACTS AND COMPOSITES**

By

ABDEL-RAZEK YOUSSEF KANDEIL, B.Sc., M.Eng.

A Thesis

**Submitted to the School of Graduate Studies
in Partial Fulfilment of the Requirements
for the Degree
Doctor of Philosophy**

Carleton University

March 1979

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HIGH TEMPERATURE FLOW AND FORGING BEHAVIOUR

OF

SUPERALLOY COMPACTS AND COMPOSITES

The undersigned hereby recommend to the Faculty of Graduate Studies and Research, Carleton University, Ottawa, acceptance of the thesis "High Temperature Flow and Forging Behaviour of Superalloy Compacts and Composites", submitted by Mr. Abdel-razek Y. Kandeil, B.Sc., M.Sc., in partial fulfillment of the requirements of the degree of Doctor of Philosophy in Mechanical Engineering.

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CARLETON UNIVERSITY
Ottawa, Ontario.

TITLE: High Temperature Flow and Forging Behaviour of Superalloy Compacts and Composites

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SCOPE AND CONTENTS: The purpose of this work was to explore the general feasibility of forging fiber reinforced composites, to improve the fundamental understanding of these materials and to contribute to the development of advanced fabrication processes.

* Equipment for isothermal isostrain rate compression testing has been designed and built around a 10,000 kg MTS servo-controlled hydraulic testing frame. The system is capable of testing up to 1200°C, and at constant strain rates between 10^{-5} and 1 s^{-1} . This equipment has been used to determine the high temperature flow behaviour of a series of hot isostatically pressed nickel-base superalloy compacts, and some metal matrix composites of these materials containing high strength tungsten wires.

Results are presented showing the form of the flow stress versus strain curves together with metallographic examinations of the forged nickel-base superalloy compacts under conditions of constant temperature and true strain rate and known starting microstructure. From these it is concluded that the flow and fracture behaviour of these materials can be greatly influenced by the forging variables and the microstructure, particularly grain size. The overall level of flow stress in the fine grained superplastic material is considerably lower than that of a material in the more conventional coarse grained condition. Higher strain rate sensitivities and improved ductilities are obtained in the former relative to the latter. Means by which grain size can be controlled in superalloy compacts are examined

and the results of the influence of the established grain size on the forging characteristics of these materials are presented.

The study of the forging behaviour of the fiber reinforced composites has been undertaken. In particular the response of the composites to variations in deformation conditions has been examined and compared to that of the matrix alone. Under identical working conditions, peak flow stresses for the composites are up to four times greater than those of the non-reinforced matrix. Furthermore, the strain rate sensitivity of the composite is approximately half that of the superplastic matrix. A rationalization of these differences is presented.

A forming limit criterion of a geometric nature has been developed which accounts for fiber cross section, stacking arrangement and volume fraction. From the model considered, gross damage may occur in the composites when they are deformed beyond a critical strain, which is a function of the volume fraction, stacking arrangement and cross section of fibers. In the present case, this critical strain is shown to be 0.34 and forming of the composites should therefore be limited to strains lower than this level.

Formability is also shown to be limited at low strains by the formation of voids at the tensile poles of the fibers, normal to the loading direction. The critical parameter in this case is the strength of the fiber/matrix interface relative to that of the matrix. Means of improving the formability are discussed.

To my parents

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LIST OF PRINCIPAL SYMBOLS

σ	flow stress
$\dot{\epsilon}$	strain rate
m	strain rate sensitivity coefficient = $\partial \ln \sigma / \partial \ln \dot{\epsilon}$
Q	activation energy
T	absolute temperature
R	gas constant
d	grain size
a	grain size sensitivity exponent
σ_o	friction stress
σ_T	tensile stress
σ_T^*	critical stress
σ_d	bond strength
f_o	strength of inhomogeneity = t_g/t
t_g	thinned thickness
t	matrix material thickness
P	applied compressive stress
q	lateral pressure
x	lateral distance measured from center
L	length of specimen
h	specimen height
μ	friction coefficient
A	instantaneous cross-sectional area of compression specimen
A_o	initial value of A
h	instantaneous height of compression specimen

h_0	initial value of h
ϵ	true strain
A_f	final cross sectional area of extruded bar
$\dot{\epsilon}_T$	total strain rate
$\dot{\epsilon}_p$	plastic strain rate
$\dot{\epsilon}_e$	elastic strain rate
E	elastic modulus
V	volume of specimen = Ah
R	extrusion ratio = billet C.S.A./product C.S.A.
P_E	extrusion pressure
H_V	Vicker's hardness
σ_e	effective stress
σ_i	internal stress due to dislocations
σ_p	internal stress due to particles
S	structure parameter
R_d	ratio of initial grain size to steady state grain size
m^*	strain rate sensitivity of E^*
E^*	apparent modulus of elasticity
p	volume fraction of undeformed particles
ϵ^*	effective strain
$\dot{\epsilon}^*$	effective strain rate
v	volume fraction of fibers
$\dot{\epsilon}_r$	radial strain rate
$\dot{\epsilon}_z$	axial strain rate
σ_{if}	internal friction stress
σ_{ef}	external friction stress

ϵ_c critical strain
 h_c critical height
 d' diameter of fiber

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1 INTRODUCTION

Materials and processing technology continue to play a key role in the development of advanced high temperature turbo-machinery. A review of potential materials systems which can meet the anticipated demands for high temperature operations has indicated that significant temperature increases of up to several hundred degrees celcius could be obtained by research and development efforts in artificial metal matrix composites, directionally solidified (DS)-eutectic composites, and ceramics¹. The potential temperature advantage of these materials over state-of-the-art materials is shown in Fig. (1).

Fiber reinforced superalloy-matrix composite materials combine superior properties of oxidation resistance, high strength coupled with ductility, and toughness at elevated temperatures. One principal drawback of these composites is that they are extremely difficult to machine or form by conventional metal working processes due to the strongly anisotropic nature of their properties. Furthermore, the forging of composite materials may be limited by the anticipated large stresses required, the numerous defects that might occur during forging, and their limited forgeability². In Canada efforts are being made to develop high temperature composite materials and the fabrication processes necessary to produce composite turbine vanes and blades for advanced industrial gas turbine engines³. Emphasis in this program has been on a direct press-to-shape approach, where final part configuration would be achieved by hot-isostatic pressing of shaped cans. Success in this program will require that close dimensional tolerances be achieved in the products, that gross distortion and internal defects can be avoided, and that acceptable mechanical properties can be obtained in the pressings^{4,5}. If any of these requirements cannot be met it will be necessary to consider the use of secondary deformation processing, either to complete the shaping operation or possibly to allow an additional degree of control on the microstructure.

Isothermal forging is one of the more important processes to have evolved in recent years⁶. In this type of forging the billet and dies are maintained at the forging temperature during deformation. Slower forging rates can therefore be used, and with highly strain rate sensitive materials forging loads may be reduced to as little as 10% of the conventional forging loads⁷. At the same time chilling at the die-billet interface is avoided⁸, lubricants become more effective⁹ and improved die filling is obtained⁷⁻¹⁰. The overall result is improved workability of deformation resistant materials. By combining this process with powder fabrication, highly alloyed casting-type alloys can be processed into homogenous wrought products^{11,12}.

Nickel-base superalloy powder compacts can be produced to have a very fine grain size such that superplastic flow is observed under certain conditions of temperature and strain rate¹³. The possibility exists therefore that composites with a superplastic matrix can be produced. By causing flow to occur superplastically around the deformation resistant fibers, it may be possible to achieve strains at low strain rates that would otherwise be difficult to achieve. Shape considerations, the need to maintain fiber-matrix integrity, and to avoid fiber damage, as well as the need to maintain superplastic flow in the matrix suggest that isothermal forging may be necessary.

The purpose of this work was to explore the general feasibility of forging fiber reinforced composites and to improve the fundamental understanding of metal matrix composites and compacts.

In order to fully appreciate the complications and limitations imposed by the strong reinforcing fibers it was decided first to characterize the forging behaviour of the candidate matrix alloy compacts without the reinforcing fibers. The aim of this part of the project was to provide a comprehensive study of the means by which grain size can be controlled in superalloy compacts, and secondly to examine the influence of the established grain size on the forging characteristics of the materials. Furthermore, since the high

temperature flow and fracture behaviour of superalloys was expected to be sensitively dependent on temperature and strain rate, these variables were also investigated. Overall, it was hoped that this work would provide an improved understanding of hot working mechanisms and superplasticity in superalloy compacts.

The lack of a comprehensive study of the forging and fracture behaviour of fiber reinforced superalloy composite materials, justifies a fundamental approach to the problem. No information was available in the literature regarding forging loads, fracture behaviour and forging defects that might occur during processing. The purpose of this study was to provide some of this information using, as a model, an advanced composite system of practical engineering interest.

2 BACKGROUND REVIEW

In this chapter a short review is provided of published literature describing the powder metallurgy processing of nickel-base superalloys and composites. The chapter opens with an overview of techniques used to produce and consolidate superalloy powders. Following this is an outline of methods used to form consolidated superalloy billets into useful shapes. Two of the more important processes covered are isothermal forging and superplastic forging. These are discussed in greater detail since they are the subjects of the experimental portion of this thesis. Following this is a general review of methods used to produce composite materials. Deformation processing of composite materials is covered in greater detail, and a particular emphasis is placed on work describing the forging behaviour of composites. Since most composites are expected to have rather limited formability and ductility, the fracture behaviour and forming limits of composites are examined in detail.

2.1 SUPERALLOY POWDER PROCESSING

During the past decade, significant advances have been made in powder metallurgy (P/M) technology. The motivation for the use of powder metallurgical processing lies in one or more of the following: (i) cost savings from reduced material input and a reduction in post-fabrication machining, (ii) improved fabricability of high strength alloys (superplastic alloys), (iii) property improvements associated with the increased homogeneity of pre-alloyed metal powders, (iv) production of advanced structural materials such as composites.

Three powder production techniques are currently being used for the manufacture of superalloy powder¹⁴. They are: (a) The Rotating Electrode Process (REP) which consists of melting the end of a rapidly rotating bar by means of an electric arc, wherein the molten metal spins off the bar and solidifies before hitting the wall of an inert

gas filled tank, Fig. (2). (b) The inert gas atomization process in which molten superalloy material is poured through a nozzle where argon gas impinges on the molten stream, atomizing it into powder. The particles solidify before hitting the container walls, Fig. (3). (c) The vacuum atomization process which is based upon the sudden exposure of a super-saturated solution of hydrogen in superalloy to a vacuum. The superalloy explodes into the vacuum chamber via a transfer tube and atomizes as the gas expands and is ejected from the metal Fig. (4).

2.2 CONSOLIDATION METHODS

Because of the high alloy content and spherical morphology of most superalloy powders, they cannot be consolidated by conventional techniques such as cold pressing¹⁵. Additionally, these powders do not sinter readily, on account of the presence of aluminum, chromium, titanium, etc. which will oxidize at the sintering temperatures¹⁶. Consequently, other techniques for powder consolidation, requiring application of pressure at elevated temperatures, have to be used. Three methods are currently employed, namely, hot isostatic pressing (HIP), hot extrusion, and hot pressing or forging. One objective of these consolidation methods is to produce preforms that can subsequently be forged into parts of specified dimension and shape. A short description of these consolidation methods follows.

(a) Hot Isostatic Pressing (HIP)

The hot isostatic pressing (HIP) process consists of canning the powder in an evacuated container (mild or stainless steel, ceramic, glass, etc.) molded to the desired shape, and exposing the can simultaneously to high temperature and pressure (typically 1000 to 1250°C and 70 to 140 MN/m²).

The conditions of temperature, time and pressure selected for hot isostatic pressing should be such that full densification and interparticle bonding are achieved.

This requires that the applied pressure is in excess of the flow stress of the powder at the

pressing temperature, and that the temperature is sufficient to allow diffusion bonding to occur between particles. These conditions define broad temperature ranges within which considerable variations in microstructure can be achieved. It has been demonstrated for example, that grain sizes in the range 2 - 8 μm can be obtained in Mar M200 compacts pressed below the γ' solvus temperature ($1200^\circ\text{C} \pm 15^\circ\text{C}$), while coarser grain sizes in the range 20 - 200 μm can be obtained at pressing temperatures above the γ' solvus¹⁷. The presence of coarse γ^* on grain boundaries during HIP treatment below the γ' solvus stabilizes the fine as-atomized grain size of powders, while recrystallization and growth to a coarse grain size occurs during pressing above the γ' solvus.

The process provides excellent uniformity of grain structure, chemical homogeneity, and the ability to produce large and complex shapes such as are required for forging preforms, as well as the ability to produce as pressed parts directly. A schematic representation of the process is shown in Fig. (5).

(b) Hot Extrusion

Direct powder extrusion combines hot compaction and hot mechanical working to yield a fully dense wrought material. In this process, containers, commonly made of stainless steel, are filled with the powder to tap density, evacuated and sealed prior to extrusion. The frictional forces developed during extrusion yield a shear component that gives rise to interparticle shearing, breakdown of prior particle boundaries and enhanced interparticle bonding¹⁶. A large number of nickel- and cobalt-base superalloy powders have been successfully extruded into fully dense rods¹⁶. The variety of shapes and sizes that can be produced is however limited. The microstructure of as-extruded bars is usually fine grained and requires subsequent heat-treatment to allow grain growth for improved high-temperature creep properties.

* The γ' (Ni₃Al,Ti) phase is the major precipitation hardening phase used for strengthening during service operation at temperatures generally lower than those used for pressing, extruding or forging.

(c) Hot Pressing or Forging

The process illustrated in Fig. (6), consists of pouring the loose powder into a die cavity under vacuum. The preferred die material is usually graphite or a molybdenum base alloy (such as TZM). Dies and powder are pre-heated prior to the application of pressure. Consolidation is achieved by applying pressure via one or two punches, made of the same material as the die. However, this process is expensive and full density in the compacts is generally not achieved¹⁶. A similar but relatively simple process is the direct forging of powder. In this process cans are filled with powder, evacuated and sealed prior to the application of pressure¹⁸.

2.3 DEFORMATION PROCESSING OF POWDER COMPACTS

Clearly, considerable cost savings can be realized by net-shape processing of powders, that is, consolidation to near final part configuration such that only subsequent machining is required. This approach is being actively pursued with respect to turbine discs and shafts^{19,20}, and also fiber reinforced guide vanes³. However, because of the uncertainties relating to dimensional control and mechanical properties in as-pressed parts, considerable work is being done to explore deformation processing of powder compacts. One school of thought is that forging after pressing provides an additional degree of control of final microstructure, and the ability to eliminate as-pressed defects such as porosity and embrittling powder surface boundary films^{17,21}. Also, with the advent of isothermal forging and superplasticity, precision close tolerance parts can be produced thus improving dimensional control and minimizing machining.

A number of routes involving various consolidation and metal working processes have been considered for producing shaped parts from metal powders. The most extensively studied are^{19,22}:

- (a) Consolidation + conventional forging (multi-stage forging)
- (b) Consolidation + secondary working (extrusion) + isothermal forging (Gatorizing)

- (c) Thermo-plastic processing + HIP + isothermal forging
- (d) Direct HIP + isothermal forging
- (e) Direct HIP to near net shape.

(a) Consolidation plus Forging Process

In this process powder is consolidated by hot isostatic pressing (HIP) or extrusion in the 1000 to 1250°C temperature range followed by conventional forging to a disc sonic* shape. The grain size of the consolidated P/M product should normally be less than about 200 μm and therefore less than that of a casting. Decreased forging loads to as much as one-tenth of that of cast and wrought superalloys should be observed⁷. Where HIP consolidation is used shaped preforms can be produced and reduced forging reductions and fewer forging operations may be needed to produce the final configuration.

(b) Isothermal Forging

The best established process for isothermal forging of superalloys is the Pratt and Whitney "Gatorizing Process", which is now being used commercially to produce IN-100 discs for the Pratt and Whitney F-100 engine¹¹. Starting with a consolidated powder billet, three stages of processing are involved⁷:

Stage 1. Involves compressive working to high strains, such as by extrusion or forging, at temperatures just below the recrystallization temperature of the alloy. Adiabatic heating causes controlled recrystallization of the billet to produce the fine grain size necessary for superplasticity.

Stage 2. Involves hot die forging at constant temperature and slow strain rate. To retain the superplastic state during forging, the forging process must be

* Simple shape which can be nondestructively tested by means of ultrasonic techniques for internal defects.

carried out at a temperature below the solvus temperature of the gamma-prime phase (γ') in the alloy.

Stage 3. Involves a heat treatment operation in which the fine superplastic grain size is destroyed and a coarse grain size is developed to re-introduce the necessary high temperature strength for service operation.

(c) Thermo-plastic Processing

Thermo-plastic processing takes advantage of the fact that when a super-alloy powder particle is cold worked, its grain size may be greatly reduced on recrystallization, and superplastic behaviour can be achieved at elevated temperatures. The strain energy can be imparted to the powder in a number of ways ranging from milling with a mortar and pestle to high energy ball milling. The most commercially attractive technique is to roll the powder through a vertical feed rolling mill. As the powder is passed through the mill, each particle is flattened into a miniature disc. The powder can then be HIPed to produce a fine grained superplastic forging preform, thus eliminating the hot extrusion step inherent in the Gatorizing Process. Alternatively, the powder might be HIPed directly to some shape, thus eliminating both the extrusion and forging operations of the Gatorizing Process.

T/P processing offers other technical and economic advantages over the conventional P/M techniques^{15,19}. Atomization efficiency is expected to be enhanced since more of the output of the atomizer can be utilized. The larger powder (+100 mesh, 149 μm) which is not readily workable by conventional P/M processing is rendered more workable by the T/P process. Furthermore, hot isostatic compaction of T/P processed powder can be achieved at lower temperatures than are possible with conventional powder. This should lead to improved HIP furnace life and lower energy consumption.

(d) Direct HIP and Isothermal Forge

It has been realized that most types of atomized powder have a sufficiently fine grain size to possess superplastic properties¹⁷. Therefore, if the powder could be consolidated to retain this inherent fine grain size, the as-pressed compact could be used as the preform for a superplastic forging operation. Irrespective of the method of compaction used, the pressing operation would be carried out at a temperature above about 0.7 of the solidus temperature of the alloy (homologous temperature* $\sim 0.7 T_M$) but below the γ' solvus temperature. The presence of the γ' phase is used to stabilize the structure developed during pressing and in particular to prevent grain growth so that the original fine grained structure is maintained, thereby ensuring superplastic behaviour during the subsequent forging operation.

2.4 SUPERPLASTICITY

Superplasticity is the ability of a metal or alloy to deform extensively under small tensile, compressive or torsional loads without suffering plastic instabilities^{23,24}. Under these conditions, generally observed at high temperatures and slow strain rates, the metal or alloy is characterized by a high ductility well in excess of conventional behaviour, and it deforms at stress levels well below the flow stress for the non-superplastic form of the material. During tensile testing, elongations before fracture of the order of 1000% or more are obtained in some cases^{25,26,27,28}, and a decrease in flow stress of one or more orders of magnitude is not uncommon^{29,30}. Furthermore, in its superplastic form, the material exhibits a higher strain rate sensitivity.

Several requirements must be satisfied if superplasticity is to occur. The lower temperature limit for its occurrence is approximately one-half the absolute melting temperature of the alloy ($\frac{1}{2} T_M$). The material must also be homogeneous, free of

* Temperature relative to melting temperature expressed in °K.

contaminants, and have a stable and extremely fine grain size of less than about $10\ \mu\text{m}$ ^{13,27,31}. Detailed investigations indicate that superplastic behaviour is caused by a shear process in the grain boundary layer²⁶. Grain boundary shear is similar to Newtonian flow* and strongly resembles the viscous flow of glass at high temperature. It is believed that the growth of gamma-prime particles, in some superalloys, combined with their movement and sliding action is responsible for the phenomenon of superplasticity³². However, a completely satisfactory mechanism has not been developed.

Superplastic behaviour is obviously attractive for use in certain forming processes. Some advantages of superplastic forming are: improved formability, reduced forming pressures, closer dimensional tolerances, minimized wastage of material, more uniform microstructure and improved service properties as compared to conventionally formed materials.

2.5 HIGH TEMPERATURE FLOW OF SUPERPLASTIC MATERIALS

The high temperature flow stress of superplastic materials is a sensitive function of strain rate, temperature and grain size, and their flow behaviour is quite different from that of conventional materials.

Above $0.5\ T_M$, a sigmoidal relationship between flow stress and strain rate, when plotted on logarithmic scales, is characteristic of superplastic materials, Fig. (7-a). A strain rate sensitivity much higher than in conventional materials is observed at intermediate strain rates, usually between 10^{-4} and $10^{-1}\ \text{s}^{-1}$. Such flow data are most commonly correlated at a given temperature by means of a power relationship of the form:

$$\sigma = K\dot{\epsilon}^m \quad (1)$$

* In which the shear stress (τ) is proportional to the shear strain rate $\left(\frac{\partial u}{\partial y}\right)$.

where σ is the flow stress, K is a constant*, $\dot{\epsilon}$ is the strain rate and m is the strain rate sensitivity coefficient which can be written as:

$$m = \left(\frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right)_{T, \text{structure}} \quad (2)$$

Figure (7-b) shows the variation of m with strain rate at constant structure and temperature. For convenience, the $\ln \sigma / \ln \dot{\epsilon}$ curve may be divided into three regions. At high strain rates, region III, flow is conventional and $m \sim 0.25$. This region embraces strain rates typical of conventional mechanical working operations. At intermediate strain rates, region II, the strain rate sensitivity increases markedly. Values ranging from 0.3 to 1.0 have been reported but generally m values of 0.5 to 0.6 are more typical³³. At low strain rates, region I, m usually decreases to values below 0.3.

In superplastic nickel-base superalloys, the high strain rate sensitivity has been shown to extend over more than four orders of magnitude and no decrease in m was detected even at the lowest creep strain rates investigated³³. However, it is possible that the transition to region I was not encompassed in this case within the experimental range investigated. In general, ductility increases with m and it is within region II that the very large elongations to failure, characteristic of superplastic materials, are observed.

The following changes in the flow behaviour of superplastic materials occur with increasing deformation temperature:

- (i) the overall level of the flow stress is reduced.
- (ii) the maximum value of m increases and is displaced to higher strain rates.
- (iii) both transitions between regions I-II and regions II-III, are displaced to higher strain rates.

* at constant structure.

- (iv) at the extremes of strain rate, the value of m is less sensitive to temperature variations than in the superplastic region II.

The temperature dependence of the flow stress is normally described in terms of an arrhenius expression which combined with the strain rate dependence leads, at constant structure, to a relationship of the form:

$$\sigma = \delta \left(\dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \right) \quad (3)$$

Here, T is the absolute temperature and Q is an activation energy related to the rate controlling restoration process³⁴.

As pointed out in the previous section, superplastic behaviour is only observed for small grain sizes ($\sim 10 \mu\text{m}$). Below this value the overall influence of decreasing the grain size on the $\ln\sigma/\ln\dot{\epsilon}$ relationship is qualitatively similar to increasing the deformation temperature or decreasing the strain rate. The effect of grain size on the flow stress of a superplastic nickel-base superalloy has been investigated³⁵. The results of this study indicate that, at constant strain rate and temperature, the flow stress σ is directly proportional to the grain size d :

$$\sigma \propto d \quad (4)$$

In superplastic nickel-base superalloys the flow stress has also been shown to depend strongly on other structural parameters and in particular on the second phase distribution and morphology, which also indirectly control grain size during dynamic recrystallization¹³. In view of its strong structure dependence, the flow stress in superplastic materials should therefore be identified unambiguously as either a yield stress (at a given strain offset), a peak stress or a steady state flow stress characteristic of large deformations at elevated temperatures³⁴.

Since high temperature deformation data is generally collected by means of creep testing and because the formalism of a rate equation is particularly convenient, constitutive equations developed for high temperature flow and for superplasticity usually

treat the flow stress as the independent variable. Hence, the strain rate is generally represented by an equation of the form

$$\dot{\epsilon} = \delta(\text{stress, temperature, structure}) \quad (5)$$

For engineering application purposes, a combination of equations 1, 3 and 4 leads to the most commonly used empirical constitutive equation for superplasticity:

$$\dot{\epsilon} = K \sigma^{\frac{1}{m}} d^{-a} \exp(-Q/RT) \quad (6)$$

Here K is a constant, m is the strain rate sensitivity exponent defined by equation (2), d is the grain size, a is the grain size sensitivity exponent, Q is the apparent activation energy for flow and $\dot{\epsilon}$, σ , R and T have their usual meaning. Care must be exercised in using equation (6) since m can vary considerably with strain rate and temperature^{26,36,37,38}. However, as pointed out previously in a nickel-base superalloy, in the superplastic range $m = 0.5$ and $a = 2$ over more than 4 orders of magnitude of strain rate.

Functional relationships other than the power law can also be used to correlate stress and strain rate. One proposed expression uses an exponential relationship and takes the form²⁶:

$$\dot{\epsilon} = A \exp\left(\frac{B\sigma}{T}\right) - A \exp\left(\frac{-B\sigma}{T}\right) \quad (7)$$

Here, A and B are regarded as material constants. It should be pointed out, however, that in superplastic nickel-base superalloys, A depends strongly on grain size as well as on other structural parameters and can therefore change during plastic flow for instance with grain coarsening or grain refinement of the material.

Some other more recent constitutive equations for high temperature flow are given in references 37 to 39. One expression takes the form:

$$-\dot{\epsilon} = K_1 (\sigma - \sigma_0) + K_2 \sigma^{\frac{1}{m}} \quad (8)$$

where K_1 , σ_0 , K_2 and m are constants determined by a least square fit³⁷ or from the fundamental physical properties of the material⁴⁰. Worthy of note is the use of a friction stress (σ_0) which is related to the structure of the material and which opposes the deformation. Again care must be taken in applying this relationship since if the structure changes during deformation then so does σ_0 ³⁸.

Generally, the above constitutive equations are simplified versions of equations derived rigorously from rate theory in an attempt to describe superplastic deformation in terms of either single or several deformation mechanisms. The reader is referred to an excellent recent review⁴¹ for a comprehensive treatment of the subject.

There is considerable controversy over the mechanism of superplastic deformation and many theories exist that attempt to describe it in terms of either single or several deformation mechanisms^{30,33,41}. Most of these theories are quite inadequate, although a few have been reasonably successful in describing some of the experimental observations. Assessment of the various mechanisms proposed requires:

- (a) approximate prediction of stress/strain rate data at particular temperatures for a given microstructure and
- (b) agreement with topological and morphological observations.

It is not intended here to discuss and assess the different models presented in the literature, but rather to present a very short description of the most relevant mechanisms in order to explain some of the results obtained in the present study.

The experimental evidence indicates that several processes are occurring simultaneously during superplastic deformation, including grain boundary sliding, dislocation motion and diffusional processes⁴¹. Consequently there is no reason to assume that there is a unique mechanism of superplasticity. Modern views on superplasticity suggest that it is more likely that there are ranges of conditions where different mechanisms predominate and produce particular combinations of mechanical properties and microstructural change.

There is general agreement that the deformation in region III takes place mainly by dislocation climb. Although, flow takes place mostly intragranularly, there is some evidence for grain boundary sliding⁴¹. In contrast, the dominant mechanism during superplastic deformation (region II) is almost certainly grain boundary sliding, and the grain boundary strains are accommodated intragranularly by diffusional flow⁴⁰ and/or dislocation motion⁴². Therefore, it seems reasonable to assume that grain boundary sliding and intragranular flow are the main mechanisms contributing to plastic flow in stages II and III. The strain rate for intragranular flow, at constant temperature, is given by McQueen⁴³

$$\dot{\epsilon} = A \sigma^n \quad (9)$$

where, A is a constant for constant structure and $n = 4$. On the other hand, for grain boundary sliding, at constant temperature, the strain rate is given by Gittus³⁰

$$\dot{\epsilon} = B \left(\frac{\sigma}{d} \right)^{n'} \quad (10)$$

where, B is a constant for constant structure and $n' = 2$.

2.6 COMPOSITE MATERIALS

2.6.1 Introduction

Most of the metal composite development has taken place in the last fifteen years. The existing and projected applications of metal composites illustrate some of the benefits. However, review of properties indicates some areas where further work is needed⁴⁴.

The two basic types of metal-matrix composites are:

- (i) synthetic (filamentary or fiber reinforced) and
- (ii) in-situ (directionally solidified eutectics).

In filamentary, or fiber reinforced composites, differences in chemical potential at fiber/matrix interfaces provide a driving force for chemical reaction and/or diffusion.

This chemical instability can produce a change in the composition and amount of one

or both phases, resulting in deleterious effects on mechanical properties^{2,45-47}. In directionally-solidified eutectic composites, the two phases are essentially in chemical equilibrium and the microstructure should be stable at elevated temperatures.

However, production of complex shapes by the latter technique is restricted because of the requirements for careful control of the temperature gradients and solidification rates. On the other hand, because of their higher melting points fiber reinforced composites have the potential for application at temperatures above those of the d.s. eutectics as shown in Fig. (1). Also, the specific rupture strengths of the refractory wire/superalloy composites are greater than those for the d.s. eutectics⁴⁸.

The following review deals with fabrication methods and forging of fiber reinforced composites only.

2.6.2 Primary Fabrication Routes of Fiber Reinforced Composites

Fabrication techniques used for different combinations of fiber and matrix materials relate directly to the type of materials involved⁴⁹. Other factors to consider are the shape to be produced and the number required. Some of the important fabrication methods utilized are listed in Appendix I. These techniques can be divided into solid phase or liquid phase processes. This study deals with composites produced by solid phase techniques and a brief description of the different processes which may be involved is provided in the following sections.

The solid state processes of diffusion bonding, hot pressing or cold pressing plus sintering, all have the prerequisite that the matrix be in either thin sheet (foil)^{2,50,51} or powder form^{3,45,51,52}. In one process, tapes are fabricated by mixing alloy powder with an organic binder to form a slurry which is passed through an orifice of appropriate dimensions and is then oven dried to form the required tape³. Alternatively, the powder may be warm rolled into high density sheets⁵¹. Sandwich structures of reinforcing fibers and matrix tape referred to as composite

tapes are rapidly becoming the customary starting material because of the ease with which these laminar structures can be cross plied, stacked and hot pressed to form the final product shape⁵³.

Several methods can be used to process composite tapes:

(i) Hot pressing

In this process the fibers are placed between the matrix tapes to form a mono- or multi-layered sandwich. Diffusion bonding, between the matrix tapes and fibers, takes place by pressing in a controlled atmosphere between heated dies. The pressure exerted by the dies causes the matrix to flow between the fibers by creep. Temporary conditions of low strength combined with high ductility can be maintained in the matrix alloy while processing⁵⁴.

(ii) Mechanical working

In this method, composites can be produced by working (extrusion, drawing or rolling) a two-phase alloy⁵⁵, or blended powder^{56,57}, thereby producing a structure which consists of fibers of a strong phase in a ductile matrix. Successful working of such materials requires that the strong phase to be plastic at the working temperature usually between 0.4 and 0.75 of the melting point (in °K)⁵⁵. Continuous hot rolling can be used to produce fiber reinforced composites, by feeding the tape of matrix material and fibers simultaneously into heated rolls as shown in Fig. (8). The main problem in continuous hot rolling is to prevent fracture of a brittle reinforcement, i.e. to promote consolidation without longitudinal extension⁴⁴.

As indicated previously Westinghouse Canada is currently engaged in a program to develop high temperature composite materials, and the fabrication processes to produce composite turbine vanes and blades for advanced industrial gas turbine engines³. One composite system includes the starting matrix Mar M200

nickel-base superalloy powder and the fibers, which are high strength tungsten-2% thoria wires. The fabrication work involves development of continuous diffusion barrier coating of the wire, monolayer composite ply production techniques, powder metallurgy compaction by hot isostatic pressing, and thermomechanical processing (TMP). The processing route under development at Westinghouse Canada is shown in Fig. (9). Similar approaches have been used to produce high quality composite blades from nickel-chromium alloy and tungsten or molybdenum reinforcing fibers⁵².

2.6.3 Forging and Fracture Behaviour of Fiber-Reinforced Composites

Simple shapes of composite materials produced by the techniques described above may be used as the starting material from which complex shapes, such as turbine blades, might be forged. However little work has been reported on the response of such materials to the forging processes.

The transverse compression behaviour of composite materials was studied by Collings⁵⁸. Using a unidirectional carbon fiber reinforced epoxy system, it was observed that loading in compression normal to the fiber direction caused shear failure along planes parallel to the fibers, Fig. (10). Constraining the composite from lateral deformation normal to the fiber direction increased the compressive stress at failure substantially. The failure mode changed to shear through the fibers on planes parallel to the direction of constraint as shown in Fig. (11). Unconstrained transverse compressive strength was not a strong function of fiber volume fraction, whereas constrained transverse compressive strength increased substantially with increasing volume fraction.

Ertürk et. al.², studied the plastic deformation behaviour of aluminum-stainless steel fiber composites under conditions of simulated forging. With the load applied perpendicular to the direction of reinforcement, it was found that

deformation occurred in a plane strain mode with no flow in the direction of fiber alignment. Types of material damage were identified and forming limits were established as will be explained later in Section 2.7.

In general the study of metal-matrix composites has mainly been concentrated on macroscopic properties and behaviour. Very little work has been done to analyze flow and fracture from a microscopic view point. In these materials, the fibers can be thought of as rod-like rigid inclusions embedded in a soft matrix. It is well known that inclusions, precipitates and dispersions are most important in controlling ductile fracture in two phase structures⁵⁹. One would therefore expect the fibers and the characteristics of the fiber/matrix interface to control ductility and formability in the composites. Several studies on the analysis of interface stresses and fracture characteristics in two phase alloy systems have been reviewed by Rosenfield⁵⁹, and more recent models are reported elsewhere⁶⁰⁻⁶⁷. The most relevant of these models are reviewed in the following section.

2.6.4 Models of Stress Analysis and Void Initiation Around Inclusions

It is a well documented fact that voids are normally concentrated in bands or regions of heavy slip and that they nucleate at inclusions or irregularities⁶⁸⁻⁷⁰. Ashby^{71,72} has developed a dislocation model of plastic flow around rigid inclusions contained within shear bands. In his work Ashby demonstrated that particles within shear bands will have tensile stresses set up at the particle poles. He then proposed a dislocation model which describes how these stresses can be relieved.

Following Ashby, the interaction between a spherical hole and a slip band as shown in Fig. (12-a) is examined. Under tensile loading, the spherical hole will tend to elongate and flatten into an ellipse, with the greatest extension occurring roughly parallel to the tensile axis and the greatest contraction roughly perpendicular to it. Now, if the hole contains a spherical rigid particle, totally bonded to the walls

of the hole, the metal will attempt to take up the elliptical shape, but it will be constrained by the particle to retain its spherical shape. This will result in the stress distribution shown in Fig. (12-b). The interface stress is tensile in the direction of elongation and compressive in the transverse direction. A crack will form at the interface when the tensile stress (σ_T) reaches a critical value, σ_T^* , which is the stress required to produce separation of the matrix and the particle. Also, it has been shown that holes will form at larger particles at lower strains than at smaller particles. In other words, ductility decreases with increase in particle diameter.

Gurland⁷³ and McClintock⁷⁴ pointed out that inclusions cause fracture by a process that may be thought of as having three stages: initiation by the fracture of inclusions or by their separation from the matrix, growth of the resulting holes, and coalescence of the holes to form a macroscopic crack. It was suggested⁷⁴ that dislocation concepts are useful mainly for the first stage, while the second and third stages can be treated by continuum plasticity because of the relatively large deformations involved in these stages. For the present work, there is no need to discuss the subject of interface dislocations further. For a more detailed treatment the reader is referred to a recent extensive review⁷⁵.

Thomason⁶⁷ has developed a stress criteria for cavity nucleation and analyzed the problem of internal necking between particles, assuming a rigid-plastic plane-strain plasticity model. He showed that decohesion will not occur if the bond strength σ_d exceeds the maximum tensile component of the plastic flow stress σ_z , or $\sigma_d > \sigma_z$. From the Von Mises yield criterion, $\sigma_z = 2k + \sigma_x$, where k is the current value of the matrix yield shear stress and σ_x a transverse stress component ($\sigma_z > \sigma_x$). These considerations illustrate the important role of a compressive transverse stress in suppressing void initiation between particles.

The effects of a superimposed hydrostatic pressure have been discussed by Rosenfield⁵⁹. The classic experiments are those of Bridgman⁷⁶, and an example is shown in Fig. (13). The hydrostatic pressures (compression) that Bridgman achieved were sufficient to increase the fracture stress by a factor of $\sim 2\%$, and to improve the reduction in area from ~ 60 to $\sim 98\%$. Similar results have been reported by French and Weinrich^{69,77}. It was found that the strain to fracture increases approximately linearly with hydrostatic pressures up to about 300 MPa. Above this pressure, the increase in the strain to fracture tended to level off, as shown in Fig. (14). Experimental evidence⁷⁸ supports the existence of a functional relationship between the hydrostatic component of stress and structural damage. This evidence includes metallographic information, density changes, and reduction limitations of sheet drawn under both ambient and high fluid-pressure environments.

One factor not yet incorporated into the continuum theories is the great difference in ductilities actually observed in different alloy systems. A great number of metallurgical variables must be taken into account before ductility can be predicted theoretically. Some of these have been identified⁷⁹, and they include volume fraction, size, spacing, shape, fracture strength, and interfacial bond strength of the particles or fibers, as well as the yield stress, strain-hardening coefficient, and grain size of the matrix. Also, the forging variables of temperature and strain rate should be considered in order to obtain a meaningful prediction of ductility.

2.7 FORMING LIMITS

Ductility of a material is generally defined as the ability to deform plastically without fracture. It is usually expressed as a measure of the strain at fracture. For metal-working applications, however, it is important to recognize that effective ductility is not a unique property of the material; it depends on localized conditions of stress, strain, strain rate, and temperature in combination with material characteristics such as

inclusion content and grain size. Processing parameters associated with die design, workpiece geometry, and lubrication determine the local stress and strain states throughout the material and consequently the fracture behaviour of the material.

From experimental evidence of the influence of stress state on ductile fracture, several criteria have been suggested for the prediction of fracture in complex stress states. Perhaps the most advanced concept related to this is that proposed by Keeler and Backofen⁸⁰ for sheet metal forming, namely the forming limit diagram. By examining the fracture strain states in various biaxial stretch forming operations, it was possible to find a critical strain locus, as shown in Fig. (15), which divides a region of strain states which produce failure from states which do not.

This concept has recently been applied to axisymmetric shapes by Kuhn et. al.⁸¹ who produced forming limit diagrams for several steels in the tension-compression quadrant. They found that a linear relationship existed between the tensile and compressive surface strains at fracture and that the slope of this line on the compressive versus tensile strain axes is one-half. It may be noted that it is a combination of strains, rather than the value of any one strain, which governs fracture.

Lee and Kuhn⁸² proposed a fracture model for axisymmetric shapes based on the concept of Marciniak and Kuczynski⁸³ of localized thinning of material around an inhomogeneity. They considered the upsetting of a circular cylinder as shown in Fig. (16-a) in terms of surface strains at a point such as A. It was assumed that there would be surface inhomogeneities at such a point such as voids, inclusions or compositional changes. It was further assumed that these inhomogeneities could be represented as thinned sections as shown in Fig. (16) b and c. Two models were considered; an 'R-model', Fig. (16-b), which represents an inhomogeneity in the radial direction and a 'Z-model', Fig. (16-c), which represents the axial direction. The strength of the inhomogeneity is indicated by the ratio of thinned thickness to matrix material thickness t_g/t and is designated f_0 .

In Lee and Kuhn's model, as the cylinder is compressed, the thinned sections change in thickness more rapidly than the surrounding material under certain conditions, the rate of change being governed by the strain ratio and material parameters such as f_0 and the work-hardening rate. The limiting condition, representative of final fracture, was taken as the point at which $f \rightarrow 0$, i.e. when this section thins to zero thickness. Similar to the results of Kuhn et. al.⁸¹, the relationship between tensile and compressive strains at fracture was linear and had a slope of one-half. It has been shown that the slope of this relationship is a characteristic of the fracture process, while the height of the line (intercept with ordinate) is dependent on material parameters.

Following the analysis of Lee and Kuhn, Hewitt derived a theoretical formability diagram for non-work-hardening, rate sensitive materials⁸⁴. The model used in this analysis was very similar to that of Lee and Kuhn, the difference being the change from a work-hardening, non-rate-sensitive material to a non-work-hardening, rate sensitive one. Then the rate of thinning of the inhomogeneity relative to the surrounding material is governed by the strain (or strain rate) ratio and f_0 as before and the strain-rate sensitivity index rather than the work-hardening exponent. It was found that the strains to fracture increase with increasing strain-rate sensitivity and the fracture loci predicted from this model of localized thinning appear to be quite reasonable.

It was pointed out by Erturk² that two types of structural damage may occur during large compressive deformation of fiber-reinforced metal matrix composites: (i) void formation at fiber-matrix interfaces; (ii) fiber breaking resulting in bulging in the direction of fiber reinforcement. The former was shown to be related to tensile stresses in the horizontal poles at the matrix-fiber interface and that lateral pressure q tends to prevent this void formation. It was further shown^{72,85} that this tensile stress is proportional to the applied compressive stress. It was hypothesized, therefore, that a critical lower limit of the pressure ratio q/p is required to prevent the formation of voids⁶⁴.

The second form of failure is related to the transverse and vertical pressures in the composite and occurs when the shear stress in the fiber exceeds a critical value in excess of its shear strength.

Metallographic observations combined with plasticity analysis were used to define the limiting value of q/p required to prevent void formation. The variation of q/p with x for a range of values of $\mu L/h$ were determined from the slab method of plasticity analysis, where x is the lateral distance measured from the center of the rectangular specimen, L is the length of specimen, h is the specimen height, and μ is the friction coefficient. The values of $2x/L$ at which the innermost void appeared, and the corresponding value of $\mu L/h$ for each height reduction of the deformed specimens were used to define the limiting value of q/p required to prevent void formation as shown in Fig. (17). The shear stress in the fiber in the direction of reinforcement at the initiation of wedge formation was calculated. Assuming the fiber shear strength as one-half its tensile strength, it was found that the shear stress in the fiber exceeded the fiber shear strength at the initiation of fiber breakage, wedge formation, and the attendant deviation from plane strain. From this result and supporting metallurgical observations, it was concluded that the transverse failure was caused by shear at a critical q/p ratio near 1.0.

The few studies on the forging of metal matrix composites, reviewed in the previous sections, have dealt with relatively simple matrix alloys. For example there has been no work reported on the forging of composites possessing superplastic matrices, and indeed there has been very little work on the high temperature flow or fracture behaviour of metal matrix composites under simulated forging conditions. In view of the growing interest in superalloy/tungsten fiber composites and the unusual flow properties of these superplastic matrix alloys it is surprising that so little appears to be known. The purpose of the experimental portion of this thesis was to examine the flow and fracture behaviour

of these systems in order to improve the fundamental understanding of metal matrix composites and to contribute to the development of advanced fabrication processes.

3 EXPERIMENTAL APPARATUS

3.1 EQUIPMENT DEVELOPMENT

At the start of this program a high temperature compression testing apparatus developed at NAE^{86,87} was available for high temperature deformation studies. This apparatus was built around a servo-controlled hydraulic testing machine*, which had been modified for constant true strain rate compression. The existing facility allowed compression tests to be carried out under controlled atmospheres at temperatures up to 1150°C, and at strain rates in the range from 10^{-5} to 1 s^{-1} . References 86, 87 provide a good description of the system.

3.1.1 Limitations of the First System

Shortly after the start of this program a number of shortcomings in the existing system became evident. In particular, the possibility of plastic flow of the supporting tooling for the compression anvils limited the maximum working loads to about 50 KN at 1100°C. In order to deform composites of a reasonable size, it was clear that it would be necessary to increase this load capacity, if possible to the rated capability of the load frame (100 KN). Furthermore, the available apparatus was incapable of providing the temperatures contemplated for the proposed study of up to 1250°C. It was therefore decided to upgrade the existing system, and the subsequent burnout of the furnace made this endeavour even more urgent.

Modifications were made to the furnace and to the tooling. The Marshall furnace was rewound with Strandel Platinum — Platinum 40% Rhodium wire and its bore was increased from 76 mm to 85 mm in order to accommodate larger anvils.

* MTS 810 Material Test System, 10,000 Kg frame capacity.

Because of this, it became necessary to modify the end fittings of the furnace. The retort tube which provided close environmental control was removed and two 304 stainless steel plates and flanges were machined to fit the top and bottom anvil supports thereby sealing both ends of the furnace. To power the modified furnace, a new power supply and control system was installed*.

To increase the load capacity of the anvils, it was suggested that the hot portions be made from fiber reinforced Mar M200, which could be fabricated by hot isostatic pressing of a composite lay-up of tungsten reinforcing fibers in a superalloy powder material. Fabrication of these anvils involved the following steps:

1. A composite lay-up was prepared, consisting of alternate layers of superalloy powder deposited on a polycarbonate adhesive film and carefully spaced reinforcing wires of hard drawn thoriated tungsten wire.
2. The lay-up was encapsulated in a protective steel envelope which was evacuated and sealed.
3. The sealed container contents were hot isostatically pressed at 1150°C/100 MPa/2 hr to produce consolidated composite billets.
4. The protective container was then removed by machining and the cylinders machined into the final configuration.
5. Inconel 601 rings and sleeves were slip fitted over the ends of the anvils. These rings and sleeves, intended to provide locations for the required threads and grooves, were then diffusion bonded** to the anvils. A photograph of the as HIPed composite anvil is shown in Fig. (18).

* Eurotherm Comptroller Model 917/SCR Unit/Powerstat.

** Task done at the turbine and generator division of Westinghouse Canada Limited.

3.1.2 Limitations of the Revised Apparatus

In spite of the additional temperature capability of the Marshall furnace from 1300°C to 1500°C, the furnace was still incapable of providing the required temperature. This was attributed to the excessive heat losses through the end fittings of the modified tooling and through the furnace walls. Addition of extra insulation in these areas provided some improvements but the furnace element life was still expected to be drastically shortened due to the high current overloading required to maintain the higher temperatures. Finally, severe oxidation problems of the tooling and specimens occurred due to difficulty in maintaining a positive pressure of inert gas within the test chamber. It was concluded that major modifications would have to be made and therefore a completely new system was designed to meet the higher range of working temperatures required.

3.2 MODIFIED EQUIPMENT

The fundamental change in the new system was the use of induction heating as opposed to resistance heating which had been used with the earlier systems. This was expected to offer advantages of higher temperature capability, faster heating rates, flexibility in system design and longer life. Also, modifications were made to increase the loading capability of the compression members. As before, an environmental chamber was provided for good atmosphere control and oxidation protection of the highly reactive die materials, but because of the change in method of specimen heating the design of these parts was drastically different to those used previously. Fig. (19) shows the general layout of the modified testing system and Fig. (20) represents a schematic cross-section of the compression train. In the following sections a brief description of the major elements of the system is presented and some design and operating aspects of the apparatus are discussed.

3.2.1 Heating and Temperature Control System

The major elements of the heating system are: the power supply*, induction coil and the susceptor. As illustrated in Fig. (20), the induction coil (24) was formed from 6.35 mm diameter copper tubing. Different combinations of coil diameter and number of turns were examined in order to obtain the required temperature and temperature distribution. It was possible to obtain a specimen temperature of 1250°C using a load coil having 7 turns and a diameter of 95.25 mm. The coil spacing was about 6 mm. The coil was connected to the induction generator by means of extension leads (13) which passed through an induction port designed to provide vacuum sealing. The extension leads were designed to allow the induction coil to be changed easily. The coils were water cooled and were insulated using a layer of an insulating varnish** which was painted on.

Referring to Fig. (20), the susceptor (26) was made of either 304 stainless steel or graphite*** tube having an internal diameter of 76.2 mm, a 12.7 mm thickness and a 114 mm length. The reason for using a susceptor was to increase heating efficiency and to provide a uniform temperature zone around the work piece. Several layers of ceramic fiber sheet**** were wrapped around the susceptor to insulate it from the coil. The susceptor was held in place by two asbestos discs, 178 mm diameter and 12.7 mm thick, part (10). Two ceramic***** insulating blocks, 178 mm diameter and 44.5 mm thick, part (8), were added to provide more thermal insulation. To minimize radio-frequency effects on the surrounding instruments, a cylindrical copper shield, part (9), was made from 1.5 mm plate, and placed around the load coil assembly.

* Lepel induction heating generator type T-7.5-3-KC-J-SW (460 V) with SCR power control.

** Glyptol trade name.

*** Supplied by Speer Carbon Company.

**** Kaowool ceramic fiber.

***** 310 ceramic foam.

Since temperature is a very important parameter in the present investigation, ideally the temperature of the compression sample should be measured and controlled during the test. However, since the specimens are rather small in comparison to the mass of the tooling, and since they must be inserted and removed from the compression tooling freely, it was more practical to measure the temperature of the compression tooling and to control the temperature either at the tooling or susceptor. Specimen temperature was deduced from calibration curves obtained by direct measurements (see Section 4.5).

Temperatures were measured using Chromel-Alumel thermocouples with EMF output read directly as temperature using digital indicators. Fig. (21) shows a schematic diagram of the temperature control system. Output from a control thermocouple is supplied to a Honeywell "Dialatrol" three-mode proportioning controller (R7355), which in turn supplies a 4-20 ma D.C. output current to the power control unit of the Lepel induction generator. The D.C. signal from the temperature controller is proportional to the difference between the measured and set-point temperatures. RF power to the heating coil is controlled by a primary saturable core reactor which provides the means of controlling the A.C. input to the primary of the Lepel plate transformer. The plate transformer steps up the line voltage to high voltage. The latter is then fed to a diode rectifier bank which converts the high voltage A.C. to high voltage D.C., which in turn is converted to high frequency by means of the Lepel high frequency oscillator.

3.2.2 Compression Tooling

Materials for the compression dies must understandably be stronger than the metal being forged. TZM grade molybdenum was thought to be a suitable candidate because of its relatively high strength at elevated temperatures (310 MPa yield strength at 1300°C)⁸⁸, and its excellent thermal conductivity which allows isothermal conditions to be obtained easily. Another feature of the TZM is its excellent machinability

compared with other high temperature, high strength materials such as ceramics. The top die (working area = 14 cm^2 and 1.5 cm thickness), which had the form of a truncated cone, was held in contact with the upper anvil by means of a 713 LC superalloy collar. No collar was necessary to hold the bottom die, and it was clamped to the lower anvil via a locating-side pin. The bottom die was 63.5 mm diameter by 15 mm thickness. In order to minimize distortion of the loading columns under high stresses at high temperatures, the top and bottom anvils were made from a nickel-base superalloy (Udimet 700) while heat resistant 347 stainless steel was used for the anvil support. These compound loading columns were attached to the load cell and to the actuator ram of the load frame.

The top and bottom anvil supports were water cooled at the ends in order to protect the load cell and the actuator ram from heat conducted through the compound loading columns. Also, it must be mentioned that the upper column, supported by the crosshead, remains stationary while the lower ram supported by the actuator ram, was used to compress the specimens against the upper anvil.

3.2.3 Environmental Chamber

Since the TZM molybdenum tooling and any exposed tungsten fibers in the composites will oxidize rapidly in air at elevated temperatures, a cylindrical chamber was built around the compression tooling and heating coil. This chamber was vacuum sealed at the top and bottom portions, by means of O-ring assemblies, to facilitate evacuation. The environmental chamber consisted of two parts, a compression chamber and a cooling chamber. The compression chamber was made from a commercial steel tube (part (23) in Fig. (20)) which was 270 mm long, 250 mm internal diameter, and which had a wall thickness of 6 mm. Again referring to Fig. (20), a steel top flange (6) and bottom plate (17) made from 347 stainless steel were welded to the top and bottom of the steel tube respectively. The top plate (5) and cooling chamber (29)

were bolted to the compression chamber and vacuum sealed by means of O-rings. The purpose of the cooling chamber was to allow the lower TZM die to cool down to a temperature where rapid oxidation would not occur ($\sim 500^{\circ}\text{C}$), prior to opening for specimen replacement. This was machined from 304 stainless steel block and extended by means of a tube, from the same material, which was welded to the bottom of the block. This was to provide enough space to allow the bottom die and anvil to be held, under vacuum or inert gas, during the cooling period. Grooves were machined in the block and provided with circulating cooling water to accelerate the cooling process.

The compression and cooling chambers were isolated by a 347 stainless steel vacuum tight door which was actuated and supported by means of two swivel shafts* mounted on the bottom plate of the cooling chamber as shown in Fig. (20). The detailed arrangements of the components are shown in Fig. (22). To protect the O-ring on the vacuum door, a water cooling coil was soldered onto the top side of the bottom plate of the compression chamber, facing the O-ring. To remove or insert specimens, the bottom anvil was lowered below the vacuum door. The latter was then closed and clamped, and the cooling chamber was flushed with argon to cool the bottom die to about 400°C . Between 10 to 15 minutes were required to cool the bottom die to this temperature. The cooling chamber was subsequently opened to exchange the specimen. This process was reversed in order to transfer the specimen to the compression zone.

To avoid damaging the system by moving the piston while the vacuum door was closed, a safety micro-switch was provided to interlock the hydraulic power and prevent the piston from moving up while the vacuum door was not fully open. This was achieved by clamping the micro-switch onto an aluminum rod which in turn was

* Edwards rotary shaft vacuum seal (6.35 mm diameter).

clamped to the piston as shown in Fig. (22). A slot was machined in the rotating handle of the vacuum door, so that it faced the micro-switch when the door was open. If the vacuum door was fully or partially closed, the micro-switch would meet the bottom face of the handle and turn off the hydraulics. The micro-switch was adjusted to open the hydraulic circuit when the bottom die was 25 mm below the vacuum door. This was to allow for the height of the specimen and to prevent squeezing the specimen against the vacuum door.

3.2.4 Atmosphere Control

To prevent oxidation of the tooling and samples, it was necessary to run the tests under a flowing argon atmosphere. Although the present equipment can operate under vacuum, this causes much more friction between the O-ring seal and the upper ram, which is transmitted to the load cell. This introduces errors in load measurement, which are particularly important at high temperatures and low strain rates where the flow stresses of the specimens are very low. It was therefore preferable to run the tests under a positive pressure of inert gas*, with the vacuum seal partially released to eliminate the friction.

The environmental chamber was evacuated by means of a two stage rotary pump (4.5 m³/hr)** capable of holding the chamber pressure to $\sim 10^{-2}$ torr. Provision has been made to allow either of the chambers to be evacuated independently or both simultaneously. Prior to heating up, the chambers were repeatedly evacuated and flushed with inert gas to ensure that traces of air were removed. During the test, a positive pressure of argon was maintained within the chamber by bubbling the escaping

* Argon supplied by Matheson of Canada Ltd.

** Edwards rotary vacuum pump, Model ED 75.

gas through an oil trap. The gas flow rate was controlled by means of a needle valve*.

A schematic diagram of the gas supply and vacuum system is shown in Fig. (23).

3.2.5 Strain Measurements

Elastic distortion of both the testing frame and the tooling can introduce substantial error in the strain measurements if the specimen height is to be monitored by means of the MTS stroke transducer**. For this reason the strain measurements were made between the dies themselves. This was achieved by using a high temperature displacement transducer to monitor the relative displacement of the compression dies.

The device consists of a set of ceramic probes which translate the motion of the dies to the body and core of a DCDT*** displacement transducer located outside the environmental chamber. The device is shown in Fig. (24). Two micrometer screws independently connect the body and core of the DCDT for adjustment and calibration purposes.

The DCDT assembly was contained in a perspex tube 230 mm long by 70 mm internal diameter, with a wall thickness of 5 mm. The tube was clamped onto the top plate of the environmental chamber and a seal was provided by an O-ring. The top plate of the DCDT chamber was machined out of aluminum and clamped to the perspex tube.

Again, a seal was provided by an O-ring. The two micrometers were actuated via two rotary shafts**** mounted in the top plate of the DCDT vacuum chamber as shown in Fig. (24). The output signal of this DCDT was fed back to the MTS console for recording purposes.

* Edwards Vacuum Comp. Model OSID.

** LVDT, linear variable differential transformer.

*** Differential transformer with built-in carrier oscillator and phase sensitive demodulator.

**** Edwards rotary shafts (vacuum seal 3.175 mm diameter).

3.2.6 Strain Rate Control

Because of the high strain rate sensitivity of metals and alloys at high temperatures, it was essential that the strain rate be maintained constant during forging simulations in order to evaluate, and separate unambiguously, the effects of the experimental variables on high temperature plastic flow.

To achieve a constant true strain rate during testing requires that the ram velocity of the testing machine remains directly proportional to the height of the specimen. The basis of this proportionality is explained in Appendix II.

Modern, closed loop, servo-controlled hydraulic machines, like the one under consideration, are generally provided with a program input which, given the proper function generator, can be used to drive the ram, under displacement control so as to satisfy the requirements of this proportionality. In the present work this was achieved by means of an analog function generator designed to operate in conjunction with the MTS controller. A block diagram of the ram position control, incorporating the function generator is presented in Fig. (25).

The analog function generator consists basically of an RC decay circuit with additional circuitry to increase the device flexibility during testing. The generator is powered by a reference stabilized DC voltage supply. The output can be set anywhere from 0 to 10 VDC by means of the "Voltage adjust" potentiometer. Ten different strain rates can be selected by means of a rotary switch. Each position of the switch corresponds to a different resistance value for the RC circuit, the capacitive element being kept constant ($10.8 \mu\text{f}$). The resistance values have been calculated to cover the required range of strain rates and are listed in Appendix III.

A voltage limit detector is used to control the generator output and in turn the amount of imposed deformation. The trip voltage can be adjusted to any value within the output range of the function generator, by means of a "trip adjust" potentiometer.

Once triggered, the limit detector circuit must be reset for further use by means of a "reset" push button. Further details of the analog function generator can be found in Reference 87.

The output characteristics of the DC displacement transducer, described in 3.2.5, were matched to those of the MTS LVDT stroke transducer, located at the base of the actuator ram. This was achieved as follows: the actuator piston was held at mid-stroke (zero volts), with the LVDT in the feedback loop, and the position of the DCDT core was adjusted by means of the micrometer screw for zero volts output. The attenuation level was subsequently adjusted, by means of an attenuator circuit designed for this purpose⁸⁷, so that a 10 v high level signal was supplied to the feedback selector when the ram was made to retract full scale. The retraction obviously depends on the particular stroke range used. The transfer from one transducer to the other in the feedback loop could then be carried out straightforwardly and the position of the ram along its stroke was not affected in the process.

Ideally, the output of the DCDT should be used for control purposes to achieve the required constant true strain rates. However, in practice, the MTS LVDT stroke transducer has been used for simplicity. The errors introduced by doing this, should not lead to significant errors in strain rate⁸⁷.

3.2.7 Performance of the Apparatus & Modifications

Prior to heating up, the compression chamber was evacuated and then flushed with argon. A low pressure of 2×10^{-2} torr was obtained in about 15 minutes. Alternate evacuation and flushing with argon was performed as explained in Section 3.2.4.

The start up procedure of the induction generator was performed with the mode switches set to "manual". A 304 stainless steel susceptor and 7 turns of 6.35 mm copper tubing forming the load coil were used initially. The tank coil link was tentatively located to a tap near the middle of the coil. Grid control was set to "30", and

power control to zero. RF power was applied after a time delay period of 5 to 30 minutes depending on the prior shut down period of the generator. This time delay period was to allow the different components of the generator to warm up prior to power application. At these settings, plate and grid meter readings were very low. The power control was advanced to 30% while grid and plate currents were adjusted to read within the specified limits by the manufacturer, namely between 0.4 to 0.6 AMPS and less than 1.95 AMPS for the grid and plate ammeters respectively. Temperature increases of about 100°C/minute were observed. However, as the temperature approached about 900°C instability occurred in the stroke of the MTS piston, causing the overload interrupts to operate and hydraulic power to shut down. Furthermore, all digital indicators and electronic units became unstable in the same time. This procedure was repeated twice and the same behaviour occurred, meanwhile extra-ordinary RF signals were detected by means of an RF detector placed close to the machine.

The experiment was repeated again, under identical conditions, as mentioned above but with vacuum instead of the argon atmosphere. When the temperature reached about 600°C, a discharge glow was observed through the Plexiglass tube of the DCDT chamber and the vacuum level dropped from 2×10^{-2} torr to about 2 torr. This discharge glow was due to ionization of the argon gas, which apparently may occur when the load coil is operated within a low pressure atmosphere⁸⁹. However, the reason for the loss of vacuum associated with the ionization process was not clear. At this point the system was opened and the different elements inside the environmental chamber were observed during the heat-up process. Arcing was observed to take place between the high potential RF lead and the inside wall of the RF port adjacent to the brass clamping nut, part 14 Fig. (20). Also, it was found that the O-ring of the RF flange (part 12 Fig. (20)) was partially burned out as a result of arcing, and this caused

the drop of the vacuum level. It must be mentioned that the clearance between the brass nut and the wall of the RF port was relatively small, about 1.5 mm.

It was apparent that any action taken to reduce the potential across the coil would help avoid ionization. For this reason, the number of turns on the load coil was reduced to 4 turns. Also, the brass clamping nut was replaced by another, machined from bakelite. Furthermore, the load coil and the RF leads were coated with an insulating varnish. These steps were sufficient to eliminate the ionization problem and the associated instability of the different electronic units. However, slight RF effects on the temperature indicators remained to be eliminated. Accordingly, all different leads were shielded* and properly grounded. Also, capacitors were connected between the negative lead of each temperature indicator and the ground to by-pass any induced RF.

A steady state temperature was reached using the stainless steel susceptor in about 45 minutes as shown in Fig. (26). The temperature difference between the susceptor and the specimen was about 120°C . To reach the contemplated specimen temperature of up to 1250°C , it would be necessary to keep the susceptor at temperatures close to 1400°C which might cause melting of the stainless steel. To avoid this problem, another susceptor, 76 mm I.D., 3.2 mm thickness and 100 mm long, was machined out of graphite tubing. More efficient heating and faster heating rates were obtained by using the graphite susceptor as shown in Fig. (26).

As the experiments proceeded, it became evident that at the highest temperatures contemplated, the DCDT environmental chamber would see temperatures of about 100°C . Since the maximum working temperature of the DCDT was limited to 65°C , it was necessary to keep the temperature below this limit. Accordingly, a special cooling system was constructed from 1.6 mm copper sheet with cooling coils

* Using braided tubing.

brazed onto it. The cooling device was positioned around the vulnerable portion of the DCDT assembly in the compression chamber. By this approach it was possible to keep the temperature of the DCDT within the specified limits.

3.3 DATA RECORDING AND ACQUISITION SYSTEM

The two important parameters to be recorded during a compression test were the developed load and the specimen height. It was therefore decided to monitor the voltage outputs of the load cell, and the DCDT which indicates the relative movement of the compression dies. The load cell output was recorded on one channel of a x-y chart recorder, while the DCDT output was recorded simultaneously on the second channel of the same recorder. At the same time the analog signals, corresponding to load and strain, were digitized and stored on magnetic tape by means of a real-time data acquisition system available at NAE*^{90,91}. Fig. (27) reproduced from Reference 91 represents a flow chart for this system. The system allowed one to vary the time interval between successive recordings and therefore the most sensible recording frequency for a given strain rate could be selected. A recording frequency of 0.4 to 1000 samples/second was available, however, for the fastest strain rates a sampling rate of 80 samples/second was used while 0.4 samples/second was used for the lowest strain rates.

To co-ordinate the beginning of the test to the start of data acquisition a special electronic circuit was designed for "automatic start/stop" that would generate the required pulses for the remote data-acquisition system's (RDAS) start/stop function⁹⁰. At the beginning of the compression test a "RUN" switch was activated⁸⁷ and the test was completed when the "TRIP" circuit was entered. Further details on RADA's remote start/stop function are given in Reference 90.

* National Aeronautical Establishment.

At the completion of the experiments the stored data was then edited and transferred to a master tape. The re-formatted data, in the form of IBM cards, was then transmitted to the NRC computation center's IBM 360/67 system for analysis. Each test was appropriately identified to facilitate individual test retrieval for data analysis purposes. The data retrieval operations and all subsequent calculations and plotting operations were monitored by a computer program designed at NAE for these purposes*. From the full scale voltage ranges for the load and displacement signals, it was a simple matter to retrieve the measured uniaxial force "F" and the measured instantaneous height h. The true stress and the true strain during compression were calculated from the uniaxial force, the instantaneous specimen height, the initial height h_0 and the initial cross sectional area A_0 of the specimen assuming that no volume change takes place during deformation. The true stress is given by:

$$\sigma = F/A \quad (11)$$

where, A is the instantaneous cross-sectional area of the compression specimen calculated from the relation:

$$A = A_0 h_0 / h \quad (12)$$

On the other hand, the true strain was derived from the axial contraction of the specimen from the relation:

$$\epsilon = - \ln[h_0/h] \quad (13)$$

* Designed by Dr. J-P. Immarigeon, NAE, NRC.

4 EXPERIMENTAL MATERIALS AND PROCEDURE

4.1 EXPERIMENTAL MATERIALS

4.1.1 Compacts

The alloy considered was Mar M200, a high strength casting-type nickel-base superalloy ($\sim 60\%$ Ni) which relies on γ' ($\text{Ni}_3\text{Al,Ti}$) precipitation hardening and solution hardening with tungsten. This is the alloy originally selected by Westinghouse Canada Limited for the development of the fiber reinforced inlet guide vanes³.

The master alloy was argon atomized. The powder chemistry and mesh size distribution are given in Tables 1 and 2.

Three mesh sizes were considered: A(+140), B(-80 to -500) and C(140/170).

A represents the coarse fraction of the powder

B represents the fine fraction of the powder

C represents a medium mesh size

The compacts were prepared by hot isostatically pressing the selected powders in evacuated and sealed stainless steel tubes, 12.7 mm in diameter by 460 mm long.

Since fine grain size is an essential requirement for superplasticity the present work will deal primarily with compacts pressed below the γ' solvus temperature ($1200^\circ\text{C} \pm 15^\circ\text{C}$)¹³. Typical pressing conditions of $1050^\circ\text{C}/70\text{ MPa}/2\text{ hrs}$ were used and the samples were furnace cooled to approximately 800°C from the HIP temperature and then air cooled to room temperature.

4.1.2 Composites

The matrix material selected for the composites was Mar M200 with a predominantly fine mesh size (group B). Analysis indicates that more than 80% of the

powder consists of -140 mesh size (Table 2). Commercial and developmental grades of thoriated tungsten wire* (0.5 mm diameter) were selected as composite fibers. It has been shown³ that a diffusion barrier coating on the fibers is necessary to prevent a loss of strength as a result of nickel-induced recrystallization of the tungsten above 1000°C. Continuous chemical vapour deposition (CVD) is used to coat the fibers with a 3-4 micron thick coating of hafnium nitride³.

Composite fabrication involves a large number of operations, some of which are proprietary. The reader is referred to the literature³ and to Appendix IV for a detailed description of the procedure. For the present experimental work, rectangular bars with a transverse cross-sectional area slightly greater than 15 by 10 mm were used. These were prepared as 200 mm long bars** following a procedure similar to that used for composite air foil shapes³.

Originally, HIPing conditions of 1050°C/103 MPa/2 hr were used. Consolidation was not complete and sound bonds between the tungsten fibers and the matrix were not achieved. Consequently, the pressing temperature was raised to 1150°C resulting in full consolidation. After HIPing the container was removed by machining and the billet ground down to parallel faces. Square specimens were subsequently cut from the bar.

4.2 SPECIMEN PREPARATION

4.2.1 Compacts

Cylindrical specimens with a height to diameter ratio of approximately 1.5 were machined from the matrix material to be investigated.

* Produced by the Lamp Division of Westinghouse Electric Corporation
Pittsburg, U.S.A.

** Task performed by Westinghouse Canada Limited.

It is usually supposed that if a cylindrical block of metal, whose height is less than about 1.5 times its diameter, is plastically pressed between rigid, parallel, frictionless dies, that it will deform homogeneously⁹². The end faces of the cylinders were grooved in order to retain the high temperature lubricant used in these tests. Specimen dimensions and configuration are shown in Fig. (28). This technique is now well accepted and appears to be quite successful in limiting frictional effects. The geometry of these grooves was based on past experience⁹³ which indicated that flat bottomed, closely spaced wide grooves gave the best results. With such a technique it would be possible to attain a strain of 0.7 without barrelling and of about 1.2 with slight barrelling⁴³.

4.2.2 Composites

After removal of the protective container by milling, the reinforced billets were ground down to parallel faces using a surface grinding machine which used a coolant. Different cutting techniques were examined to minimize mechanical damage to the fibers and matrix during the cutting process. Originally, spark machining was used, but this was abandoned because of the poor surface finish produced and the slow cutting rates involved. A low speed diamond impregnated saw* has been used, with a cutting blade 1.6 by 76 mm diameter operating at about 200 RPM. In spite of the good surface finish produced by this method, the very slow cutting rates involved (about 30 hours to cut a 10 by 15 mm cross section) and the need for successive dressing of the blade made this method impractical. However, one specimen was obtained by this method for comparison with other cutting techniques. Diamond-dressed cutting wheels** (1.6 by 229 mm diameter and 3450 RPM) have been used without any apparent damage to the specimen.

* Isomet Low Speed Saw 11-1180, Buehler Ltd.

** 1010 Cutt-off Machine, Buehler Ltd.

However, metallographic examination of the cut faces revealed a few internal cracks in the fibers. Subsequent grinding did not improve the situation suggesting that these defects might have been introduced prior to or during the compaction stage. The cut surfaces obtained by the last two methods have been compared and no significant differences were observed. Therefore, the diamond-dressed cutting wheel was used throughout this work, and the cut faces finished by surface grinding.

For the bulk of this work square compression blanks of 10 by 10 mm and 15 mm height were produced, as shown in Fig. (29). In contrast to the cylindrical compact specimens, the end faces of the composite specimens were not grooved for lubricant retention.

4.3 HIGH TEMPERATURE LUBRICANTS

In order to promote homogeneous flow, lubrication must be provided during compression so as to keep friction between the specimen faces and anvils as low as possible. At high temperatures, powdered glass lubricants have been used successfully^{13,93,94,95} to decrease the effect of friction and barrelling.

Proper lubrication has been shown⁹³ to occur when the glass is fluid at the testing temperature but sufficiently viscous so that it does not flow too easily and is, therefore, retained within the specimen grooves. In the present work, glass lubricants* in the form of fine powders (~200 mesh) have been used. A suspension of the selected powder in acetone was applied to the specimen using a fine brush. A compacted glass powder cake thus forms at both ends of the specimen. During compression, the viscous lubricant gets trapped in the grooves and is gradually forced out by hydrostatic pressure as the ridges of the grooves collapse. In this way fresh lubricant is continuously force fed between the dies and the grooved specimen during compression.

* Sodium/calcium borosilicates, with varying amounts of magnesia and alumina — Supplied by Ferro-Industrial Products, Oakville, Ontario, Canada.

It was also standard practice to coat the sides of the specimen to prevent oxidation. Lubrication breakdown occurs at large strains for the grooved matrix specimens (above $\sim 70\%$) and at relatively lower strains for the flat (not grooved) composite specimens, so that barrelling and edge overlapping tend to become more evident.

4.4 METALLOGRAPHY AND CHARACTERIZATION OF THE As-HIPed MATERIALS

4.4.1 Compacts

Sections from the as-HIPed Mar M200 Compacts were mounted and polished for metallographic examination. The following polishing procedure was used.

Initial grinding was carried out on 240 grit wet silicon carbide papers followed by successively finer grinding on 320, 400 and 600 grit wet silicon carbide papers, the specimens being ultrasonically cleaned between grits. Final polishing was carried out on diamond wheels using 9μ , 3μ and 1μ diamond paste successively, again cleaning ultrasonically between pastes. Two etching solutions were used, one to reveal γ morphology and distribution (Inco etch*) and the other to reveal grain boundaries (Marble's reagent**). Examinations were carried out by means of optical and electron microscopy. One specimen from each of the three mesh sizes was examined. Also, the microhardness of each of these materials was determined by means of a hardness tester***. Five different readings were performed and the average value determined for each specimen****.

4.4.2 Composites

The purpose of the metallographic examinations of the composites was to determine the nature, extent, and distribution of any voids or damage in the composite in the as-HIPed condition prior to forging. One specimen from each as-HIPed composite

* Inco etch: 50 ml HCL, 25 ml HNO₃, 2 gm CuCl₂, 200 ml H₂O.

** Marble's reagent: 10 gm CuSO₄, 50 ml HCL, 50 ml H₂O.

*** MINILOAD hardness tester, Leitz Wetzlar, Germany.

**** All microhardness tests were carried out at room temperature.

bar was sliced normal to the direction of fiber alignment and ground as described in Section 4.2.2. The ground specimens were mounted and polished following a technique similar to that outlined in the previous section. However, greater care and longer times were necessary to polish the composites due to the difference in hardness, and hence the polishing rates, of the matrix and the tungsten fibers. Murakami's reagent* was used to reveal the microstructure of the fibers while Inco etch or Marbles reagent were used for the matrix of the composite. Specimens were covered partially with stop-off lacquer to facilitate applying the different etching solutions to the same specimen.

To obtain further indications of any differences in the mechanical properties of the reinforced matrix and that of the matrix without reinforcement, microhardness measurements were performed on the two materials using the MINILOAD hardness tester. Five measurements were taken for each sample and the average was determined.

Cracks have been observed in the reinforcing tungsten wires of the composites in the as-HIPed condition. Therefore detailed metallographic examinations of the composites were undertaken to determine the origin of the cracks. This was performed using an electron microprobe analyzer**.

4.5 TEMPERATURE CALIBRATION

The temperature of the compression specimen was calibrated using a Chromel-Alumel type (K) thermocouple which was bent and embedded into a dummy specimen placed between the compression dies. The bent thermocouple was checked against a reference thermocouple (type (S)) and found to be accurate to within $\pm 1^\circ\text{C}$. The calibrations of the set point, displayed chamber temperature*** and specimen temperature**** were performed and the results are presented in Fig. (30) for

* Murakami's reagent: 10 g $\text{K}_3\text{Fe}(\text{CN})_6$, 10 g KOH or KaOH, 100 ml water.

** SEM 2A, AEI Ltd., England.

*** Intertechnology model 400A digital temperature indicator (Type K thermocouple).

**** Displayed on a Thermo Electric digital temperature indicator (Type S thermocouple).

the original system and Fig. (31) for the modified equipment. The graph allows one to select the appropriate set point for a required specimen temperature in the range from 800°C to 1200°C. The reproducibility of the specimen temperature, prior to a test, was within $\pm 2^\circ\text{C}$. During a test the stability was within 2 to 3°C.

4.6 TESTING PROCEDURE

Prior to a series of tests, with the induction generator set for the external mode of operation, the Honeywell temperature controller was adjusted for the desired temperature and the furnace brought up to temperature. It generally took 15 to 20 minutes for the temperature to stabilize at the set point.

The output of the LVDT and DCDT transducers were matched as explained in Section 3.2.6. Once the output of the transducers was matched, very little if any adjustment was needed between successive tests. Only when the temperature was changed and a new series of tests started was a totally new adjustment necessary.

To remove or insert specimens, the bottom anvil was lowered below the vacuum door which was then closed and clamped. The bottom die was allowed to cool under argon to about 400°C. This operation took about 15 minutes. The cooling chamber was subsequently flushed with argon, and the furnace was raised by raising the MTS cross head, while the bottom anvil was lowered simultaneously. The glass-coated sample was inserted on top of the bottom die and the system was immediately closed. The cooling chamber was then evacuated and the process was reversed in order to transfer the specimen to the compression zone. A temperature drop within the compression chamber of about 100°C occurred as a result of the above operation. It generally took 5 to 10 minutes for the temperature to stabilize again at the set point. During this time the various operations required to initiate the real-time data acquisition system were carried out.

The appropriate load and stroke ranges were selected on the MTS controller according to the cross section and the testing condition of each specimen. For all compacts a stroke range of ± 15 mm was adequate, while a range of ± 35 mm was used for the larger size composites. The full scale voltage ranges for the load and displacement measurements were adjusted on the x-y chart recorder. The various safety switches were set to prevent self-compression or damage of the testing train. The required strain rate was selected by adjusting the resistive value of the RC circuit and the amount of imposed deformation was adjusted by means of the "trip adjust", as explained in Section 3.2.6.

4.7 EXAMINATIONS OF STRUCTURAL CHANGES PRIOR TO LOAD APPLICATION

Since all test specimens were soaked at the testing temperature for about $\frac{1}{2}$ an hour to stabilize the temperature prior to the load application, and since the flow behaviour of the matrix material is a strong function of the microstructure, it was necessary to examine the effect of the soaking period on the microstructure of the material. For this purpose eight specimens 6.35 mm diameter by 5.0 mm high were machined from the as-HIPed matrix material. One specimen was heated for $\frac{1}{2}$ an hour at a temperature of 850°C and one of the remaining specimens was heated at one temperature in the range from 950°C to 1250°C at temperature intervals of 50°C . These specimens were quenched in oil at the end of the soaking period, mounted, polished and etched following the procedures described in Section 4.4.1. Plastic-carbon replicas were prepared following the standard procedures described in Reference 96 and the replicas were examined by means of a transmission electron microscope.

4.8 FLOW BEHAVIOUR OF MATRIX MATERIAL

Tests were carried out at temperature intervals of 50°C in the range from 950°C to 1200°C . At each temperature, 5 strain rates were used between

10^{-1} s^{-1} and 10^{-5} s^{-1} . The ranges of temperature and strain rate were selected to provide conditions where both conventional and superplastic flow should occur and encompass the kinds of temperature and strain rate involved in industrial isothermal forging operations. Tests above 1100°C were carried out using the induction heating compression train, while the lower temperature tests were carried out using the Marshall furnace. The most rapid tests for the Mar M200 compacts lasted approximately 5 seconds. At the lowest strain rates, however, the tests took about 2 hours to achieve 60% strain.

To examine the effects of grain size on the flow behaviour of the matrix material, a four inch rod of the as-HIPed matrix material was heated at 1230°C for 4 hours under a flow of argon and then furnace cooled to room temperature. Compression specimens were machined from the heat treated bar to the configuration shown in Fig. (28). Compression tests were carried out at six different strain rates and at 1150°C . The results were compared to those of the as-HIPed material tested under the same conditions of temperature and strain rate. Also, one specimen was prepared for metallographic examination.

It has been suggested that the peak flow stress of the material at different strain rates might be obtained with one single specimen by changing the strain rate during the test⁹⁷. The author's original thought was that substantial errors might be introduced as a result of the structural changes taking place during deformation and also due to the increasing frictional constraint at higher strains. However, this suggestion was examined experimentally as follows. The peak flow stresses at six different strain rates between 10^{-5} s^{-1} and 10^{-1} s^{-1} were obtained at 1150°C using individual tests i.e. one specimen for each strain rate. These tests were repeated at the same temperature using one single specimen and the strain rate was changed immediately beyond the peak. Again, the same test was repeated using one single

specimen but in this case the strain rate was shifted after about 15% to 20% deformation beyond the peak. The peak flow stresses were calculated for each case and compared.

4.9 MESH SIZE EFFECT

Test specimens were also machined from consolidated rods of the three mesh sizes, A, B and C, to the configuration shown in Fig. (28). These specimens were lubricated prior to testing following the procedures of Section 4.3. Tests were carried out at temperature of 1000°C. At each temperature, four strain rates were used between 10^{-2} s^{-1} and 10^{-4} s^{-1} .

4.10 EFFECT OF PRETRAINING (GATORIZING) ON THE FLOW BEHAVIOUR.

Two approaches were examined to explore the effect of pretraining (Gatorizing) on the flow behaviour of the matrix material.

4.10.1 Extrusion Prior to Forging

Extrusion work was carried out at ambient temperature using a hydrostatic extrusion apparatus, described elsewhere⁹⁸. This is capable of generating pressures up to 1600 MN/m² on billets up to ~38 mm diameter. Samples of Mar M200 were machined to the billet shape shown in Fig. (32), with diameters D chosen to give reduction ratios* of 5, 10, 15 and 20%. Extrusion was achieved using castor oil as the pressure medium and ROCOL** (R.T.D.) as a lubricant. Standard compression specimens as shown in Fig. (28) were machined from the extruded billets, and compression tests were carried out at strain rates of 10^{-1} , 10^{-2} and 10^{-3} s^{-1} and a temperature of 1100°C. One specimen from each extruded billet was mounted and polished to facilitate ductility evaluations and microhardness measurements across

$$* \quad \text{Reduction ratio} = \frac{A_o - A_f}{A_o}$$

** ROCOL Limited, England.

the section. These hardness readings provide an indication of the amount and distribution of strain energy induced in the billet by the different extrusion ratios. Also, one specimen of the as-HIPed material (without extrusion) was mounted and microhardness readings taken across the sample.

4.10.2 Primary Forging

In these experiments, specimens were prestrained to 60% deformation at strain rates of $8.8 \times 10^{-2} \text{ s}^{-1}$ or $2.1 \times 10^{-1} \text{ s}^{-1}$ then unloaded and restrained in situ at each one of the following strain rates; 10^{-2} , 1.4×10^{-3} , 3×10^{-4} and 10^{-4} s^{-1} . Each test was continued up to a true strain of ~ 1.2 . Another set of specimens was prestrained at a strain rate of $8.8 \times 10^{-2} \text{ s}^{-1}$, to increasing amounts of true strain ranging from 0.1 to 0.6 at which time the tests were interrupted and the strain rate immediately changed to $1.4 \times 10^{-3} \text{ s}^{-1}$. These tests were carried out at temperatures of 1100°C and 1150°C .

To examine the microstructural changes taking place during deformation, another series of specimens was deformed at 1100°C and 10^{-1} s^{-1} to increasing amounts of true strain ranging from 0.1 to 0.8 and the deformed specimens were quenched in oil within 1 to 2 seconds of the end of the test. These specimens were examined metallographically following the procedures described in Section 4.4.1.

4.11 FLOW BEHAVIOUR OF COMPOSITES

As with the matrix material, compression tests were carried out at temperature intervals of 50°C but in the range from 1050°C to 1200°C . This was to avoid the large stresses which might be developed at the lower range of temperatures. At each temperature, 5 strain rates were used between 10^{-1} s^{-1} and 10^{-5} s^{-1} . Again, tests above 1100°C were carried out using the induction heating system, while the lower temperature tests were carried out using the Marshall furnace.

To examine the effects of strain on the amount of damage introduced into the composite, four composite specimens were forged at 1100°C and $3 \times 10^{-4} \text{ s}^{-1}$, to strains of 3%, 6%, 10% and 30% respectively. These specimens were examined metallographically. Also, to examine the influence of strain rate and temperature on the strain induced damage, two specimens were forged at 1100°C and 1180°C to a true strain of 10% at $3 \times 10^{-4} \text{ s}^{-1}$, and two others were forged at 1100°C to a strain of 30% at $0.88 \times 10^{-1} \text{ s}^{-1}$ and $9.6 \times 10^{-5} \text{ s}^{-1}$. Again these specimens were examined metallographically.

4.12 EXPERIMENTAL ERRORS

Errors from several sources may affect the experimental stress/strain results obtained in this study. These can be broadly classified as errors due to imperfect performance of the apparatus and errors due to experimental procedures. Four types of error affect the operation of the experimental apparatus. These have been discussed elsewhere⁸⁷ for a system similar to that used in the present study. The first type of error arises from the drift in offset or gain of the MTS servo-amplifier. This is inherent in the MTS system and no consideration was given to altering the equipment to overcome this in the present study. The second source of error is associated with drift in any offset or gain of the operational amplifier of the function generator itself. The third arises from possible non-linearities in the output of the feedback stroke transducer, and the last from elastic-plastic interactions in the specimen.

The stability of the complete system was determined for various positions of the piston along its stroke and the results (reproduced from Reference 87) are presented in Table 3, together with errors in strain rate for the stroke range under consideration. It is evident from Table 3 that the errors involved increase rapidly with decreasing strain rate, and it was suggested that the use of the apparatus should be limited to strain rates higher than 10^{-4} s^{-1} .

It should be mentioned that the reproducibility of these strain rates is controlled by the temperature sensitivity of the resistance in the RC circuit. As given by the manufacturer's specification, a typical percent change in resistance due to temperature fluctuations in the range $30^\circ \pm 10^\circ\text{C}$ is of the order of 0.5%. This results in a relative error in strain rate of the same magnitude.

As mentioned previously, the fourth substantial source of error in strain rate arises from the interactions between the elastic strain and plastic strain in the specimen. It should be recalled that the aim of the present exercise is to try to keep the specimen plastic strain rate constant. However, it is the total strain rate $\dot{\epsilon}_T$ (to which both the elastic strain rate ($\dot{\epsilon}_e$) and the plastic strain rate ($\dot{\epsilon}_p$) contribute) which is in fact maintained constant*:

$$\dot{\epsilon}_T = \dot{\epsilon}_p + \dot{\epsilon}_e \quad (14)$$

It is shown in Appendix V that the difference between the total strain rate, that is the strain rate imposed by the function generator and the specimen plastic strain rate is a function of the elastic modulus of the specimen, of its height and of the rate of increase of developed load. The error in strain rate is shown to be:

$$\Delta\dot{\epsilon} \approx \frac{h}{EV} \frac{dL}{dt} \quad (15)$$

if it is assumed that no volume change takes place during deformation.

Clearly the rate of increase of developed load will depend on the temperature, the strain rate and the mechanical properties of the material under investigation. During high temperature deformation; the loading rate is initially maximum as the material deforms elastically and then gradually decreases with the onset of plastic flow. For some materials it reaches a minimum with steady state flow and for others it can

* The elastic distortion of the testing train is not a source of error as long as the displacement transducer in the feedback loop, is that which monitors the compression gap.

become very slightly negative if work softening occurs to a sufficient degree. Experiments with a superalloy (see Section 5.2) using the present apparatus have shown that the initial loading rates can be as high as 70 KN/s at the highest strain rate used*. For the range of strain rates investigated, this leads to a difference between the specimen plastic strain rate and the imposed strain rate of 4 to 10%. The error during flow at intermediate and high strains decreases and can vanish altogether when the loading rate goes to zero as is frequently observed.

In analysing a high temperature flow curve, the errors in the measured yield stress, peak flow stress or steady state stress are even smaller than the errors discussed above as they are equal to the errors in strain rate multiplied by the rate sensitivity of the material. This stems from the generally observed relation:

$$\sigma = k\dot{\epsilon}^m \quad (16)$$

where k is a constant at constant structure and m is of the order of 0.2 to 0.6 for the materials investigated so that:

$$\frac{d\sigma}{\sigma} = m \frac{d\dot{\epsilon}}{\dot{\epsilon}} \quad (17)$$

Thus, although the error in strain rate can be as high as 10% under rapid loading or as high as 5% due to DC instabilities at low strain rates, the actual relative error in the quantity of-relevance i.e., the peak flow stress is less than 3%.

The reproducibility of the flow curves was examined by forging four identical specimens of Mar M200 compacts, at 1100°C and $1.4 \times 10^{-3} \text{ s}^{-1}$. All the flow curves fall within the scatter band shown in Fig. (33). The maximum variation in the flow stress can be observed at the peak and equal to $\sim 3\%$. Therefore, the overall experimental error in flow stress, for the present study, is estimated to be less than 6%.

* 10^{-1} s^{-1} for a cylindrical specimen 10 mm high. At the lowest strain rate used, 10^{-4} s^{-1} , the initial loading rate drops to 20 N/s.

It should be noted with regard to the true stress — true strain calculations that the recorded loads include a contribution due to friction and barrelling, and therefore would be slightly larger than in the case of frictionless, homogeneous deformation. In the present investigation, however, friction and barrelling were minimized by the use of the molten glass lubricants as described in Chapter 3. Barrelling only became apparent at the higher strains and was almost absent during the first part of the compression cycle. Frictional effects were therefore judged to be negligible up to a strain of ~ 0.5 and the results presented in the following chapter were not corrected for friction.

Two other sources of error have been neglected. The first is the error in the displacement measurements due to the collapse of the grooves in the specimen end, faces and the extrusion of the molten glass lubricant at the specimen/anvil interface. And, the second is the temperature increase due to adiabatic compression at the higher strain rates.

5 EXPERIMENTAL RESULTS

5.1 CHARACTERIZATION OF THE As-HIPed MATERIALS

5.1.1 Compacts

Fully dense compacts were produced under the pressing conditions of 1050°C/ 2 hrs/69 MN/m² as shown in Fig. (34-a). Since the γ' solvus is 1200°C $\pm$ 15°C, the microstructure consists of the γ austenitic solid solution and coarse particles of the agglomerated γ' phase, as shown in Fig. (34-b), which is stable at the pressing temperature. The grain sizes and gamma-prime particle sizes present after pressing are in the range 2-8 μ m and 0.4-1.5 μ m respectively. The presence of the coarse γ' on grain boundaries during HIPing stabilizes the fine as-atomized grain size of the powders. Only minor changes were observed in the microstructure of the compacts with different mesh sizes. However, compacts produced from the fine fraction of the powder, group B, produced higher average microhardness readings compared to the compacts produced from the coarse and medium fractions of the powder (group A and C respectively). The microhardness readings were 432, 460 and 442 for the mesh sizes A, B and C respectively.

The effect of the soaking period prior to forging on the microstructure of the matrix material is shown in Fig. (35). These representative replica electron micrographs were produced as described in Section 4.7. No microstructural change are observed with soaking temperatures up to 1150°C; at this temperature a slight change can be observed. However, at 1200°C (close to the γ' solvus) grain growth becomes more pronounced, and at 1250°C severe grain growth has occurred. This grain growth is believed to be a result of the dissolution of the stabilizing phase at high temperatures.

5.1.2 Composites

Representative optical micrographs of sections normal to the direction of fiber alignment are shown in Figs. (36) and (37) for HIPing temperatures of 1050°C and 1150°C respectively. Consolidation was not complete and sound bonds between the tungsten fibers and the matrix were not achieved in the composites pressed at 1050°C, Fig. (36). However in the 1150°C pressings an even distribution of the fibers with no voids or damage can be observed, Fig. (37-a), while voids in the matrix due to incomplete consolidation during HIPing as a result of a non-uniform distribution of the fibers, are visible in Fig. (37-b). These micrographs were taken from the same specimen, and therefore they illustrate the variation in structure that may be expected from location to location in a given part. These defects shown in Fig. (37-b) might lead to early formation of strain induced damage in the composites during the forging operation due to premature fiber-fiber contact.

During routine metallographic examinations of one of the composite billets prior to forging, several cracks were observed in the tungsten wires, as shown in Fig. (38). Microprobe examinations revealed the presence of the HfN deposit on the surface of the wires and also HfN penetrating into the wires in each of the cracks examined. This clearly indicates that the cracks were present in the wires prior to deposition of the HfN diffusion coating. To determine whether these defects were unique, or whether this is a recurring problem, a sample from a second billet was prepared and examined in the same way. The results of these examinations are as follows.

In the section examined, 88 of the 320 fibers present, or approximately 25%, were found to contain cracks. Electron microprobe examinations were carried out on six cracks chosen randomly from different fibers in the sample. Hafnium was observed in large amounts in all of the cracks examined. Fig. (39) shows the X-ray scan for hafnium at the location of the horizontal microprobe trace. The X-ray scan was set for

hafnium (Bragg angle 22.94° ; LiF crystal, Xe counter) and had a full scale range of 3000 counts per second. It can be seen that there is a considerable Hf concentration (as indicated by the X-ray intensity) where the X-ray scan crosses both the wire surface and the crack. Similarly, Fig. (40) shows an over-all X-ray image of another crack, using the same X-ray conditions as described above. Again it can be seen that there is a concentration of Hf, both on the wire surface and in the crack.

Since every crack examined in the microprobe analysis was found to contain hafnium, the observations indicate that the cracks were not formed during compaction or cutting of the specimen as previously thought but rather were introduced prior to the vapour deposition step possibly during drawing or straightening of the wires.

A slight decrease in the microhardness of the matrix within the composite material, compared to the equivalent non-reinforced matrix, was observed. The average microhardness was found to be 438 DPH for the composite matrix compared to 460 DPH for the non-reinforced matrix. Several factors may influence the observed difference in microhardness. The first, which should cause a relative decrease in the microhardness of the composite matrix, is the difference in the HIPing temperature, namely, 1150°C for the composite and 1050°C for the matrix. It might be argued that at the higher temperature more γ' dissolves and a larger grain size is produced which in addition to other microstructural changes should lower the microhardness of the material. To resolve this question, the effect of the HIPing temperature was examined by comparing the microhardness of two non-reinforced compact, HIPed at 1150 and 1050°C respectively. The average microhardness was found to be 379 DPH for the former as opposed to 460 DPH for the latter.

Two additional factors which may influence the microhardness of the composite matrix are the higher effective strains and strain rates experienced by the composites during HIPing as a result of the presence of hard to deform fibers within the matrix.

its finer grain size. It will be appreciated by the reader that while fine grain size leads to easy flow at high temperatures due to grain boundary sliding, at lower temperature it has the opposite effect and leads to hardening according to the well known Hall-Petch relationship.

$$\sigma = A + B d^{-\frac{1}{2}} \quad (18)$$

where, σ is the yield stress, A and B are constants and d is the average grain size. Therefore, it was anticipated that if a coarse grain size could be developed in Mar M200 prior to extrusion, lower extrusion pressures would be obtained. For this reason, a second set of the Mar M200 bars, each 8 cm long, were heated at 1230°C which is above the γ' solvus, and soaked for 4 hrs to allow grain growth. One specimen was machined from each bar to the configuration given in Fig. (32) then extruded as described in Section 4.10.1. Lower extrusion pressures were obtained as shown in Fig. (55). However, the extrusion pressures for this annealed material were still much higher than those observed for the other nickel-base superalloys which suggested that other microstructural parameters, such as γ' amount and morphology, are influencing the plastic flow stress at room temperature. To examine this question, another set of the Mar M200 bars were heated at 1230°C for 4 hrs then furnace cooled to 1090°C and soaked at this temperature for 24 hrs and cooled in-situ to room temperature. The objective of this particular heat treatment was to produce a large grain size and to cause agglomeration of the second phase which reduces the resistance to flow at room temperature, as will be discussed in the following chapter. Again specimens were machined from the heat treated bars and extruded as above. As expected, the extrusion pressures

sensitivity (m) increases with temperature indicating enhanced superplasticity. On the other hand the values of (m) tend to level off at high temperatures for the superplastic region, while for the conventional region this tendency to level off can be observed at both high and low temperatures.

The peak flow stresses of the Mar M200 forged at 1150°C and at six different strain rates are shown in Fig. (50). These results were obtained by three different methods as explained in Section 4.8. It can be seen that the results are in close agreement where flow occurs superplastically in the range of strain rates between 10^{-4} s^{-1} and 10^{-3} s^{-1} . At higher strain rates, close agreement is only observed when results from individual tests are compared with those obtained using one test in which the strain rate change was done immediately beyond the peak. In contrast, when the peak flow stresses are obtained by changing the strain rate after some deformation beyond the peak, the values are lower than those obtained by the other two methods. The deviation appears to increase with increasing strain rate. This deviation is attributed to grain refinement and other microstructural changes which take place during deformation, as will be described in Section 5.2.4. The differences at the very slow strain rates are related to experimental errors as discussed in Section 4.12. However, it is evident that the peak flow stress of the Mar M200 tends to level off at very slow strain rates. It was difficult to estimate the strain rate sensitivity exponent for this region of strain rate because of the relatively high experimental errors involved. However, the values appear to be of the order of 0.1 to 0.2, and therefore indicate a further transition from superplastic to creep deformation as discussed by Edington et. al.⁴¹

The peak flow stresses of the Mar M200 annealed at 1230°C prior to forging and tested at 1150°C are shown in Fig. (51). The results for the As-HIPed material obtained under identical testing conditions are reproduced in the same graph for comparison. The strain rate sensitivity exponent for the annealed material is constant

over the whole range of strain rates investigated and equal to 0.2. This value is identical to that obtained for the fine grained As-HIPed material, tested under conditions where conventional flow occurs.

5.2.2 Effect of Mesh Size

The effect of mesh size on the peak flow stress of the Mar M200 compacts is shown in Fig. (52). Compacts with the coarser mesh size gave the highest peak flow stresses at all strain rates and temperatures used. Compacts made from the full range of particle sizes but predominantly of a mesh size below -140 produced the lower flow stresses. It should be noted that differences in peak flow stresses for the three mesh sizes are almost constant at all strain rates investigated and do not seem to depend strongly on temperature.

5.2.3 Effect of Prestraining

(a) Extrusion

Extrusions with up to 0.15 reduction ratio were obtained without any apparent damage or surface cracking, Fig. (53). At higher reduction ratios the billets cracked severely during extrusion, Fig. (54). A specimen of the non-extruded end of the billet was mounted, polished and etched for metallographic examination to check the existence of any voids or damage prior to extrusion. No voids or damage were observed. From these observations it can be concluded that the material has a very poor workability at room temperature.

The extrusion pressures recorded for the different reduction ratios as well as some other data obtained from the literature^{99,100} are shown in Fig. (55). Comparing the two curves for Mar M200 and U700, it can be seen that the extrusion pressures for the former material are much higher.

This was attributed to the higher yield strength of the Mar M200 due to

its finer grain size. It will be appreciated by the reader that while fine grain size leads to easy flow at high temperatures due to grain boundary sliding, at lower temperature it has the opposite effect and leads to hardening according to the well known Hall-Petch relationship.

$$\sigma = A + B d^{-\frac{1}{2}} \quad (18)$$

where, σ is the yield stress, A and B are constants and d is the average grain size. Therefore, it was anticipated that if a coarse grain size could be developed in Mar M200 prior to extrusion, lower extrusion pressures would be obtained. For this reason, a second set of the Mar M200 bars, each 8 cm long, were heated at 1230°C which is above the γ' solvus, and soaked for 4 hrs to allow grain growth. One specimen was machined from each bar to the configuration given in Fig. (32) then extruded as described in Section 4.10.1. Lower extrusion pressures were obtained as shown in Fig. (55). However, the extrusion pressures for this annealed material were still much higher than those observed for the other nickel-base superalloys which suggested that other microstructural parameters, such as γ' amount and morphology, are influencing the plastic flow stress at room temperature. To examine this question, another set of the Mar M200 bars were heated at 1230°C for 4 hrs then furnace cooled to 1090°C and soaked at this temperature for 24 hrs and cooled in-situ to room temperature. The objective of this particular heat treatment was to produce a large grain size and to cause agglomeration of the second phase which reduces the resistance to flow at room temperature, as will be discussed in the following chapter. Again specimens were machined from the heat treated bars and extruded as above. As expected, the extrusion pressures

obtained for this case were significantly lower than the previous two cases as shown in Fig. (55).

The microhardness measurements made across the extruded billets of the As-HIPed material, for the different reduction ratios, are presented in Fig. (56). It can be seen that deformation is highly inhomogeneous with larger amounts of cold work introduced close to the periphery of the billet. It was anticipated that this amount of stored energy might be sufficient to cause recrystallization, during heating prior to forging and produce even finer grain sizes that are required for enhanced superplasticity. The true stress-true strain curves of compacts extruded to a reduction ratio of 0.15 prior to forging are shown in Fig. (57), and will be discussed in the following chapter.

(b) Primary Forging

The true stress-true strain curves for the Mar M200 compacts preforged at 1100°C to different amounts of strain are shown in Fig. (58). The top curve represents the flow at a primary strain rate, $\dot{\epsilon}_1 = 8.8 \times 10^{-2} \text{ s}^{-1}$ and temperature, $T = 1100^{\circ}\text{C}$, while the numbers, 1, 2, 3 and 4, indicate the points at which the test was interrupted and the strain rate changed to $\dot{\epsilon}_2 = 1.4 \times 10^{-3} \text{ s}^{-1}$. The flow curve of the As-HIPed compact tested at a strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$, is shown on the same graph for comparison. The As-HIPed material gave a peak flow stress (σ_p) of 59 MN/m^2 whereas the preforged materials gave peak flow stresses of 45, 32, 25 and 22 MN/m^2 , under identical testing conditions. Similar results were obtained at 1150°C , as shown in Fig. (59). At this temperature the As-HIPed material gave a peak flow stress (σ_p) of 33 MN/m^2 at a strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$ whereas the preforged materials gave peak flow stresses of 30, 26, 23 and 13

MN/m² under identical testing conditions. This decrease in peak flow stress, expressed as a percent reduction of peak flow stress in the As-HIPed material is shown in Fig. (60) for each prestrain condition at the two temperatures.

The secondary flow curves for the Mar M200 compacts, preformed at 1100°C to about 60% strain, are shown in Fig. (61) and Fig. (62) for two different initial (primary) strain rates. The percentage reductions of secondary peak flow stress relative to that of the As-HIPed material for identical testing conditions are given in Tables 6, 7 and 8. No significant change can be observed as a result of changing the initial strain rate ($\dot{\epsilon}_1$) from $0.88 \times 10^{-1} \text{ s}^{-1}$ to $2.1 \times 10^{-1} \text{ s}^{-1}$. However, a slight decrease in the percentage reduction of σ_{peak} can be observed with decreasing the strain rate $\dot{\epsilon}_2$. This reduction is believed to be due to grain growth or other microstructural changes as a result of the longer testing period at the slow strain rates.

5.2.4 Effects of Forging on Microstructure

The microstructures shown in Fig. (63) are representative of the structures developed in the Mar M200 compacts forged at 1100°C and 10^{-1} s^{-1} to different strains. They show a microduplex arrangement of γ and γ' grains with γ/γ' , γ/γ and γ'/γ' interfaces randomly distributed throughout the material. Some degree of structural inhomogeneity is evident in all cases, although this is greatly diminished at the higher strain as shown in Fig. (63-c). It can be observed also that the initial fine grains are gradually replaced by new grains of an even finer size. During forging, the coarse grained compacts show partial recrystallization localized mainly along prior grain boundaries, Fig. (64). The carbon replicas of Fig. (65) indicate that the recrystallized grain size is the same order of magnitude in the two compacts and therefore depends

only on the deformation conditions. In a separate study on the same material¹³ it was shown that dynamic recrystallization is responsible for these structural modifications.

The effect of temperature on the microstructure developed during forging under identical conditions of strain and strain rate is shown in Fig. (66). Two regions of fine and coarse grain size can be observed at the two lower temperatures namely, 950°C and 1000°C. The coarse grain regions appear to be relatively large particles which have undergone a very little deformation, or which did not deform at all, and which have therefore maintained their As-HIPed grain sizes. Most of the deformation has taken place in the "interparticle" regions which contain finer particles and grain sizes, and which therefore exhibit lower flow stresses. At the higher temperatures, however, a more uniform structure is obtained as shown in Fig. (66c and d). It is also important to notice that the grain size of the deformed zones increases with temperature, while larger amounts of the second phase have been dissolved. These structural changes have a great influence on the ductility of the compacts and will be discussed in more detail in the following chapter.

5.3 EFFECTS OF PROCESSING VARIABLES ON DUCTILITY OF COMPACTS

Cracking occurs along powder particle boundaries during compression of the Mar M200 compacts as evidenced by the photographs of Figs. (67) and (68). The severity of the cracking varies with the processing conditions and this has been used to obtain a qualitative estimate of the relative ductilities from one test condition to another.

The ductility of the powder compact increases with temperature as illustrated in Fig. (67). These compacts were deformed to 60% strain at an identical strain rate of 10^{-3} s^{-1} . This increase in ductility with temperature is characteristic of the hot working of superalloys and is well documented¹⁰¹. Within the range of strain rates examined, and at a fixed temperature of 1100°C, the apparent ductility increases with decreasing

strain rate Fig. (68). The soundness of the compact forged at $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$ is reasonable considering that some of the apparent porosity is due to carbides being pulled out of the matrix during polishing. However, over the range of strain rates examined no ductility minimum or maximum was observed as reported for IN-100 compacts¹⁰². Again, cracking was more severe along the boundaries of the large particles as depicted in Fig. (68-a and b).

Obviously one would expect more extensive cracking to occur with larger amounts of deformation. To determine the amount of damage introduced to the matrix material with deformation, five specimens were tested under identical conditions of 1100°C and 10^{-1} s^{-1} , to increasing amounts of strain. However, little damage was introduced even at an 80% deformation as shown in Fig. (69). It is important to mention here that the strains contemplated for the composite material are well below this value as will be discussed later.

The effect of cold working by extrusion* prior to forging on the ductility of the matrix material is indicated in Fig. (70). These specimens were forged at 1100°C to the same strain of ~ 0.6 and at strain rates of 10^{-1} , 10^{-2} and 10^{-3} s^{-1} . Comparing these results with the results presented in Fig. (68) for the non-extruded material, it can be seen that the extruded specimens were cracked severely during forging at fast strain rates which indicates the lower ductility of this material at high temperature. It is also apparent that cracking is more severe, and therefore ductility is lower at faster strain rates.

5.4 FLOW BEHAVIOUR OF COMPOSITES

The results on the flow characteristics of the composite material are presented in Figs. (71, 72, 73 and 74) for different temperatures and strain rates. The general

* 15% reduction ratio.

features of the stress-strain curves are similar to those for the matrix material. The curves show an initial elastic portion followed by work hardening up to a peak flow stress. However, beyond the peak flow stress the material work softens and the flow stress decreases with strain until a minimum value is reached at a strain of ~ 0.3 . At higher strains, the flow stress increases again as shown in Fig. (72). Since the composites forged at the other temperatures were not forged to high enough strains, this minimum in flow stress was not observed Figs. (71, 73 and 74).

The strain rate dependence of the peak flow stresses at the different temperatures are presented in Fig. (75) on a logarithmic scale. More than a 4-fold increase in peak flow stress can be observed with an increase in strain rate from 10^{-4} to 10^{-2} s^{-1} while the peak flow stresses at the lowest temperature are about 3 times larger than those at the higher temperature. The strain rate sensitivity exponent for all temperatures is ~ 0.25 which is typical of conventional deformation behaviour and suggests a low degree of superplasticity.

5.5 FRACTURE BEHAVIOUR OF COMPOSITES

The practical working operation which would be considered for composite turbine vanes is unlikely to involve deformations much larger than a few percent. It is therefore important to know whether such strains can be introduced without inducing significant damage, and whether improved formability can be obtained by controlling the deformation parameters. To this end, composite specimens were deformed to progressively larger strains at various strain rates and temperatures, and the effects of prior deformation history on structural integrity examined.

5.5.1 Classification of Forging Induced Defects

A variety of defects have already been shown to be present within the As-HIPed material (Section 5.1.2). However, most of the damage observed in the forged composites seems to have been introduced during the forging process. This damage includes

fiber cracking Fig. (76), extensive cavities at the fiber/matrix interfaces Fig. (77), matrix cracking Fig. (78), and occasional random microvoids in the matrix as shown in Fig. (79).

The number of cracked fibers increased slightly after forging. In most cases cracks started at the periphery of the fibers and propagated inward across the fiber and sometimes extended to the adjacent ones as shown in Fig. (76-a and b). These cracks run longitudinally along the fibers to a considerable length as shown in Fig. (76-c). This is to be expected because of the brittle nature of the highly cold worked tungsten. Obviously, fiber cracking is expected to result in loss of composite strength and is therefore detrimental to the composite system.

Cracking was often observed at the fiber/matrix interface, particularly at locations normal to the loading direction as shown in Fig. (77). In most cases separation was initiated between the hafnium nitride coating and the tungsten fibers as evidenced by the photographs of Fig. (80). Occasionally matrix cracking was observed to occur in areas where no cavities had been formed at the fiber/matrix interface shown in Fig. (78).

Voids can be seen in the matrix of the forged composites and situated along prior powder particle boundaries and particle interstices, as shown in Fig. (79). Occasionally they are seen at grain boundaries inside powder particles as shown in Fig. (78). These voids tend to concentrate in areas where localized high deformation has taken place, mainly at the tensile poles of the fibers normal to the loading direction.

5.5.2 Initiation and Growth of Defects

The photographs in Figs. (81) to (85) provide some insight into the initiation and propagation of strain induced damage in the composites. The As-HIPed material is shown in Fig. (81) for comparison. As discussed in Section 5.1.2 a variety of defects

exist in the As-HIPed material. These defects are important since they may grow or act as initiation sites for new defects during forging.

Little increase in damage can be observed in composite samples forged to low strains ($\sim 3\%$) at 1100°C and $3 \times 10^{-4} \text{ s}^{-1}$, Fig. (82). However, at higher strains ($\sim 6\%$) cracking starts by void formation at the poles of the fibers normal to the loading direction as shown in Fig. (83). Most of the fiber cracking seems to be parallel to the loading direction and tends to concentrate in areas where fibers came into contact, Fig. (83-a). Fig. (83-b) indicates that damage tends to fall in a shear band between the fibers. Further deformation, Fig. (84), increases the size of the voids and initiates new ones. Finally transmatrix cracks propagate along the direction of the maximum shear stress, as shown in Fig. (85).

The severity of the damage induced during forging varies with forging conditions. The effect of strain rate was examined by comparing two specimens deformed to identical strains at 1100°C but at different strain rates ($0.88 \times 10^{-1} \text{ s}^{-1}$ and $9.6 \times 10^{-5} \text{ s}^{-1}$) and the results are shown in Fig. (86). Severe damage was observed in the composite forged at the faster strain rate ($0.88 \times 10^{-1} \text{ s}^{-1}$) Fig. (86-a), while somewhat less damage was observed in the composite forged at the slower strain rate ($9.6 \times 10^{-5} \text{ s}^{-1}$), Fig. (86-b). Fig. (86-a) also indicates that fracture is concentrated in bands parallel to the direction of maximum macroscopic shear stress in the specimen.

Fig. (87) shows a transverse section through a composite specimen forged at 1180°C and $3 \times 10^{-4} \text{ s}^{-1}$. It can be seen that the voids are mainly formed at the fiber/matrix interface, while no voids or damage can be observed within the matrix itself. Comparing Figs. (84) and (87) for two composites, forged to strains of 0.1 at the same strain rate of $3 \times 10^{-4} \text{ s}^{-1}$, but at two different temperatures of 1100°C and 1180°C respectively, it can be seen that a larger number of damage free fibers exist in the composite forged at the higher temperature as shown in Fig. (87). Also, fewer matrix

voids initiate at the higher temperature, while the sizes of the interface cavities were larger than were formed at the lower temperature. These observations will be discussed in the following chapter.

Careful examination of the forged composites revealed areas denuded of blocky γ as indicated by the white layers around the fibers shown in Fig. (87-b). At high magnification, Fig. (88) (region A), it can be seen that the material in these bands has a recrystallized and twinned grain structure, with a grain size larger than that in the adjacent matrix (region B). These white bands or layers are particularly prominent in regions containing voids or cracks, as indicated by the arrows in Fig. (89-a, b, c), and in shear bands where highly concentrated plastic deformation has occurred as shown in Fig. (89-d). Microprobe examinations revealed high concentrations of aluminum on the interior surfaces of the voids adjacent to the denuded zones as shown in Fig. (90-a). In contrast there was no evidence of a concentration of aluminum at the fiber/matrix interface where the interface was intact, Fig. (90-b). Similar results were obtained for chromium as shown in Fig. (91).

6 DISCUSSION

6.1 INTRODUCTION

The objective of this work was to explore the general feasibility of forging fiber reinforced composites and to improve the fundamental understanding of these materials, in order to contribute to the development of appropriate advanced fabrication processes, such as isothermal or superplastic forging.

In order to fully appreciate the complications and limitations imposed by the strong reinforcing fibers it was decided first to characterize the forging behaviour of the candidate matrix alloy compacts without the fibers. The aim of this part of the project was to provide a comprehensive study of the means by which grain size can be controlled in superalloy compacts, and secondly to examine the influence of the established grain size on the forging characteristics of these materials. The factors which were thought might influence grain size and which have therefore been examined in this work include, (a) material variables such as mesh size, (b) consolidation variables of temperature and pressure and (c) thermomechanical processing operations such as cold working by extrusion, or primary hot working prior to forging. Also, since the high temperature flow and fracture behaviour of superalloys was expected to be sensitively dependent on temperature and strain rate⁴¹, these variables were also investigated. Overall, it was hoped that this work would provide an improved understanding of hot working mechanisms and superplasticity in nickel-base superalloy compacts.

The lack of a comprehensive study of the forging and fracture behaviour of advanced composite materials, such as those considered for use in advanced turbine blades, justifies a fundamental approach to the problem. No information was available in the literature regarding forging loads, fracture behaviour and forging defects that

might occur during processing. Work along these lines was to be undertaken in the present study. Also, the flow and fracture behaviour of these materials as a function of temperature, strain rate and strain were to be investigated.

6.2 MICROSTRUCTURES OF THE STARTING MATERIALS

The motivation for the use of powder metallurgy processing has been discussed already in the introduction. The most significant benefits as far as this study is concerned, are the improved homogeneity and superior workability that can be achieved over conventionally cast materials.

The microstructures of the starting powders, shown in Fig. (92), do indicate that dendritic segregation exists on a much finer scale than in conventionally cast material. The coarse particles of eutectic γ / γ' which exist in cast alloys were absent, and the grain sizes were at least an order of magnitude smaller than is observed in equivalent cast alloys.

Examinations of the microstructures of the as-HIPed materials, Fig. (34), showed that these attractive features of the powders are carried over into the consolidated powder compacts. In particular, by pressing below the γ' solvus temperature, the fine grain size of the powders was retained, and the fine dendritic segregation was partially eliminated.

In the starting powders the γ' phase appears to be only partially precipitated, and an important reaction during hot isostatic pressing is the precipitation and gradual agglomeration of the equilibrium volume fraction of γ' . It is these particles which act as obstacles to grain boundary movement during pressing. The as-pressed materials therefore contain these relatively coarse particles of averaged or blocky γ' , and smaller particles of cuboidal secondary γ' which are believed to have formed during the slow cool from the pressing temperature in the autoclave. Both types of γ' particles can be

seen in Fig. (34-b). These particles are believed to influence the subsequent flow behaviour of the material during forging, as will be discussed in detail later.

Notwithstanding the fact that the material showed generally favourable response to powder processing, several undesirable features were observed in the compacts. For example, many observations were made of relatively large powder particles remaining undeformed in the compacts. These larger particles tended to have somewhat larger grain sizes than were observed elsewhere in the compacts, and it will be shown later (Section 6.5) that they were particularly resistant to deformation during forging. At the same time it is well known¹⁰² that the particle surface boundaries in these compacts tend to act as preferential sites for the precipitation of carbides during pressing. Examination of the compacts and composites, in the unetched condition, showed that some carbide-like particles were dispersed throughout the matrix and some were located on the powder surface boundaries. It was found during the hot working studies that cracking occurred preferentially along these boundaries, and therefore this may be a result of the presence of embrittling carbide or possibly oxide films, or the result of strain discontinuities between the coarser-grained deformation resistant particles and the softer fine-grained particles. These matters will be discussed later in Section 6.5.

In the case of the compacts, it was found that all pressing temperatures of 1050°C and above gave fully dense materials. Since the equilibrium volume fraction of γ increases with decreasing temperature, lower pressing temperatures are expected to be more effective in retaining fine grain size. In the case of the composites however, this temperature was not adequate to produce full consolidation and sound bonds between the tungsten fibers and the matrix. This was shown in the photographs of Fig. (36) for a composite material HIPed at 1050°C. Matrix cavities and fiber cracking were observed in several areas of the composite blank. At this temperature, the matrix was not soft enough to flow and close the voids without producing damage into the fibers. Consequently,

the HIPing temperature for the composites was raised to 1150°C to obtain full consolidation.

In some isolated cases, however, voids were still observed in the as-HIPed composites essentially in areas where fibers were not uniformly distributed, Fig. (37-b). Such areas can also be expected to act as preferential sites for damage initiation during the forging operation, due to premature fiber to fiber contact. Therefore, it is recommended that efforts be made to ensure proper alignment of fibers at the lay-up stage prior to HIPing. This would also tend to diminish any local inhomogeneity in the properties of the final product.

6.3 OBSERVATIONS ON EXTRUSION PRESSURES

Hydrostatic extrusion at room temperature was carried out on several Mar M200 powder compacts, since this was a convenient way of introducing cold work to the samples prior to forging. During this work it was found that extrusion pressures for a given reduction ratio diverged from values published in the literature^{99,100} for similar nickel-base superalloys. Before discussing the effects of this cold work on forging behaviour it is informative to examine the extrusion behaviour of the compacts.

Several empirical equations have been proposed relating extrusion pressure (P_E) to extrusion ratio (R) and material properties. For example an equation of the type:

$$P_E = a (\ln R + n) \quad (19)$$

where a and n are constants depending on the material and extrusion conditions, has been widely used¹⁰³. Similarly, Pugh¹⁰⁴ found that an equation relating extrusion pressure, Vickers hardness (H_v) and extrusion ratio could describe his data:

$$P_E = (5.78 H_v + 62) \ln R \quad (20)$$

While Lowe and Goold¹⁰⁵ found their results were best described by the equation:

$$P_E = (4.24 H_v + 144.5) \ln R \quad (21)$$

where the extrusion pressure is given in MN/m².

Not unexpectedly the extrusion pressures for the Mar M200 compacts were considerably higher than have been reported for other nickel-base superalloys^{99,100}, since the materials and conditions were different, Fig. (55). However, it was surprising that the published data for a number of different alloys were observed to follow a linear relationship with extrusion ratio. This suggests that metallurgical factors such as alloy composition, grain size, precipitate content and morphology, and prior processing history which influence hardness and yield strength, have little effect on extrusion behaviour.

To rationalize the present data, and to demonstrate the relationship between microstructure, or mechanical properties, and extrusion pressure, a variety of heat treatments were carried out to produce different microstructural conditions in the Mar M200 extrusion billets.

The extrusion pressures presented in Fig. (55), for the Mar M200 billets subjected to different heat treatment regimes, suggest that the flow of the nickel-base superalloys at room temperature can be greatly influenced by the microstructural condition of the material. The relationship between microstructure and flow strength of the material will be discussed below. As mentioned previously (Section 5.2.3(a)) three distinct microstructures were investigated.

Firstly, the as-HIPed material Fig. (34), had a very fine grain size, with grains consisting of the γ austenitic solid solution and relatively coarse particles of the agglomerated γ' phase. The grain sizes and gamma-prime particle sizes are in the range 2-8 μm and 0.4 - 1.5 μm respectively. This material produced the highest extrusion pressures at all reduction ratios used. Secondly, the as-HIPed and annealed material (1230°C/4 hrs), shown in Fig. (93-a) possessed a very large grain size in the range 10 to 50 μm , and an ultra fine gamma-prime particle size. Extrusion pressures were reduced by 24% in this case.

Several factors are thought to influence the extrusion pressures of the material. The first is directly related to the grain size, and according to the Hall-Petch

relationship an increase in grain size should lead to a decrease in yield strength or hardness. Therefore, according to Equation (21), a decrease in extrusion pressure should be observed. However, it is clear that the observed reductions in the flow strength of the annealed material are less than would be expected if grain size was the only factor governing the flow strength of the material at room temperature. The second factor affecting extrusion pressure is the amount and morphology of the second phase. Fine γ' particles, within the grains of the annealed material, work as obstacles to dislocation movement. The larger the amount of this fine γ' the more significant the strengthening effect and the higher the flow strength of the material. By reducing the strengthening effect of the second phase, the extrusion pressures were lowered by a further 9% as shown in Fig. (55). This was achieved by causing the ultra fine γ' particles of the coarse grain material to agglomerate by appropriate heat treatment (1090°C/24 hrs). The microstructure of such material is shown in Fig. (93-b).

These experiments demonstrate quite clearly that hydrostatic extrusion pressures for nickel-base superalloys are critically dependant on the grain size and precipitate structure in the material. These latter are, of course, the metallurgical variables that influence yield strength and hardness of the material. Unfortunately the published data on hydrostatic extrusion pressures for other nickel-base superalloys cannot be interpreted in any detailed way, and the validity of these data is uncertain.

6.4 FLOW BEHAVIOUR OF COMPACTS

6.4.1 Form of the Flow Curves

The flow curves of the matrix material, obtained at constant true strain rate of $3 \times 10^{-4} \text{ s}^{-1}$, are reproduced in Fig. (94). A comparison with the results presented in Figs. (41 to 46), indicates that the effect of increasing the temperature on flow is very similar to that of decreasing the strain rate. Both lead to reduced flow stresses and to decreasing amounts of flow softening. Most of the flow curves consists of an

initial pseudo-elastic region followed by work hardening to a peak stress. Beyond the peak the material flow softens until finally a steady state regime of flow is reached during which the flow stress remains essentially constant with increasing strain. At high enough temperatures and low enough strain rates, no flow softening occurs and instead the flow stress rises continuously to its steady state level.

The form of these curves can be rationalized in terms of the competition between several physical processes which are occurring simultaneously during flow. These processes can be divided into two groups: those that lead to flow hardening and those which result in flow softening. When a balance between hardening and softening effects is struck, the flow stress becomes constant and the material subsequently remains in a steady state regime of flow¹⁰⁶.

As the as-HIPed compacts begin to deform, their flow strength rises due to work hardening. The microstructural changes which occur during work hardening have not been examined in detail, but the process can be understood in terms of dislocations and their interaction with each other and with precipitate particles in the material. As the movement of dislocations through the lattice becomes more difficult, larger stresses are required to maintain the imposed strain rate. The increase in stored energy associated with this strain hardening can eventually become large enough to initiate dynamic recrystallization, a process known to produce decreases in flow strength during high temperature flow¹⁰⁶⁻¹⁰⁸. In Mar M200 compacts, the nuclei are known to form mainly along grain boundaries¹³ (see also Figs. (64,65)) so that as deformation proceeds, the grain size is gradually refined as new waves of recrystallization occur with increasing strain. Deformation also tends to accelerate the coarsening and redistribution of the second phase throughout the material. These changes lead to a precipitate morphology which is far less efficient in retarding flow. These dynamic structural transformations can be followed in the micrographs of Fig. (63) which show

the microstructures of the present material forged to different strains. Grain refinement can be observed in Fig. (63-a, b and c). While Fig. (63-d, e and f) show the second phase coarsening and change in morphology from a cuboidal shape to a more spherical and therefore less coherent morphology. It is clear that in these compacts, the flow strength decreases with strain because the microstructure of the material reverts to a condition which offers less resistance to plastic flow than that provided by the original microstructure.

The absence of any detectable flow softening or peak in the flow curves of compacts forged at very high temperatures and very slow strain rates can be attributed to the highly effective nature of the work softening processes which occur under these forging conditions. In these cases the stored strain energy never gets large enough to provide the driving force for the initiation of dynamic recrystallization and the grain size is not refined. There is some evidence in fact that the grain size tends to grow during flow, as shown in Fig. (66-d), and therefore it appears that the steady state grain size is a function of the working conditions of temperature and strain rate.

It is quite clear from the above discussion that the microstructures of compacts have a very strong influence on their plastic flow behaviour. Although it is not possible to determine what proportion of flow softening is due to the different microstructural changes, it is unlikely that the averaged γ' in the as-HIPed compact provides much strengthening and therefore can not account for a large proportion of the subsequent flow softening. Conversely, there is some evidence that changes in grain size are responsible for the majority of the flow softening in similar materials.¹⁰⁹

Changes in grain size are important in high temperature flow because these nickel-base superalloy compacts deform to some degree by grain boundary sliding and exhibit some degree of superplasticity. Therefore, before any further insight can be made into the hot working behaviour of these materials, the results on the effects of grain size on flow should be examined carefully.

6.4.2 Effect of Grain Size

The results presented in Fig. (51) for the peak flow stress of the annealed and as-HIPed materials indicate clearly that grain size has a substantial effect on the high temperature flow strength of the compacts. It is important to mention that the grain size of the annealed material was estimated to be in the range from 10 to 50 μm , and that of the as-HIPed material was estimated to be 2 to 8 μm .

The relatively high strain rate sensitivity exponent $m = 0.6$, in the case of the fine grained compacts, compared to $m = 0.3$ in the coarse grained material, indicates that superplasticity is strongly dependent on the presence of a fine grain size.

The ratio of the peak flow stress of the annealed material to that of the as-HIPed material at different strain rates is shown in Fig. (95). From this figure, it can be seen that, at the slowest strain rate, the peak flow stress of the annealed material is higher by almost one order of magnitude than that of the as-HIPed material. However, at high strain rates this ratio tends towards a constant value close to unity.

This difference is attributed to changes in deformation mode and grain size sensitivities in going from high to low strain rates. At low strain rates, flow occurs to some degree by grain boundary sliding in both materials and the strong grain size dependence gives rise to a large difference in flow strength. By contrast, at high strain rates, flow occurs mainly intragranularly, and the reduced grain size dependence in this case results in a proportionately smaller difference in flow strength.

6.4.3 Means of Controlling Grain Size

Since plastic flow in the Mar M200 compacts appears so strongly dependent on grain size, it is important to discuss the means by which grain size can be controlled during processing. As stated previously, grain size can be controlled in a variety of ways and some of these have been examined in the present study. A finer mesh size was selected for the compacts in the hope that the initial grain size of the as-HIPed

material would be smaller. Secondly, the compacts were cold worked in the hope that the associated stored energy would be sufficient to initiate dynamic recrystallization sooner. Finally, the effects of forging variables themselves on the grain size produced during deformation were examined and will be discussed below.

(a) Mesh Size Control

Although there is no detailed evidence of a direct relationship between grain size and particle size in argon atomized powders¹¹⁰ it might be argued that the finer particles should have finer grain sizes. Indeed the results of Fig. (92) indicate that this is the case. However, the grain size can vary within the same particle, particularly in the larger ones, as shown in Fig. (92-b). It must be emphasized also, that the morphology of the grains varies with particle size. Larger particles tend to have somewhat equiaxed grains, while the fine ones tend to have a more pronounced dendritic structure, Fig. (92-a).

Compacts made from the finer mesh portion of the powder exhibited a slightly higher degree of superplasticity. Such enhancements of superplasticity, in the form of lowered flow stresses are shown in Fig. (52). Since the average grain size of the larger particles was estimated to be 2 to 4 times the grain size of the smaller particles, and since the flow stress is directly proportional to the grain size³⁵, it is clear that the reductions observed are less than would be expected from the grain size effect alone. This observation might be attributed to the higher strengthening effects of the dendritic structure in the fine particles as opposed to the more homogeneous microstructure in the coarser particles.

(b) Prior Cold Working

The true stress-true strain curves of compacts extruded to a reduction ratio of ~ 0.15 prior to forging are shown in Fig. (96). The flow curves of the

as-HIPed material tested under identical conditions are reproduced in the same figure for comparison. The general features of the flow curves are similar in both types of material. However, the extruded compacts show slightly lower flow stresses and higher rate of flow softening. This flow softening may be attributed to grain refinement and structural modifications which might be induced during exposure to the forging temperature, either before or during the initial deformation.

It should be recalled that relatively little strain energy is introduced during hydrostatic extrusion within the bulk of the material. This was clearly demonstrated by the hardness measurements made across the extruded bars (Section 5.2.3 (a), Fig. (56)). Little driving force is therefore available for recrystallization in most of the bulk of the material. Unfortunately the limited ductility of the compacts at room temperature did not allow larger extrusion ratios to be obtained. Because of these limits, the hot working operation itself appears more effective at initiating grain refinement at the temperatures examined, as will be discussed below.

(c) Prior Hot Working

The microstructures shown in Fig. (63) indicate that when Mar M200 compacts are deformed at fast strain rates, the initial grains are gradually replaced by finer grains. The fine intragranular γ' tends to coarsen and agglomerate. A microduplex structure of γ and γ' equiaxed grains gradually develops with increasing strain Fig. (63-f). As pointed out previously, these microstructural changes are responsible for reducing the flow stresses during secondary forging. This is demonstrated in Figs. (58 and 59).

The general features of the secondary flow curves are similar to those of the as-HIPed material described in Section 5.2. Also, it can be observed (Figs. 58 and 59)

that as the initial strain is increased, a transition in flow behaviour takes place from one where the compacts flow soften during deformation to one where flow hardening occurs instead. In all cases, however, the flow stress developed at high strains tends to converge to a unique constant level. It is clear that this steady state stress level appears to be independent of prior history and is governed by the imposed strain rate at a particular temperature.

The effect of temperature on the percentage reductions of the peak flow stresses of the matrix material preforged to different levels of strain, is presented in Fig. (60). Higher reductions are obtained at the lower temperature. This can be attributed to the finer grain sizes developed during primary forging at the lower temperature. The microstructures shown in Fig. (66) supports this view. As the amount of prestrain increases the microstructure developed becomes closer to the steady state structure. Therefore, on decreasing strain rate at high values of strain, the reduction of flow stress is less pronounced. This effect is observed at both temperatures examined, as shown in Fig. (60).

6.4.4 Strain Rate Sensitivities and Modelling of High Temperature Flow

As pointed out earlier (Section 5.2), the measured strain rate sensitivities may vary substantially with the microstructural condition of the material, as well as with the deformation conditions of temperature and strain rate. This can only be rationalized if the mechanisms by which flow takes place are considered more closely.

Two main mechanisms are thought to contribute to high temperature plastic flow in stages II and III, namely grain boundary sliding ($m = 0.5$) and intragranular flow ($m = 0.25$). These two processes occur simultaneously and act additively, each contributing different amounts to the total deformation. Grain boundary sliding contributes to the overall rate of deformation at a strain rate $\dot{\epsilon}_1$, and intragranular

flow contributes at a strain rate $\dot{\epsilon}_2$. The overall rate of deformation is thus obtained by adding the appropriate proportions of the two quantities:

$$\dot{\epsilon} = f_1 \dot{\epsilon}_1 + f_2 \dot{\epsilon}_2 \quad (22)$$

where f_1 and f_2 are the proportions of the total strain due to grain boundary sliding and intragranular flow respectively. These proportions depend upon the microstructural features of the material and the deformation conditions of temperature and strain rate.

In the fine grained material and at low strain rates, deformation occurs mainly by grain boundary sliding i.e. $f_1 \gg f_2$. However, at higher strain rates, grain boundary sliding cannot easily maintain the required deformation and an increased proportion of intragranular deformation takes place. The transition from a high strain rate sensitivity ($m \sim 0.5$) at low strain rates, to a low value ($m = 0.25$) at fast strain rates, Fig. (48), is therefore due to the change in the dominant mode of deformation from grain boundary sliding to intragranular flow. On the other hand, in the coarse grained material the initial grain size is too large, and the strains achieved by grain boundary sliding are negligible. The dominant mode of deformation is therefore intragranular flow, and $f_2 \gg f_1$. In this case a strain rate sensitivity exponent of about 0.3 was obtained over the entire range of strain rates examined, as shown in Fig. (51).

The temperature dependence of "m", shown in Fig. (49), can be attributed to variations in the proportions f_1 and f_2 of the two processes which contribute to deformation. At higher temperatures f_1 increases leading to increases in "m" in the range from 0.25 to 0.5. However, this does not explain why in some cases the observed values of "m" fall outside this range? This may be due partly to contributions from other deformation processes which have not been considered. For example, diffusion controlled creep is usually observed to produce a strain rate sensitivity exponent of 1.0⁴¹. Another possibility is that microstructural changes

may take place during deformation prior to the peak flow stress and may exert a larger effect on flow stress at slower strain rates than at higher strain rates. If this situation existed then the strain rate sensitivity plot would be constructed from peak flow stress data for non-identical materials. We can consider these possibilities more thoroughly if we first consider the various stress components which are thought to make up the measured flow stress during hot working.

At hot working temperatures, plastic flow occurs by the generation and movement of dislocations¹¹¹. The motion of these dislocations is hindered by various obstacles such as other lattice defects, or precipitate particles. The resistance to plastic flow is therefore influenced by the ease with which the moving dislocations can overcome these obstacles, as well as the rates at which dislocations are introduced into, or removed from the material. The apparent flow stress measured in the compression tests therefore contains a number of separate contributions, each of which is associated with a particular type of obstacle.

At the temperatures examined, both thermal and athermal obstacles exist in the Mar M200 compacts. Thermal obstacles may be overcome by the combined effects of stress and lattice thermal vibrations. They are responsible for the thermally activated nature of flow. Athermal obstacles on the other hand are too large for atomic thermal vibrations to be of any significance and therefore temperature has little effect on their resistance to flow. In the present compacts the athermal obstacles are the fine intragranular γ' particles, the fine secondary carbide particles, and "immobile" dislocations.

The flow stress can thus be written as:

$$\sigma = \sigma_e + \sigma_i + \sigma_p = \sigma_e + \sigma_o \quad (23)$$

where σ_e is an effective stress arising from the presence of thermal obstacles, σ_i is an average internal stress associated with other dislocations and σ_p is another athermal stress necessary to overcome the second phase particles.

A sudden change in strain rate (or temperature) during flow gives rise to an instantaneous change in σ_e , but not in σ_i or σ_p . However, with time both σ_i and σ_p will change as the microstructure of the material adjusts to become either more resistant to plastic flow or becomes softer. This rationalization is important because it provides a simple explanation of the variations in flow stress and strain rate sensitivities observed in this work.

If the structure of the material remains constant (σ_i and σ_p are constants) then the strain rate (and temperature) dependence of flow stress will indicate the way that σ_e is influenced by these variables. For example the effective stress increases with strain rate and decreases with temperature. The effective stress is also a function of the density of dislocations available for thermal activation. In the hypothetical case where thermal obstacles are completely absent, no strain rate or temperature sensitivity is expected and the flow stress is simply equal to σ_0 . It should be quite evident that if $\sigma_0 \gg \sigma_e$, low strain rate sensitivity of flow is expected. Indeed, low strain rate sensitivity of about 0.12 was observed in the coarse grained Mar M200¹³. In this material both σ_p and therefore σ_0 were very high due to the strengthening effect of the fine intragranular γ' .

A strain rate sensitivity of 0.5 was obtained for fine grained microduplex compacts, as shown in Fig. (97). These materials were obtained by preforming the compacts to 60% deformation at strain rates of $8.8 \times 10^{-2} \text{ s}^{-1}$ or $2.1 \times 10^{-1} \text{ s}^{-1}$, then unloading and immediately restraining in-situ at different strain rates (see Section 4.10.2). Since this preforming treatment should lead to an effective decrease in starting grain size, one would expect the resultant strain rate sensitivity to be at least as large as that observed for the as-HIPed material. However, a lower value of "m" of 0.5 was observed and an anomalous situation exists. This can be explained in terms of the changes which occur in the microstructure during testing. During straining to the peak

the as-HIPed material undergoes microstructural changes which differ at high and low strain rates. The slower the rate of deformation the longer the time to the peak, and the more significant are the changes which occur. Consequently the apparent strain rate sensitivity is not obtained at constant strain rate. Coarsening of the fine γ_2 and annihilation of lattice defects are more pronounced at the lower strain rates, thereby leading to larger reductions in σ_p and σ_i than at high strain rates. This has the effect of lowering the peak flow stress more at low $\dot{\epsilon}$ than at high $\dot{\epsilon}$ and the apparent strain rate sensitivity increases accordingly. Conversely, in the preformed compacts, a fine microduplex structure is produced prior to the reforging and the deformation takes place mainly by grain boundary sliding. In this case only minor microstructural changes take place during straining to the peak, and a true strain rate sensitivity of 0.5 is obtained.

From the previous discussion it should be quite evident that the instantaneous flow stress in the compacts is strongly dependent on the current microstructure. Therefore, any constitutive law which describes the plastic flow behaviour of a material at elevated temperature should account for the microstructural changes which take place during deformation. This could be expressed as a set of coupled equations

$$\sigma = f(\dot{\epsilon}, T, S) \quad (24)$$

$$\frac{dS}{dt} = f(S, \dot{\epsilon}, T) \quad (25)$$

Here the flow stress is a function of strain rate $\dot{\epsilon}$, temperature T and a structure parameter S which is itself a function of strain, strain rate and temperature, or more generally of the thermomechanical history of the material. The structure parameter is included to account for the effects on flow of microstructural features such as dislocation density, size and shape of grains, and precipitate morphology. The differential equation, Eq. 25, is included to account for changes which may be induced

during deformation. Assuming that the major microstructural changes occurring during superplastic flow are changes in the microduplex grain size, then, for constant strain rate and temperature Eqs. 24 and 25 can be written as

$$\frac{d\sigma}{dt} = \left(\frac{\partial \sigma}{\partial d} \right)_{\epsilon, T} \frac{d(d)}{dt} \quad (26)$$

where d is the grain size.

Changes in flow stress should therefore be controlled by the functional relationship between stress and grain size and by the direction of change in grain size.

Since the flow stress in a superplastic material deformed at constant strain rate and constant temperature is directly proportional to its grain size, then the direction of change in flow stress will be the same as the direction of change in grain size¹⁰⁹.

This was indeed observed in the Mar M200 compacts. Referring to Figs. (58 and 59), at point 1, shortly beyond the peak, little change in microstructure had occurred and the ratio (R_d) of initial grain size to steady state grain size is expected to be greater than unity. Hence, the material work softens and the grain refinement continues with deformation. At higher strains point 4, the material had been work softened sufficiently that the grain size became finer than the steady state grain size i.e. $R_d < 1$. On retesting the material, work hardening occurs and the flow stress increases until a steady state stress is reached.

To illustrate this phenomenon further, the flow stresses of the matrix material forged at the same strain rate but at different temperatures have been reproduced in Fig. (98), along with the steady state microstructure corresponding to each individual forging condition. The microstructure of the as-HIPed material is also reproduced on the same figure for comparison. It is clear that the steady state grain size increases with temperature as indicated by the arrow, Fig. (98). Considering that very little change in the microstructure has taken place during the soaking period prior to forging at these temperatures (see Section 5.1.1), it is believed that the microstructural changes

are produced during forging. It is also interesting to notice that the grain size produced at 1150°C is slightly larger than that of the as-HIPed material, which indicates that in this case grain growth has taken place. Referring to the flow curve obtained at this temperature Fig. (98), it can be observed that gradual work hardening occurred until a steady state flow stress was reached without any detectable flow softening or peak.

In contrast, at the lower temperatures the material work hardens rapidly to a peak flow stress. Beyond the peak, the material work softens until finally a steady state flow stress is reached at higher values of strain. Dynamic recrystallization to a finer grain size is believed to be the cause of this softening process. This is evidenced by the very fine microstructure produced Fig. (98-b). It will be appreciated that in an engineering part of complex shape that either one or both of these processes (work hardening and/or flow softening) may take place during forging. This will depend on the local strain and strain rate which are developed, and on the local microstructure which exists. Therefore, in designing a practical forging operation, it is important to control both the amount and rate of deformation introduced at particular locations, not only to avoid fracture, but also to produce the microstructure whose mechanical properties best meet the service requirement of the end product. One should also appreciate that most commercial parts, produced in the manner described above, will be heat treated prior to use. At the moment the influence of the as-forged microstructure on heat treatment response, and hence in-service properties, is not known. Further work is needed in this area.

To provide some insight into the high temperature flow behaviour of the Mar M200 during the initial stages of deformation, the stress-strain relationships were examined in what appeared to be the elastic portions of the flow curves. From the stress-strain curves recorded for each set of testing conditions, it is possible to determine an apparent modulus of elasticity, which will be referred to as E^* . These values are

obtained from the initial slopes of the stress-strain curves, at low strains, where stress appears to vary linearly with strain. The values of E^* obtained at different temperatures, as a function of strain rate, are shown on a logarithmic scale in Fig. (99). The general pattern of these curves is very similar to the curves of peak flow stress shown in Fig. (48). Two regimes of behaviour can be identified where the values of E^* vary linearly with log. strain rate. At low strain rates ($< 10^{-2} \text{ s}^{-1}$), the slope of these plots (m^*) is close to 0.5, while at higher strain rates ($> 10^{-2} \text{ s}^{-1}$) the slope falls to about 0.1.

It is important to mention that the values of E^* obtained from the stress-strain curves are in the range from $\frac{E}{5}$ to $\frac{E}{70}$, where E is the dynamic elastic modulus¹¹². These very low values of the apparent elastic modulus indicate that at high temperatures the initial strains, occurring at very low stresses, are a result of plastic flow rather than elastic deformation. By analogy with the strain rate sensitivity of flow stress (m), one can describe m^* as a strain rate sensitivity of "apparent modulus", or more correctly as a strain rate sensitivity of micro-plastic flow stress, where:

$$m^* = (\partial \ln E^* / \partial \ln \dot{\epsilon})_{T, \text{Structure}} \quad (27)$$

The values of m^* given above suggest that micro-plastic flow occurs by the same mechanisms as macroscopic flow, with grain boundary sliding and superplasticity occurring at low strain rates, and intragranular flow occurring at higher strain rates. Furthermore, at higher temperatures the overall level of E^* is reduced and the transitions between the two regions are displaced to higher strain rates.

The above observations suggest that the apparent modulus of elasticity E^* might be used to calculate the strain rate sensitivity for high temperature flow. This method is quite fast and relatively simple and therefore has obvious merit. Furthermore, little deformation is needed to determine the apparent elastic modulus, and therefore negligible microstructural changes are introduced.

The flow process producing these micro-plastic strains may occur as a result of the relaxation of residual elastic stresses, and therefore be similar to the processes which occur during standard stress relaxation tests. Indeed stress relaxation tests have been widely used by Hart and his co-workers^{113,114} to determine strain rate sensitivities, in which case the plastic strain rate is determined from the natural rate of stress relaxation. However, the above correlation is between apparent modulus and imposed strain rate, and therefore it is less clear how natural relaxation would influence the measured strain rate sensitivities. The fact that a correlation is obtained between values of m and m^* indicates that elasticity effects are negligible, and therefore micro-plasticity occurs through the long range movement of dislocations in direct response to the applied load.

6.5 FRACTURE BEHAVIOUR OF COMPACTS

It has been shown by many investigators^{68,115,116} that fracture in ductile metals occurs by the development of voids at sites where stress concentrations occur, such as grain boundaries, inclusions and second phase particles. In compacts made from powder by hot isostatic pressing, such as the superalloy used in this study, voids tend to initiate at the particle boundaries. It has been suggested that powder boundary cracking is due primarily to the presence of embrittling carbides¹¹⁷ and the morphology of this carbide film has been found to influence ductility¹⁰². The available evidence suggests that the coarser particles in argon atomized powders have slightly coarser grain sizes as indicated in Fig. (92). During HIPing these particles remain undeformed because of their higher resistance to flow. Similarly, during forging, most of the deformation takes place in the regions of fine grains and these grains recrystallize to an even finer grain size. In contrast the coarser particles show little evidence of deformation and recrystallification, and hence discontinuities are created at their surface boundaries.

Assuming that the volume fraction of the undeformed particles in a compact is p , and if the imposed strain and strain rate during forging are ϵ and $\dot{\epsilon}$ respectively, then the effective strain and strain rate in the deforming regions will be ϵ^* and $\dot{\epsilon}^*$ respectively, where

$$\epsilon^* = \frac{\epsilon}{1 - p} \quad (28)$$

and

$$\dot{\epsilon}^* = \frac{\dot{\epsilon}}{1 - p} \quad (29)$$

Obviously any increase in p increases both ϵ^* and $\dot{\epsilon}^*$ which accelerates void initiation and growth.

It is the concentration of strain around these coarse, undeformed and unrecrystallized particles which promotes void initiation and fracture as demonstrated in Fig. (100). This is in line with the results of Luton and Tegart¹¹⁶ which show rapid growth of cracks along the entire grain boundary length of the unrecrystallized grains of nickel and nickel-iron alloys. In the case of the coarse grained material, deformation is concentrated in the "grain boundary mantle" due to the higher strengthening effect of the γ' in the interior of the grains. Therefore, cracks develop at points of stress concentration at the grain boundaries, as shown in Fig. (64). The net result is that ductility in the coarse grained materials is considerably less than in the fine grained materials under similar working conditions.

The effect of temperature on the fracture behaviour of the Mar M200 compacts is shown in Fig. (67). When these observations are compared with the flow stress results of Figs. (94 and 98), it can be seen that the stronger materials are, in general, less ductile. These observations are in line with the classical interpretation of fracture known as the Ludwik-Davidenkov hypothesis¹¹⁸, which states that a metal can respond to an applied load by either yielding or fracturing, and the observed fracture stress and ductility reflect a competition between these two processes. In superalloys flow is known to be sensitive

to changes in temperature and strain rate^{29,30,41}, thus, fracture occurs more readily at low temperatures and fast strain rates. The data of Figs. (67 and 68) suggest that these ideas hold qualitatively for the fracture of the superalloy compacts, and that deformation at high strain rates and/or low temperatures results in less ductility because flow is more difficult.

6.6 FLOW BEHAVIOUR OF COMPOSITES

In order to compare the flow behaviour of the composites with that of the matrix, some of the stress strain curves are reproduced for both materials in Figs. (101 to 104) for identical testing conditions. Figs. (101 and 102) illustrate the difference in behaviour at the higher temperature while Figs. (103 and 104) are for the lower temperature. It can be seen that the peak flow stresses are much higher in the case of the composites than those observed for the matrix. Similar observations were recorded by, Ertürk⁶⁴ who observed ~ 30% increase in the compressive flow stress of the composite relative to that of the matrix forged under similar condition. However, in Ertürk work the tests were carried out at constant ram speed and the effect of temperature and strain rate on the flow and fracture behaviour of the composite material was not studied.

The increase in peak flow stress for the composites can be attributed to several factors. Firstly, the hard fibers act as inclusions and increase the resistance to flow of the composite by increasing the internal shear resistance of the material. Secondly, the true strain rate of the matrix within the composite is much higher than the imposed strain rate on the specimen. Assuming perfectly rigid fibers, the true matrix strain rate $\dot{\epsilon}^*$ can be expressed as

$$\dot{\epsilon}^* = \frac{\dot{\epsilon}}{1 - v} \quad (30)$$

where $\dot{\epsilon}$ is the imposed strain rate and v is the volume fraction of fibers. For a 40% value, the true matrix strain rate becomes 1.67 times the imposed strain rate thereby producing higher flow stresses. It must be emphasized that the above relationship assumes homogeneous

deformation of the entire volume of the matrix within the composite, $(1 - v)$. However, even higher localized strain rates are expected due to the inhomogeneous deformation, which would result in higher flow stresses in the composites than would be expected from this simple volumetric correction. A third factor can be related to the directionality of flow in the specimens. In the case of homogeneous deformation such as that experienced by the cylindrical matrix specimen the radial strain rate

$$\dot{\epsilon}_r = \frac{1}{2} \dot{\epsilon}_z \quad (31)$$

where $\dot{\epsilon}_z$ is the axial strain rate (the imposed strain rate). In the case of the composites, however, the strain rate in the direction of fiber alignment is negligibly small and it can be assumed that plane strain conditions apply. Under these circumstances, the lateral strain rate $\dot{\epsilon}_x$ will be equal to that in the axial direction as illustrated in Fig. (105). This means that the lateral stress component in the composite specimen will be higher than the radial stress component in the matrix, which will raise the overall axial flow stress of the composite.

Peak flow stresses in the matrix and in the composites are replotted as a function of strain rate and temperature in Figs. (106 to 109) on normal and logarithmic scales respectively. The relatively low strain rate sensitivity of the composite, Figs. (108 and 109) namely 0.25 is equivalent to that of the matrix material at higher strain rates. This observation supports the view that the flow behaviour of the composite is governed by the flow of the matrix and that, the actual strain rate of the matrix is higher than the imposed one, as explained earlier. In other words, the true matrix strain rate causes flow to occur in a manner typical of higher rates of deformation, which in some cases is sufficient to reduce the strain rate sensitivity.

More generally, the difference in flow behaviour between compacts and composites can be rationalized in terms of differences between the various stress components that are thought to make up the measured stress. As discussed earlier (Section 6.4.4), with regard to the compacts strained at a constant rate, the flow stress is given by Eq. 23:

$$\sigma = \sigma_e + \sigma_i + \sigma_p$$

whereas in the case of the composites, it is proposed that

$$\sigma = \sigma_e + \sigma_i + \sigma_p + \sigma_{if} + \sigma_{ef} \quad (30)$$

where σ_{if} is an internal friction stress originating from the presence of the fibers and is constant for a given fiber distribution, fiber diameter and volume fraction. The external friction stress σ_{ef} arises due to the relative absence of lubrication at the specimen-tooling interface in the case of the composites, as opposed to the almost friction free case for the compacts. The rest of the terms are as defined earlier.

The introduction of the reinforcing fibers brings in an additional component σ_{if} originating from the shear stresses at the fiber matrix interfaces. This frictional component should depend on the amount of fiber surface area and should therefore increase with an increase in fiber volume fraction or a decrease in fiber diameter at constant volume fraction. The term σ_{if} is expected to be athermal in nature resulting in an increase in flow strength which is nearly independent of strain rate for a given temperature. The results on peak flow stress shown in Figs. (105 and 107) support this view. However, a slight decrease in σ_{if} can be observed with temperature. As deformation proceeds beyond the peak, the decrease in stress is attributable to the same microstructural changes that take place in the compacts. However, when a strain of about 0.3 is reached, the flow stress rises again suggesting that a critical stage has been reached in the deformation process, and possibly a change in deformation behaviour has occurred.

It is thought that the critical strain, which corresponds to the minimum observed stress, is related to the volume fraction of the fibers. It is also suggested that the rise in the forging load is due to the formation of fiber chains which result in hard "skeletons" within the flowing matrix when the fibers come in contact. The configuration of fibers in these chains will depend upon the volume fraction of fibers, the fiber diameter and the flow strength of the matrix. As noted previously, this latter is a

function of strain rate, temperature, and current microstructure. It is interesting to notice that the rise in the flow stress beyond the critical value is less pronounced at the slower strain rates. This could be due to the longer time available for the fibers to rearrange and slide, and to allow the loads to relax. Other factors which might influence the formation of these chains are the frictional conditions at the interface between tooling and forging blank and the geometry of the forging blanks themselves.

6.7 FRACTURE BEHAVIOUR OF COMPOSITES

As pointed out earlier (Section 5.5.1) most of the void nucleation and growth occur at the tensile poles of the fibers normal to the loading direction. Also, it has been shown that these voids are more extensive at fast strain rates and/or at low temperatures. This void formation is likely to be a function of the secondary tensile stress components developed at the horizontal poles and of the relative strengths of the matrix and interface as discussed by Ertürk⁶⁴. This secondary tensile stress is proportional to the applied vertical compressive stress^{74,85}, which is strongly dependent on the temperature and rate of deformation. The lateral tensile stress at the interface leads to a tendency for separation of the matrix from the fibers and void formation at these locations. Therefore, damage is more extensive at fast strain rates and/or at low temperatures.

Referring to Figs. (85-a) and (86-b), it can be observed that damage (void formation) is minimum in the central regions of the composite blanks adjacent to the top and bottom dies. This might be related to the effect of the lateral pressure component resulting from friction at the die/billet interfaces as discussed by Ertürk². Although friction was minimized by the use of glass lubricant, and frictional forces were reduced further by the use of small width to height ratio of composite blanks, the effect of the lateral pressure in retarding void formation is evident. This again points out to the importance of the hydrostatic stress in retarding void formation and improving the ductility of fiber reinforced composites.

The evidence of Fig. (80) indicates that the bond strength between the hafnium coating and the tungsten fibers is lower than that of the coating and the matrix material. Clearly, any increase in the bond strength at the interface and/or decrease in the flow resistance of the matrix by appropriate powder selection and subsequent processing of the composites should be beneficial to their formability. This would normally be achieved by selecting the fine mesh size powder and pressing at as low a temperature as possible to develop a fully consolidated superplastic matrix. However a conflict might arise, since it is possible that lower HIPing temperatures could also degrade the fiber-matrix bond strength, thereby increasing void nucleation during forging and lowering formability⁴⁹.

It appears that the recrystallization and grain growth in the denuded regions shown in Figs. (88 and 89) is a consequence of the dissolution of the γ' precipitate. As discussed earlier (Section 5.1.1), the presence of stable blocky γ' would normally be expected to impede recrystallization and grain growth by acting as an obstacle to dislocation and grain boundary migration.

The dissolution of γ' may be due to a local lowering of the γ' solvus temperature in the matrix adjacent to the fibers. This might be caused by diffusion of γ' stabilizing elements from the matrix and into the tungsten fibers, or diffusion of tungsten or hafnium from the fibers and into the matrix. A mechanism of this type would be expected to produce a uniform band of recrystallization around the fibers. Alternatively the dissolution of γ' may be a deformation induced instability, whereby frictional heating and γ' particle shearing in slip or deformation bands causes a reversion (re-solutioning) process to occur, as observed by Wallace and Terada¹²⁰.

It has been frequently observed that the formation of the denuded zones is associated with the existence of voids or cracks (Section 5.5.2). Based on this observation and the higher concentrations of aluminum at the surfaces of the cavities (Section 5.5.2),

it appears that the migration of aluminum from the denuded regions to react with any gases in these cavities could be responsible for the dissolution of the γ' precipitate.

However, in some cases the γ' free regions were observed in regions which do not contain voids or cracks, Fig. (88).

Irrespective of the mechanism leading to the formation of the γ' denuded and coarse grained bands, it is quite clear that these regions exhibit reduced resistance to void or crack nucleation and growth. A similar observation has been made by Wallace and Terada who observed rapid crack propagation along γ' free shear bands in powder fabricated astroloy samples, subjected to high strain rate deformation.

The flow pattern of a superplastic eutectic of lead-tin after a 58% reduction in height during plane strain compression is given in Fig. (110-a) (reproduced from ref. 121). The flow pattern of a composite material forged to 53% reduction in height is presented in Fig. (110-b). The similarity between the two cases is quite evident. Also, a similarity exists between the fracture patterns of the composites Fig. (111) and those obtained by Jain and Kobayashi¹²² for aluminum 7075-T6 pressed under plane strain condition at room temperature and those obtained by Rosen and Dow for compression of reinforced plastic composites⁶³.

It is of interest to note that the experimentally observed fracture patterns, shown schematically in Fig. (111), are in general agreement with the slip line field theory applied to plane strain deformation in rigid perfectly plastic materials. As emphasized earlier plane strain conditions can be assumed to exist during compression of the fiber reinforced composites, since there is negligible strain in the direction of fiber alignment, thereby implying that the pattern of deformation is the same for all planes normal to this direction. Fig. (112-a) shows a schematic of such a plane. Shear is initially concentrated along the diagonals and the four blocks of metal, labelled I to IV, start to move by sliding over one another as solid, undeformed bodies. Fig. (112-b) shows the slip line field for an

ideal rigid-plastic block with height to width ratio of 0.5. Similarities between the ideal patterns and the experimentally observed ones are evident.

Little damage was introduced in some isolated regions in the composite material deformed to 6% strain at 1100°C and $2.8 \times 10^{-4} \text{ s}^{-1}$, as shown in Fig. (83).

Very little damage was observed in specimens deformed below this value. This suggests that the maximum strains which can be applied to the composite material without introducing significant damage are limited to some values slightly less than 6%. This value is very small relative to the limiting strains obtained from the forming limit criterion established by Ertürk⁶⁴. Although there are differences in the composite materials, specimen geometries and forging conditions in the two studies; it appears that what was considered sound regions (in ref. 2 by the same author) contain transmatrix voids and voids at the fiber/matrix interfaces.

It should be emphasized that the present open-die forging simulation presents a particularly severe test since appreciable secondary tensile stresses are generated normal to the direction of compressive loading thereby increasing the stresses which create the major damage. In contrast, with closed-die forging the large components of hydrostatic compressive stress would tend to reduce the tensile components, thereby enhancing ductility and formability. In effect the present tests and results only provide a lower bound criterion of forming limit in the composites. Some closed-die forging simulations are necessary before the full potential of the HIP + forge approach can be assessed.

6.8 FORMING LIMITS IN COMPOSITES

It was shown in Section 6.6 that a critical strain exists in the composites where the load again increases after first falling from its peak value. This critical strain was shown to be the strain at which the fibers come into contact with each other.

Beyond the critical strain, the integrity of the fibers could be jeopardized due to the concentration of loads at points of fiber to fiber contact, thereby suggesting a

forming limit of an essentially geometric nature. Attempts to predict this critical strain can be made by considering the following hypothetical models for the flow behaviour of a simple composite material.

The models, Figs. (113 and 114), assume a regular distribution of perfectly rigid fibers, with uniform circular cross sections, embedded in a perfectly ductile matrix. It is also assumed that deformation is frictionless so that the lateral displacement of the fiber is uniform and no bulging takes place. Furthermore, the axial strain remains equal to the lateral strain i.e. the model assumes perfect plane strain deformation. Considering only the shaded area for simplicity, Figs. (113 and 114), and assuming;

d' = diameter of fiber

h_0 = initial height of the unit specimen

h_c = critical height of the unit specimen at which the fibers are in contact ($= d'$)

ϵ_c = critical strain

v = volume fraction of fiber

Considering the first model, Fig. (113)

$$\begin{aligned} v &= \frac{\pi d'^2}{2h_0^2} \\ \epsilon_{c1} &= \ln \left(\frac{h_0}{h} \right) \\ &= \ln \left[\left(\frac{\pi d'^2}{2v} \right)^{0.5} / d' \right] \\ &= \ln \left(\frac{\pi}{2v} \right)^{0.5} \end{aligned} \quad (31)$$

Similarly for the second model, Fig. (114)

$$\begin{aligned} v &= \frac{\pi d'^2}{4h_0^2} \\ \epsilon_{c2} &= \ln \left[\left(\frac{\pi d'^2}{4v} \right)^{0.5} / d' \right] \\ &= \ln \left(\frac{\pi}{4v} \right)^{0.5} \end{aligned} \quad (32)$$

These relationships have been plotted, as shown in Fig. (115). It can be seen that, for the same volume fraction of fiber, the critical strain for the first model is much higher than that of the second. For example, at $v = 0.4$, which is the volume fraction of the material investigated, the critical strain for the first model is 0.684 which is nearly twice that for the second and this ratio increases further with the increase in fiber volume fraction, as shown in Fig. (115). It is important to mention that the above models represent upper and lower limits of the critical strain for any composite with fibers having circular cross sections, and any other parallel and uniform distribution of similar fibers should lie in between these limits. The critical strains observed experimentally, Fig. (72), are less than those predicted by the present models, particularly at fast strain rates. This may be due to the non-uniform distribution of fibers and/or the inhomogeneous flow in the composite materials as will be emphasized later.

In Ertürk's work², specimens were forged up to 66% reduction in height which is equivalent of a true strain of 1.08. The critical strain for a volume fraction of 0.25, which is typical of his material, should lie between 0.92 and 0.57 (depending on the fiber arrangement) as explained in the above analysis. Therefore, it appears that the fiber breakage and the deviation from plane strain deformation observed might also be related to the fiber-fiber contact in addition to the axial tensile stress in the fibers due to shearing of the matrix at the fiber-matrix interface.

It must be pointed out that the maximum volume fraction of fibers in the above models is limited to $\frac{\pi}{4}$. For higher volume fractions of fibers, it would be necessary to assume that the fibers had other cross sectional shapes. A model of a composite material consisting of a regular pattern of hexagonally shaped fibers and a rigid plastic matrix has been assumed by Mileiko¹²³, as shown in Fig. (115-a).

Considering the composite cell, Fig. (115-b), and following an analysis similar to that used in the circular fiber models, the following critical strain relationship can be obtained

$$\epsilon_{c3} = \ln \frac{1}{\sqrt{v}} \quad (33)$$

This relationship has been plotted in Fig. (117). It should be noted that the hexagonal fiber model allows a volume fraction range between zero and 100%, while the maximum volume fraction is limited to $\frac{\pi}{4}$ for the circular fiber.

It has been frequently observed that fibers come into contact in isolated regions of the forged composite blanks, even though the imposed macroscopic strains are well below the critical strain Figs. (83, 85, 87). This may be due to the non-uniform distribution of the fibers, or alternatively the inhomogeneous deformation of the composites. In order to determine the critical conditions when cracks initiate, accurate information on the local strain distribution must be available. This could be achieved through plasticity theory solutions such as those given by finite element techniques. To illustrate this point further, the effective strain distribution of a cylindrical specimen at 20% reduction in height is shown in Fig. (118) (after Kobayashi¹²⁴). In the case of the composite, the strain in the direction of fiber alignment is negligibly small and plane strain conditions apply, Section 6.6. However, while higher strains are exhibited by the composite matrix, the general pattern of strain distribution will be similar to that of the non-reinforced material, i.e. a concentration of strain at the core and edges of the specimen. This means that while the imposed macroscopic strain is less than the critical strain, the core and edges of the specimen may be deformed beyond the critical value. As a result, severe damage is expected to occur in these regions, as shown in Fig. (119). Therefore, the volume fraction of fibers should be reduced in these regions to avoid fiber to fiber contact and the subsequent damage. In general, the localized volume fraction of fiber must satisfy the criterion

$$\epsilon_c > \epsilon$$

where, ϵ_c is the critical strain and ϵ is the local compression strain.

The existence of this critical strain points to an upper bound criterion of forming limit. However, from the results presented earlier, limits in formability might also arise from the decohesion at the fiber-matrix interfaces during deformation at low strains. Therefore, it is the flow behaviour and formation of "non-critical" microvoids in the composite material, below the critical strain, which in the end will govern its formability.

6.9 PRACTICAL IMPLICATIONS

The present results demonstrate that powder processing can be employed to provide enhanced formability in otherwise difficult to work materials. Moreover, control of the powder processing operations can lead to further gains in workability, with superplastic properties being achieved under certain conditions. It is the inherent superplasticity of certain fine mesh powders which may be exploited directly during pressing, thus negating the use of the intermediate working process normally used to develop superplasticity. This should transfer to reduced cost, increased flexibility in preform design and a wider scope of potential applications.

The advantages gained with regard to formability, when dealing with the fine mesh compacts (type B), are small but not negligible, particularly at the lower strain rates which would be contemplated for the forging of the composites. It is therefore possible that, by selecting an even finer mesh size, resistance to flow could be diminished further and ease of shaping increased.

The available evidence suggests that the coarser particles in argon atomized powders have slightly coarser grain sizes. During HIPing these particles remain undeformed because of their higher resistance to flow. Similarly, during forging, most of the deformation takes place in the regions of fine grains and these grains recrystallize to an even finer grain size. In contrast the coarser particles show little evidence of deformation and recrystallization, and hence discontinuities are created at their surface boundaries. Therefore, it might be possible to improve the ductility of the powder compacts by isolating the coarse mesh size of the powder prior to consolidation.

In an engineering part of complex shape, work hardening and/or flow softening may take place during forging. This will depend on the local strains and strain rates which are developed, and on the local microstructure which exists. Therefore, in designing a practical forging operation, it is important to control both the amount and rate of deformation introduced at particular locations, not only to avoid fracture, but also to produce the microstructure whose mechanical properties best meet the service requirement of the end product. One should also appreciate that most commercial parts, produced in the manner described above, will be heat treated prior to use. At the moment the influence of the as-forged microstructure on heat treatment response, and hence in-service properties, is not known.

Significant reductions in the flow stress of the Mar M200 nickel-base superalloy can be obtained by preforming the material at fast strain rates and/or low temperatures. In doing so, an ultrafine microstructure is developed and enhanced superplasticity is obtained in subsequent hot working processes. The higher forging loads and the increasing defects which may be produced during initial forging at relatively low temperatures and/or very fast rate of deformation defines the practical range of the forging conditions.

Cold working prior to forging can also be used to reduce forging load in the compacts. The reductions observed here have been small, due to the small amounts of cold work that were introduced. The limited cold workability of compacts precludes the practical exploitation of this precise approach, and clearly composite materials would not be amenable to this process. Nevertheless, the cold working of as-atomized powders, as in the T/P process, and the use of cold worked powders to produce composites could lead to materials with enhanced matrix superplasticity and superior workability. As pointed out in the discussion, any decrease in matrix flow strength should lead to lower forging pressures and reduced lateral tensile stress at the horizontal poles of the fibers which ultimately lead to void initiation or fracture.

Forging of composite materials is limited by the large stresses required and the numerous defects that occur during forging. The peak flow stresses for the composites are up to 4 times higher than those for the non-reinforced matrix, tested under identical working conditions. Improved ductility can be obtained by forging at high temperatures (below the γ' solvus) and at slow strain rates. The present results suggest that the maximum strains which can be applied to the present composite material, without introducing significant damage, are limited to some values slightly less than 6%. However, it should be emphasized that the present open-die forging simulation presents a particularly severe test since appreciable secondary tensile stresses are generated normal to the direction of compressive loading thereby increasing the stresses which create the major damage. In contrast, with closed-die forging the large components of hydrostatic compressive stress would tend to reduce the tensile component thereby enhancing ductility and formability. In effect the present tests and results only provide a lower bound criterion of forming limit in the composites.

It is shown that a critical strain exists in the composites where the load again increases after first falling from its peak value. This critical strain is shown to be the strain at which the fibers come into contact with each other. Beyond the critical strain, the integrity of the fibers could be jeopardized due to the concentration of loads at points of fiber-fiber contact. This critical strain is function of the volume fraction, stacking arrangement and cross section of fibers. In the present case, this critical strain is shown to be 0.34 and forming of the composites should therefore be limited to strains lower than this level.

The present results indicate that the bond strength between the hafnium coating and the tungsten fibers is lower than that of the coating and the matrix material. Clearly, any increase in the bond strength at the interface and/or decrease in the flow resistance of the matrix by appropriate powder selection and subsequent processing of the

composites should be beneficial to their formability. This would normally be achieved by selecting the fine mesh size powder and pressing at as low a temperature as possible to develop a fully consolidated superplastic matrix. However a conflict might arise, since it is possible that lower HIPing temperatures could also degrade the fiber-matrix bond strength, thereby increasing void nucleation during forging and lowering formability.

6.10 SUMMARY AND CONCLUSIONS

Equipment for isothermal isostrain rate compression testing has been designed and built around a 10,000 kg MTS servo-controlled hydraulic testing frame. The system is capable of testing up to 1200°C, and at constant strain rates between 10^{-5} and 1 s^{-1} .

It has been shown that superplastic flow can be obtained in nickel-base superalloy compacts hot-isostatically pressed below the γ' solvus. The flow and fracture behaviour can be greatly influenced by microstructure, particularly grain size, and consequently thermomechanical history is important in determining formability of these materials.

The overall level of flow stress in the fine grained superplastic material is considerably lower than that of material in the more conventional coarse grained condition. Higher strain rate sensitivities and improved ductilities are obtained in the former relative to the latter. Furthermore, in the superplastic region an enhanced degree of superplasticity can be obtained by further refinement of the grain size. However, a unique steady state grain size is produced during flow, which depends on steady state flow stress and which is independent of initial grain size. The lower the temperature and/or the faster the strain rate, the higher the stress and the finer the grain size.

It is also shown that during superplastic deformation, the flow curve can either exhibit work hardening or flow softening depending on the ratio (R_d) of initial grain size to steady-state grain size. These materials flow soften when $R_d > 1$ and work harden if $R_d < 1$.

The apparent modulus of elasticity of Mar M200 compacts is very sensitive to the rate and temperature of deformation. The strain rate sensitivities obtained are very

similar to those for the peak or steady state flow stresses, particularly in the superplastic region. Therefore it is suggested that the same processes governing the flow behaviour of compacts at higher strains are operating from the beginning of deformation. The mechanisms of this micro-plastic flow are the same as those operating at higher strains, namely grain boundary sliding and intragranular flow.

It was found that cold work by hydrostatic extrusion produced slightly lower flow stresses at elevated temperatures. This flow softening may be attributed to grain refinement and structural modifications which might be induced during exposure to the forging temperature, either before or during the initial deformation.

Ultra-fine grain sizes were developed by initially forging the compacts at high strain rates. When these materials were reformed at lower strain rates, up to 60% reductions in flow stress were obtained. Finer grain sizes and consequently higher reductions in flow stress were obtained with lower temperatures. The benefits of preforming at low temperatures, in terms of lower flow stresses in subsequent forging, are quite evident. This practice is however limited by the higher forging loads and the greater number of defects that might be produced.

It has been demonstrated that the extrusion pressures at room temperature of the Mar M200 nickel-base superalloy can be greatly influenced by metallurgical factors such as alloy composition, grain size, precipitate content and morphology. The as-HIPed fine grained material produced the highest extrusion pressures at all reduction ratios used. Lower extrusion pressures were obtained in the solution treated coarse grained material. Further reductions were obtained by aging the coarse grained material following the solution treatment, prior to extrusion.

The peak flow stresses for the composites were up to 4 times higher than those for the non-reinforced matrix, tested under identical working conditions. The strain rate sensitivity of the composites is approximately half that of the superplastic matrix. The apparent low strain rate sensitivity of the composite material has been explained by:

the change in deformation mode from axisymmetric in the compacts to plane strain in the composites, the larger effective strain rates in the flowing matrix of the composites, and the introduction of an additive internal stress component which is independent of strain rate and which originates from the presence of the rigid fibers.

A forming limit criterion of a geometric nature has been developed which accounts for fiber cross section, stacking arrangement and volume fraction. From the model considered, gross damage may occur in the composites when they are deformed beyond a critical strain, which is a function of the volume fraction, stacking arrangement and cross section of fibers. In the present case, this critical strain is shown to be 0.34 and forming of the composites should therefore be limited to strains lower than this level.

Formability is also shown to be limited at low strains by the formation of voids at the tensile poles of the fibers, normal to the loading direction. The critical parameter in this case is the strength of the fiber/matrix interface relative to that of the matrix. Means of decreasing the flow resistance of the matrix should therefore improve the formability of the composites.

6.11 SUGGESTIONS FOR FURTHER WORK

In the light of the present study, a number of suggestions for further work can be made.

- (a) Measurement of the internal stresses as a function of strain would allow the strain dependence of the effective stress to be determined. This in turn could be used to calculate the effective strain rate sensitivity and the activation energy which would provide improved understanding of the hot working mechanisms and superplasticity.
- (b) A detailed physical examination is required to determine the role that each phase plays during hot working of nickel-base superalloys.
- (c) A more systematic examination of the relationship between particle size and grain size in atomized powder would be useful, together with a study of

- methods such as splat quenching which might be used to achieve significant decreases in as-atomized grain size.
- (d) Variables which influence the microstructure of the as-atomized powders need to be examined in order to produce materials whose mechanical properties best meet the processing and service requirement of the end product.
 - (e) The influence of the as-forged microstructure on heat treatment response and mechanical properties of the compacts needs to be investigated in detail. This again may be important in developing microstructures that are best suited to the engineering application of the finished part.
 - (f) Examinations are required of the amount of cold work and/or the subsequent heat treatment needed to cause significant recrystallization to take place in powder compacts. It should be noted that while little reductions in flow stress were obtained by cold working the present material, it apparently can be used to good effect, as demonstrated by the T/P processes.
 - (g) Further work is needed to confirm the mode of deformation occurring at small strains, and to determine the relationship between apparent modulus of elasticity and the deformation variables.
 - (h) The present open-die forging simulation presents a particularly severe test. Some closed-die forging simulations are necessary before the full potential of the HIP + forge approach can be assessed.
 - (i) The present study has established the validity of a forming limit of geometric nature. Although, as has been pointed out in this work, limits in formability might also arise from decohesion at the fiber-matrix interface. A forming limit of a microscopic nature which takes into account the deformation variables such as strain rate, temperature and frictional constraints is required.

REFERENCES

1. C.P. Blankenship, "Trends in High Temperature Materials Technology for Advanced Aircraft Turbine Engines", Society of Automotive Engineers, National Aerospace Engineering and Manufacturing Meeting, (1975).
2. R. Ertürk, H.A. Kuhn and A. Lawley, "Forging of Metal-Matrix Composites an Experimental and Analytic Approach to Process Design", NTIS Tech. Rep. No. AD-774-757, Drexel Univ., Jan. (1974).
3. P.J. Mazzei, G. VanDrunen and M.J. Hakim, "Powder Fabrication of Fiber-Reinforced Superalloy Turbine Blades", Proc. of the Conference on Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications, AGARD-CP-200, (1976), p. 5c. 7.
4. P.J. Mazzei, G. VanDrunen and N. Chung, "High Temperature Artificial Composite Alloys for Industrial Gas Turbines", Proc. of the 1977 TMS-AIME Fall Meeting Chicago, Oct. (1977).
5. N. Chung and G. VanDrunen, "Tensile Behaviour of Tungsten-Superalloys Composite", Proc. of the 1977 TMS-AIME Fall Meeting Chicago, Oct. (1977).
6. R. Hewitt, J-P.A. Immarigeon, W. Wallace, A. Kandeil and M.C. de Malherbe, "Isothermal Forging at the National Aeronautical Establishment", DME/NAE Quarterly Bulletin No. 1979(1), National Research Council of Canada, Ottawa, April (1979).
7. R.L. Athey and J.B. Moore, "Progress Report on the GatorizingTM Forging Process", SAE paper 751047, Society for Automotive Engineers, Nov. (1975).
8. Anon, "Isothermal Forging Makes a First Good Impression", Iron Age, Oct. (1977), p. 43.
9. S.Z. Figlin, V.V. Boyutsov, A.V. Bakharev and Yu. A. Kalpin, "Advantages of Hot Isothermal Forging", Light Metal Age, Oct. (1973), p. 13.
10. C.H. Hamilton and A.W. Stacher, "Superplastic Forming of Ti-6Al-4V", Metal Progress, Vol. 109, No. 3, March 1976, p. 34.
11. M.M. Allen, "Iso-Forging of Powder Metallurgy Superalloys for Advanced Turbine Engine Applications", AGARD Conference Proceedings No. 200, April (1976), p. P5.
12. J.N. Fleck and L.P. Clark, "Advancements in Titanium Powder Processing", SAMPE Quarterly, Oct. (1976), p. 10.

13. J.P.A. Immarigeon, G. VanDrunen and W. Wallace, "The Hot Working Behaviour of Mar M200 Superalloy Compacts"; Superalloys, Metallurgy and Manufacture, Third International Symposium on Superalloys, Seven Springs, Pa., Claitor's Pub. Div., Baton Rouge, (1976), p. 463.
14. L.P. Clark, "Powder Metallurgy Production Processes", AGARD Report No. 627, March (1975).
15. J.S. Benjamin and J.M. Larson, "Superalloys by Powder Metallurgy Techniques", AIAA Aircraft Systems and Technology Meeting, Texas, Sept. (1976).
16. G.H. Gessinger and M.J. Bomford, "Powder Metallurgy of Superalloys", International Metallurgical Reviews, Vol. 19, (1974), p. 51.
17. W. Wallace, J.P.A. Immarigeon, J.M. Trenouth and P.D. Powell, "Control of Grain Structure During Superalloy Powder Processing", Proceedings of the Conference on Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications, AGARD-CP-200, (1976), pp. P.9-1.
18. C.A. Morris and J.W. Smythe, "Comparison of Astroloy Powder Consolidation Processes Forging and Testing of Astroloy Powder Billet", in Superalloys-Processing, Proc. 2nd. Int. Conf., MCIC-72-10, Met. and Cer. Inf. Centre, Battelle Columbus Lab., Ohio (1972), p. Y1.
19. C.H. Symonds and F.A. Thompson, "Nickel Superalloy Powder Production and Fabrication to Turbine Discs", Proceedings of the Conference on Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications, AGARD-CP-200, (1976), p. 3.
20. L.J. Fielder, "Advancements in Powder Production and Consolidation", Proceedings of the Conference on Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications, AGARD-CP-200, (1976), p. 4B.
21. A.J. Deridder, A.F. Hayes, R.W. Koch, J.F. Radowich and R. English, "Deformation Processing of Superalloy Hot Isostatic Press Deforms", Presented at the 106th AIME Annual Meeting, March (1977).
22. S.H. Reichman "Low Cost P/M Superalloy Applications in Turbines", The International Journal of Powder Metallurgy & Powder Technology, Vol. 11, No. 4, Oct. (1975).
23. R.H. Johnson, "Superplasticity", Metallurgical Review No. 146, (1970), p. 115.
24. S.H. Reichman, B.W. Castledine, and J.W. Smythe, "Powder Metal Nickel Superalloys May Be Formed Superplastically", SAE Journal, Vol. 78, No. 4, April (1970), p. 59.
25. R.H. Johnson, "Superplasticity Means Low-Force Metal Forming", Canadian Machinery and Metalworking, Feb. (1970), p. 54.
26. A.S. Krausz, "The Forming of Superplastic Alloys", Journal of Metals, April (1973), p. 24.

27. W. Johnson, "Review of Metal Working Plasticity", *Metallurgia and Metal Forming*, April (1972), p. 128.
28. H.W. Hayden, R.C. Gibson and J.H. Brophy, "The Relationship Between Superplasticity and Formability", in *Metal Forming Interrelation Between Theory and Practice*, ed: by A.L. Hoffmann, Plenum Press, N.Y. (1971), p. 475.
29. T.H. Alden in *Treatise on Materials Science and Technology, Plastic Deformation of Materials*, Vol. 6, ed. by R.J. Arsenault, Academic Press, New York (1975), p. 225.
30. J. Gittus in *Creep, Viscoelasticity and Creep Fracture in Solids*, A Halsted Press Book, John Wiley and Sons, New York (1975), p. 509.
31. J.W. Edington, "Physical Metallurgy of Superplasticity", *Metals Technology*, March (1976).
32. T.D. Kelso, "The Advent of Superplastic Superalloys", *Manufacturing Engineering & Management*, May (1970), p. 55.
33. H.W. Hayden, S. Floreen, and P.D. Goodell, "The Deformation Mechanisms of Superplasticity", *Metallurgical Transactions*, Vol. 3, (1972), p. 833.
34. C.M. Sellars and W.J. McG. Tegart, "Hot Workability", *International Metallurgical Reviews*, Vol. 17, (1972), p. 1.
35. H.W. Hayden and J.H. Brophy, "The Interrelation of Grain Size and Superplastic Deformation in Ni-Cr-Fe Alloys", *Transactions of the ASM*, Vol. 61, (1968), p. 542.
36. T.G. Langdon and F.A. Mohamed, "The Mechanical Characteristics of Superplastic Deformation", Presented at the IV Interamerican Conference on Materials Technology, Venezuela, June-July, (1975).
37. N. Paton, "A Constitutive Equation for High Temperature Flow", *Transactions of the ASME*, Paper No. 75-Mat-13, presented at the Conference on Micromechanical Modelling of Flow and Fracture, N.Y., June (1975).
38. M. Suery and B. Baudalet, "Theoretical and Experimental Constitutive Equations of Superplastic Behaviour: Discussion", *Journal of Materials Science*, Vol. 10, (1975), p. 1022.
39. W.J. Evans and G.F. Harrison, "The Development of a Universal Equation for Secondary Creep Rates in Pure Metals and Engineering Alloys", *Metals Science*, Sept. (1976), p. 307.
40. M.F. Ashby and R.A. Verrall, "Diffusion-Accommodated Flow and Superplasticity", *ACTA Metallurgica*, Vol. 21, Feb. (1973), p. 149.
41. J.W. Edington, K.N. Melton and C.P. Cutler, "Superplasticity", *Progress in Materials Science*, Vol. 21, No. 2, (1976), p. 61.

42. R.C. Giffkins, "Grain-Boundary Sliding and its Accommodation During Creep and Superplasticity", *Metallurgical Transactions A*, Vol. 7A, Aug. (1976), p. 1225.
43. H.J. McQueen and J.J. Jonas in *Metal Forming: Interrelation Between Theory and Practice*, ed. by A.L. Hoffmann, Plenum Press, N.Y. (1971), p. 393.
44. N.J. Parratt, "Some Features and Problems of Metal Composites", *Proceedings of Int. Conf. on Composite Materials*, Boston, April (1975), Pub. AIME, p. 395.
45. D.W. Petrasek, R.A. Signorelli, and J.W. Weeton, "Refractory-Metal-Fiber-Nickel-Base Alloy Composites for Use at High Temperatures", NASA TN D-4787, Sept. (1968).
46. S.A. Golovanenko, B.A. Klypin, A.M. Maslov and S.B. Maslenkov, "Interaction Between the Filaments and the Matrix in Nickel Alloys Reinforced with Tungsten Filaments", *Fiz. Khim. Obrab. Mater.*, July-Aug. (1975), Vol. 4, p. 144 (in Russian).
47. O. Ochiai, K. Shimomura, M. Mizuhara and Y. Murakami, "Effects of Interfacial Reaction on Deformation and Fracture Behaviour of Tungsten Fiber-Nickel Matrix Composites", *Trans. J.I.M.*, Vol. 16, (1975), p. 463.
48. R.A. Signorelli, "Metal Matrix Composites for Aircraft Propulsion Systems", *Proceedings of Int. Conf. on Composite Materials*, Boston, April (1975), Pub. AIME, p. 411.
49. J.W. Weeton, "Fiber Reinforced Superalloys, Ceramics, and Refractory Metals and Directionally Solidified Eutectics", *Composites: State of the Art*, *Proceedings of Sessions of the Metallurgical Society of AIME*, Detroit, Fall (1971), p. 258.
50. J.F. Dolowy, "Fabrication and Processing Mechanisms Active in Aluminum-Boron Composites", *Metal-Matrix Composites — Symposium of the Metallurgical Society of AIME*, May (1969), p. 7.
51. W.D. Brentnall and I.J. Toth, "Fabrication of Tungsten Wire Reinforced Nickel-Base Alloy Composites", NASA CR-134664, Oct. (1974).
52. B.V. Aleksandrov et al., "Hot Forging of a Heat-Resistant Fibrous Composite Material", *Kuznechno-Shtampovnoe Proizvodstvo*, No. 6, (1975), p. 18.
53. I.J. Toth, W.D. Brentnall, and G.D. Menke, "Making a Product from Composites", *Journal of Metals*, Oct. (1972), p. 37.
54. Athey et al., U.S. Pat. No. 3, 711, 936, Jan. (1973).
55. R.M. Anderson and P.D. Caton, "Production of Fibre-Strengthened Material by Mechanical Working of a Duplex Alloy", *Journal of the Institute of Metals*, Vol. (96), (1968), p. 108.
56. G.I. Friedman, "Fiber Strengthened Superalloys", NTIS U.S. Department of Commerce, January (1974).

57. J. Greenspan and F.J. Rizzitano, "Development of Superalloy Matrix-Refractory Metal Fiber Composites by Coextrusion of Blended Powders", National Technical Information Service (NTIS), Sept. (1972).
58. T.A. Collings, "Transverse Compressive Behaviour of Unidirectional Carbon Fiber Reinforced Plastics", Composites, Vol. 5, No. 3, May (1974), p. 108.
59. A.R. Rosenfield, "Criteria for Ductile Fracture of Two-phase Alloys", Metallurgical Reviews, No. 121, (1968), p. 29.
60. F.A. McClintock, "On the Mechanics of Fracture from Inclusions", Ductility, ASM, Metals Park, Ohio, (1968), p. 255.
61. B.H. Jones, "Strength and Fracture Characteristics of Filamentary Composites", ASME Paper No. 70-DE-31, May (1970).
62. B.W. Rosen, "Thermomechanical Properties of Fibrous Composites", Proceedings of the Royal Society of London, A-319, Oct. (1970), p. 79.
63. B.W. Rosen and N.F. Dow, "Mechanics of Failure of Fibrous Composites", Fracture, An Advanced Treatise, edited by Harold Liebowitz, Vol. 7, Academic Press, New York, (1972), p. 611.
64. T. Ertürk, H.A. Kuhn, and A. Lawley, "Forging of Metal-Matrix Composites — Forming Limit Criteria", Metallurgical Transactions, Vol. 5, Nov. (1974), p. 2295.
65. G.A. Cooper, "The Structure and Mechanical Properties of Composite Materials", Rev. Physics Technol., Vol. 2, (1971), p. 49.
66. M.A. McGuire and B. Harris, "Crack Propagation in Metal-Matrix/Metal-Fiber Composites: I. Strength and Toughness", J. Phys. D: Appl. Phys., Vol. 7, (1974), p. 1788.
67. P.F. Thomason, "The Influence of Matrix/Particle Cohesive Bond Strength on the Internal Necking Mechanism of Ductile Fracture", Metal Science Journal, Vol. 5, (1971), p. 64.
68. K.E. Puttick, "Ductile Fracture in Metals", Phil. Mag. Vol. 4, (1959), p. 964.
69. L.E. French and P.F. Weinrich, "The Effects of Hydrostatic Pressure on the Mechanism of Tensile Fracture of Aluminum", Metallurgical Trans. A, Vol. 6A, June (1975), p. 1165.
70. G.Y. Chin, W.F. Hosford and W.A. Backofen, "Ductile Fracture of Aluminum", Transactions of the Metallurgical Society of AIME, Vol. 230, April (1964), p. 437.
71. M.F. Ashby, "The Theory of the Critical Shear Stress and Work Hardening of Dispersion-Hardened Crystals", Proc. of Metallurgical Society Conference entitled "Oxide Dispersion Strengthening" Vol. 47, N.Y., (1966), p. 143.

72. M.F. Ashby, "Work Hardening of Dispersion-hardened Crystals", *Phil. Mag.*, Vol. 14, (1966), p. 1157.
73. J. Gurland and J. Plateau, "The Mechanism of Ductile Rupture of Metals Containing Inclusions", *Transactions of the ASM*, Vol. 56, (1963).
74. F.A. McClintock, "On the Mechanics of Fracture From Inclusions", *Ductility*, ASM, Metals Park, Ohio, (1968), p. 255.
75. H. Lilholt, "Metal Matrix Composites", *Proceedings of the Fourth Nordic High Temperature Symposium, Nortemps - 75*, Vol. 11, (1975), p. 1.
76. P.W. Bridgman, "Studies in Large Plastic Flow and Fracture", Cambridge, Mass. (Harvard Press), (1964).
77. L.E. French and P.F. Weinrich, "The Influence of Hydrostatic Pressure on the Tensile Deformation and Fracture of Copper", *Metallurgical Transactions A*, Vol. 6A, April (1975), p. 785.
78. L.F. Coffin and H.C. Rogers, "Influence of Pressure on the Structural Damage in Metal Forming Processes", *Transactions of the ASM*, Vol. 60, (1967), p. 672.
79. J. Gurland, "Fracture of Metal-Matrix Particulate Composites", in *Composite Materials*, Vol. 5, *Fracture and Fatigue* ed. by L.J. Broutman, Academic Press, (1974), p. 45.
80. S.P. Keeler and W.A. Backofen, "Plastic Instability and Fracture in Sheets Stretched over Rigid Punches", *Trans. ASM*, Vol. 56, (1963), p. 25.
81. H.A. Kuhn, P.W. Lee and T. Ertürk, "A Fracture Criterion for Cold Forming", *Journal of Eng. Mat. and Technology*, *Trans. ASME*, Oct. (1973), p. 213.
82. P.W. Lee and K.A. Kuhn, "Fracture in Cold Upset Forging - A Criterion and Model", *Met. Trans.*, Vol. 4, (1973), p. 969.
83. Z. Marciniak and K. Kuczynski, "Limit Strains in the Process of Stretch - Forming Sheet Metal", *Int. J. Mech. Sci.*, Vol. 9, (1967), p. 609.
84. R.L. Hewitt, "A Theoretical Forming Limit for Forging of Rate-Sensitive Non-Work-Hardening Material", *DME/NAE Quarterly Bulletin* No. 1978(1), April (1978), p. 1.
85. J.N. Goodier, "Concentration of Stress Around Spherical and Cylindrical Inclusions and Flaws", *ASME Trans.*, Vol. 55, (1933), p. 39.
86. J-P.A. Immarigeon and T. Terada, "A High Temperature Compression Train for the Thermomechanical Treatment of Superalloys", *LTR-ST-785*, NRC, OTTAWA, Aug. (1975).
87. J-P.A. Immarigeon, P. Adams and R. Kulchyski, "Analog Function Generator For Constant True Strain Rate During Uniaxial Compression With an MTS Testing Machine", *LTR-ST-786*, NRC, OTTAWA, Aug. (1975).

88. R.W. Gilbert and J.V. Houston, "TZM-New Alloy Broadens Applications for Molybdenum", Metal Progress, Nov. (1962).
89. "High Frequency Heating", Review, Vol. 1, No. 10, published by LEPEL High Frequency Laboratories Inc.
90. C.I. Perratt, "A NAE/NRC Structure & Materials Laboratory Real-Time Data Acquisition System (to be published).
91. R.B. Kulchyski, "Application of the Mobile Real-Time Data Acquisition System and the Programmable Remote Job Entry System for Digital Measurement-Analysis of Load and Displacement Signals in Uniaxial Compression Experiments", ST-222, NRC, Ottawa, Nov. (1976).
92. W. Johnson and P.B. Mellor in Engineering Plasticity, Van Nostrand Reinhold Company, London, (1975), p. 10.
93. J-P.A. Immarigeon, "Hot Compression of Armco Iron and Silicon Steel Near the Curie Temperature", Ph.D. Thesis, McGill Univ., (1974).
94. A.T. English and W.A. Backofen, "Recrystallization in Hot-Worked Silicon-Iron", Trans. TMS AIME, Vol. 230, April (1964), p. 396.
95. J.F. Alder and V.A. Phillips, "The Effect of Strain Rate and Temperature on the Resistance of Aluminium, Copper, and Steel to Compression", J. Inst. Met., Vol. 83, (1954-55), p. 80.
96. A. Phillips, V. Kerlins, and B.V. Whiteson, "Electron Fractography Handbook", Tech. Report ML-TDR-64-416, Air Force Materials Laboratory, Jan. (1965).
97. J. Goldak, Carleton Univ., Private Communication.
98. R.L. Hewitt, W. Wallace and M.C. deMalherbe, "An Apparatus for Isostatic Compaction and Hydrostatic Extrusion up to 16 Kbars", Canadian Metallurgical Quarterly, Vol. 14, No. 1, (1975), p. 63.
99. J.C. UY, C.J. Nolan, and T.E. Davidson, "The Hydrostatic Extrusion of Nickel-Base Superalloys at Room Temperature", Trans. ASM, Vol. 60, (1967), p. 693.
100. C.P. Blankenship and C.A. Gyorgak, "Thermomechanical Processing of the Nickel-Base Alloy U-700", NASA TND 6418, Lewis Research Centre, Cleveland, Ohio.
101. R.S. Cremisio and H.J. McQueen, "Some Observations of Hot-Working Behaviour of Superalloys According to Various Types of Hot Workability Tests", Proc. 2nd Int. Conf. on Superalloys - Processing, MCIC Report, Sept. (1972), p. G1.
102. W. Wallace, J-P.A. Immarigeon, J.M. Trenouth and B.D. Powell, "Control of Grain Structure During Superalloy Powder Processing", LTR-ST-812, National Research Council of Canada, Nov. (1975).

103. H.L.I.D. Pugh and K. Ashcroft, "Hydrostatic (Ramless) Extrusion of Metals by Liquid Pressure", Symposium of the Physics and Chemistry of High Pressures", London Society of Chemical Industries, (1962), p. 163.
104. H.L.I.D. Pugh, Bulleid Memorial Lectures, Univ. of Nottingham, (1965).
105. B.W.H. Lowe and D. Goold, "An Account of Some Recent Experimental Work on the Hydrostatic Extrusion of Non-Ferrous Metals", Proc. Inst. Mech. Eng., Vol. 182 (1967-68), Part 3C, p. 197.
106. H.J. McQueen and J.J. Jonas, "Recovery and Recrystallization During High Temperature Deformation", in: Plastic Deformation of Materials, ed. R.J. Arsenault, Treatise on Materials Science and Tech., 6, Academic Press, (1975), p. 393.
107. R. Lagneborg, "Dislocation Mechanisms in Creep", International Metallurgical Reviews, Vol. 17, (1972), p. 130.
108. M.J. Luton and C.M. Sellars, "Dynamic Recrystallization in Nickel and Nickel-Iron Alloys During High Temperature Deformation", ACTA Metallurgica, Vol. 17, Aug. (1969), p. 1033.
109. J-P.A. Immarigeon and P. Floyd, "Structural Instabilities during Superplastic Forging of Nickel-Base Superalloy Compacts", To be published.
110. W. Wallace, Unpublished Results, National Aeronautical Establishment, NRC.
111. S. Fulop and H.J. McQueen, "Mechanisms of Deformation in the Hot Working of Nickel-Base Superalloys", Proc. of the 2nd Int. Conf. on Superalloys — Processing, MCIC Report, Sept. (1972), p. H-1.
112. "High Temperature High Strength Nickel Base Alloys", The International Nickel Company, N.Y. 10004, (1978).
113. E.W. Hart, "A Phenomenological Theory for Plastic Deformation of Polycrystalline Metals", ACTA Metallurgica, Vol. 18, June (1970), p. 599.
114. D. Lee and E.W. Hart, "Stress Relaxation and Mechanical Behaviour of Metals", Metallurgical Trans., Vol. 2, April (1971), p. 1245.
115. H.C. Rogers, "Prediction and Effects of Material Damage During Deformation Processing", Metal Forming: Interrelation Between Theory and Practice, ed. by: A.L. Hoffmanner, Plenum Press, N.Y.-London, (1971), p. 453.
116. M.J. Luton and W.J. McG. Tegart, "Fracture Behaviour of Nickel and Nickel-Iron Alloys Under Hot-Working Conditions", Metal Sciences J., Vol. 3, (1969), p. 142.
117. L.N. Moskowitz, R.M. Pelloux and N.J. Grant, "Properties of IN-100 Processed by Powder Metallurgy", Proc. of the 2nd Int. Conf. on Superalloys — Processing, MCIC Report, Sept. (1972), p. Z1.

118. D.C. Drucker, "A Continuum Approach to the Fracture of Metals", in Fracture of Solids, ed. by D.C. Drucker and J.J. Gilman, Inter-Science Publishers, N.Y.-London, (1963), p. 3.
119. G. VanDrunen, Westinghouse Canada, Private Communication.
120. W. Wallace and T. Terada, "The Nature of "White Streaks" observed in a Nickel-Base Superalloy", Metallurgical Transactions, Vol. 4, Oct. (1973), p. 2481.
121. A.H. Shabaik, "Analysis of Forming Processes: Experimental and Numerical Methods", Presented at the Winter Annual Meeting of the ASME, AMD, Vol. 28, Dec. (1978).
122. S.C. Jain and Shiro Kobayashi, "Deformation and Fracture of an Aluminum Alloy in Plane-Strain Side-Pressing", in: Advances in Machine Tool Design and Research, Proc. of the Int. MTDR Conf., Vol. B, Sept. (1970), p. 1137.
123. S.T. Mileiko, "Steady State Creep of a Composite Material with Short Fibers", J. Mat. Sci., Vol. 5, (1970), p. 254.
124. S. Kobayashi, "Theories and Experiments on Friction, Deformation, and Fracture in Plastic Deformation Processes", in: Metal Forming Interrelation Between Theory and Practice, ed. by A.L. Hoffmann, Plenum Press, N.Y.-London, (1971), p. 325.
125. B.M. Watts and M.J. Stowell, "The Variation in Flow Stress and Microstructure during Superplastic Deformation of the Al-Cu Eutectic", J. Mat. Sci., Vol. 6, (1971), p. 228.
126. M.J. Luton, J-P. Immarigeon and J.J. Jonas, "Constant True Strain Rate Apparatus for Use With Instron Testing Machine", J. Physics E: Sci. Instruments, Vol. 7, (1974), p. 862.

TABLE 1

CHEMICAL ANALYSIS OF Mar M200 MATRIX ALLOY POWDER (w/o)

Ni	C	Co	Cr	Al	Ti	Nb	W	Zr	B	N ₂	O ₂	S
Bal.	0.022	11.08	9.80	5.23	2.10	1.02	12.82	.047	.018	.0014	.0060	.006

TABLE 2

PARTICLE SIZE DISTRIBUTION OF Mar M200 MATRIX ALLOY POWDER

Mesh	w/o
+80	0.3
+100	4.3
+120	5.5
+140	6.6
+170	9.3
+230	15.6
+270	7.8
+325	8.8
+500	23.2
-500	18.6

TABLE 3

ERRORS IN STRAIN RATE DUE TO DC INSTABILITIES IN THE SERVO
CONTROLLER INCLUDING THE FUNCTION GENERATOR

Prog. input (volts)	Drift [†] mm/sec	Absolute error in strain rate* s ⁻¹	% error in strain rate at strain rates of ^{††}					
			10 ⁻⁵	s ⁻¹	10 ⁻⁴	s ⁻¹	10 ⁻³	s ⁻¹
9.5	19 10 ⁻⁶	20 10 ⁻⁷	20		2		0.2	
7	6 10 ⁻⁶	8.6 10 ⁻⁷	8		0.8		0.08	
4	1.2 10 ⁻⁶	3 10 ⁻⁷	3		0.3		0.03	
2	10 10 ⁻⁶	50 10 ⁻⁷	50		5		0.5	

† measured over a 2000 s time span using an infinite time constant for the generator

* for a 10 mm high specimen as used in the present investigation

†† for stroke range 3 (10v $\equiv$ 15mm).

TABLE 4

σ PEAK AT HIGH $\dot{\epsilon}$ / σ PEAK AT LOW $\dot{\epsilon}$ FOR DIFFERENT TEMPERATURES

Test Temp. °C	950	1000	1050	1100	1150	1180
Ratio	4.63	8.57	15.44	21.61	24.3	26.96

TABLE 5

STRAIN RATE SENSITIVITY EXPONENT $m = (\partial \ln \sigma / \partial \ln \dot{\epsilon})_{T, \text{Structure}}$ AS A
FUNCTION OF TEMPERATURE

Temp. °C	m_1 (superplastic region)	m_2 (conventional region)
950	0.43	0.14
1000	0.55	0.16
1050	0.57	0.22
1100	0.58	0.28
1150	0.58	0.31
1180	0.58	0.32

TABLE 6

EFFECT OF PRESTRAIN ON DECREASE IN PEAK FLOW STRESS RELATIVE
TO AS-HIPed MATERIAL ($\dot{\epsilon}_1 = 0.88 \times 10^{-1}$, $\dot{\epsilon}_2 = 1.4 \times 10^{-3}$)

Temperature, °C	Prestrain, %	σ_{peak} , MN/m ²	d, MN/m ²	% reduction in σ_{peak}
1100	7	45	14	24
	23	32	27	46
	43	25	34	58
	61	22	40	62
1150	9	30	3	9
	26	26	7	21
	29	23	10	30
	73	13	20	61

TABLE 7

EFFECT OF STRAIN RATE ON DECREASE IN PEAK FLOW STRESS RELATIVE
TO AS-HIPed MATERIAL ($\dot{\epsilon}_1 = 0.88 \times 10^{-1}, s^{-1}$ & $T = 1100^\circ C$)

Strain rate $\dot{\epsilon}_2, s^{-1}$	$\sigma_{peak}, MN/m^2$	d, MN/m ²	% reduction in σ_{peak}
1.03×10^{-2}	61	78	62
1.36×10^{-3}	22	40	64
3.04×10^{-4}	10	16	62
9.60×10^{-5}	6	8	55

TABLE 8

EFFECT OF STRAIN RATE ON DECREASE IN PEAK FLOW STRESS RELATIVE
TO AS-HIPed MATERIAL ($\dot{\epsilon}_1 = 2.1 \times 10^{-1}, s^{-1}$ & $T = 1100^\circ C$)

Strain rate $\dot{\epsilon}_2, s^{-1}$	$\sigma_{peak}, MN/m^2$	d, MN/m ²	% reduction in σ_{peak}
1.03×10^{-2}	55.5	103.5	65
1.36×10^{-3}	22.5	39.5	64
3.04×10^{-4}	10.7	15.3	59
9.60×10^{-5}	5.7	7.3	56

APPENDIX I

POTENTIAL COMPOSITE FABRICATION METHODS

General Method	First Step	Consolidation
1. Powder Metallurgy	Pack fibers in matrix or 'Slip cast matrix about fibers' or Use fugitive binder to hold fibers together	Sinter, hot press, or hot isostatic press
2. Foil Metallurgy	Use fugitive binder or "glue" to bond fibers to foil or Press foils (flat or grooved) about fibers Flash bond or continuous bond (roll) foils about fibers	Diffusion bond in hot press or hot isostatic press braze
3. Casting	Cast entire part Cast matrix about fibers — individual unit or continuous tapes	None — when casting entire part. Continuous tapes — hot press or hot isostatic press
4. Electro-deposit	Electroplate or form shapes or tapes electrophoresis	Hot press or hot isostatically press
5. Vapor deposit	CVD Ion plate Vapor-ion beam	Hot press or hot isostatic press
6. Metal spray	Plasma spray Molten metal spray	Hot press or hot isostatic press

APPENDIX II

BASIC REQUIREMENTS FOR A CONSTANT TRUE STRAIN RATE

Under conditions of axisymmetric compression, the true incremental strain in a cylindrical specimen is given by the relation:

$$d\epsilon = dh/h \quad (1)$$

where h is the instantaneous height of the specimen. The true strain rate can, therefore, be written as

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{1}{h} \frac{dh}{dt} \quad (2)$$

If $\dot{\epsilon}_c$ is the required constant strain rate, Eq. 2 can be rearranged to read:

$$\frac{dh}{dt} - \dot{\epsilon}_c h = 0 \quad (3)$$

Thus, to achieve a constant true strain rate during testing requires simply that the conditions prescribed by the above differential equation are met or, solving for h , that:

$$h = h_0 e^{-\dot{\epsilon}_c t} \quad (4)$$

Here, h_0 is the initial height of the cylinder.

It should be noted that when these conditions are met, the ram velocity of the testing machine remains directly proportional to the height of the specimen as suggested by Eq. 2, i.e.:

$$V_{RAM} = \frac{dh}{dt} = \dot{\epsilon}_c h \quad (5)$$

A number of devices are described in the literature which provide for a constant strain rate with electromechanical testing machines^{125,126}. Modern, closed loop, servo controlled hydraulic machines, like that under consideration*, generally have a program input which, given the proper function generator, can be used to drive the ram, under stroke control, so as to satisfy the requirements of Eq. 4. The similarity between the form of Eq. 4 and that for the discharge of a capacitor through a resistive load i.e.

* MTS 810 test system; 10,000 kg load frame

$$V = V_0 e^{-\frac{t}{RC}} \quad (6)$$

suggests a simple and direct means of obtaining a constant true strain rate with the present testing machine: a decaying analog exponential signal, generated by a RC circuit can be fed to the summing junction of the servo controller with the result that the ram motion will be exponential and have its position described by Eq. 4. Furthermore, the strain rate can easily be adjusted as will by varying the time constant of the RC circuit, since from Eqs. 4 and 6:

$$\dot{\epsilon}_c = 1/RC \quad (7)$$

APPENDIX III STRAIN RATE ADJUSTMENTS

Rotary Switch Position	Resistance	MΩ	Strain Rate* $\dot{\epsilon}$, s ⁻¹			
			actual		nominal	
0	0.094		9.84	10 ⁻¹	1	10 ⁻¹
1	0.296		3.13	10 ⁻¹	3	10 ⁻¹
2	1.052		8.80	10 ⁻²	1	10 ⁻¹
3	2.923		3.17	10 ⁻²	3	10 ⁻²
4	9.210		1.00	10 ⁻²	1	10 ⁻²
5	2.995	10	3.09	10 ⁻³	3	10 ⁻³
6	1.000	10 ²	9.26	10 ⁻⁴	1	10 ⁻³
7	3.290	10 ²	2.81	10 ⁻⁴	3	10 ⁻⁴
8	1.000	10 ³	9.25	10 ⁻⁵	1	10 ⁻⁴
9	3.648	10 ³	2.54	10 ⁻⁵	2.5	10 ⁻⁵

* calculated from $\dot{\epsilon} = \frac{1}{RC}$ with $c = 10.8 \mu f$.

APPENDIX IV

COMPOSITE PLY PROCESS

Quantities of the powder are sent to a commercial supplier who combines them with proprietary mixtures of organic binders to form a slurry. The slurry is dispensed through an orifice from a mixing container and onto a moving sheet of polyethylene. The required tape thickness is achieved by an adjustable plate which removes excess slurry as the slurry passes under the plate. The sized tape is oven dried and wound onto dispensing rolls with protective waxed paper interleaving. For our purposes, a tape width of 200 mm and a thickness of 0.25 mm are provided.

The tape and coated wire are combined to form an airfoil shape by the following steps.

1. A silicone rubber mold is formed with a cavity shape corresponding to the desired airfoil, with an allowance for consolidation shrinkage.
2. A low melting point bismuth alloy is cast into the mold to form an airfoil replicate that can be deformed and easily machined.
3. The casting is flattened, without distortion, to produce a simplified shape.
4. The flattened casting is mounted on a suitable base and successive milling machine cuts are made. The thickness of material removed is dependent on the desired fibre volume fraction, the known matrix tape shrinkage factor, and the measured thickness of the pressed composite ply, as described later.
5. The machined surface is photographed after each cut to provide a trace for each ply pattern. Suitable indexing marks are provided so that each ply can be placed relative to another in the proper position as they are stacked.
6. Duplicate shapes of matrix alloy tape are cut for each ply.

7. The required fibres for each ply are positioned in a heated die which is grooved to provide the desired fibre spacing.
8. Matrix alloy tape is positioned on one side of the aligned fibres and pressed so that the fibres adhere to the tape. The tape and fibres are then reversed and another tape layer is placed over the fibres to form the composite ply.
9. Both surfaces of each ply are covered with a layer of polyethylene sheet and pressed to allow the matrix tape to flow between each fibre.
10. After removal of the polyethylene sheets, the plies are ready for stacking, and forming back to the original airfoil shape. The stacking is performed on a lacquer-coated, split ceramic mold having the desired airfoil shape as a cavity. It is at this point that cross-plyies could be incorporated for off-axis strengthening. Sufficient layers of unreinforced tape are wrapped around. The composite bundle to ensure at least 0.50 mm of uniform matrix alloy cladding so that no fibres are exposed after consolidation.
11. The shaped ply bundle is inserted into the container that is used for HIP-consolidation. The container is airfoil shaped and is sealed at only one end to make possible the insertion of the bundle and to maximize gas exit area for binder outgassing.
12. The container and its contents are placed in a retort and outgassed in hydrogen to remove all traces of the binders present in the matrix alloy tape. A typical outgassing cycle consists of initially evacuating the retort and backfilling with hydrogen before heating commences. A maximum heat-up rate of 100°C per hour is employed to 300°C. At this temperature, the retort is evacuated and back filled with hydrogen again. The bundle is subsequently heated to 650°C, after which the furnace is cooled to room temperature to complete the cycle.

13. Following removal from the retort, the root section shell is welded to the container. This part of the container also includes the evacuation tube that is required for pre-HIP processing. Dry matrix powder is added to the container through the evacuation tube to form the root section.
14. At this point, the container is ready for evacuation and sealing.

APPENDIX V

ERRORS IN PLASTIC STRAIN RATES

During uniaxial compression the total deformation is made up of two components: the elastic component and the plastic component so that:

$$\epsilon = \epsilon_e + \epsilon_p \quad (1)$$

The amount of elastic deformation during flow is a function of the modulus of elasticity of the material and of the instantaneous developed stress:

$$\epsilon_e = \frac{1}{E} \frac{L}{A} \quad (2)$$

Here A is the instantaneous cross sectional area of the cylindrical specimen undergoing plastic flow. The rate of change of the elastic portion of the deformation is:

$$\frac{d\epsilon_e}{dt} = \dot{\epsilon}_e = \frac{1}{E} \left[\frac{1}{A} \frac{dL}{dt} - \frac{L}{A^2} \frac{dA}{dt} \right] \quad (3)$$

Assuming that no volume change takes place during the deformation, i.e. $Ah = V = \text{constant}$, where h is the instantaneous height of the specimen, and from the definition of total strain rate:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{1}{h} \frac{dh}{dt} \quad (4)$$

the terms of Eq. 3 can be rearranged to read:

$$\dot{\epsilon}_e = \frac{h}{EV} \left[\frac{dL}{dt} + L\dot{\epsilon} \right] \quad (5)$$

From Eq. 1, it is clear that the total strain rate is made up of the elastic and the plastic strain rate components so that

$$\dot{\epsilon}_p = \dot{\epsilon} - \dot{\epsilon}_e \quad (6)$$

Combining Eqs. 5 and 6,

$$\dot{\epsilon}_p = \dot{\epsilon} \left(1 - \frac{hL}{EV} \right) - \frac{h}{EV} \frac{dL}{dt} \quad (7)$$

or

$$\dot{\epsilon}_p \sim \dot{\epsilon} - \frac{h}{EV} \frac{dL}{dt}$$

since the term $\frac{hL}{EV}$ is negligible with respect to 1*. Thus, the difference between the plastic strain rate and the nominal strain rate imposed by the hydraulic testing machine is

$$\Delta \dot{\epsilon} \approx \frac{h}{EV} \frac{dL}{dt}$$

* Typically of the order of 10^{-3} to 10^{-4}

FIG. 1: PROJECTED WORKING TEMPERATURES OF TURBINE BLADE ALLOYS

FIG. 2: ROTATING ELECTRODE PROCESS (REP) FOR POWDER PRODUCTION

FIG. 3: INERT GAS ATOMIZATION PROCESS FOR PRODUCING SUPERALLOY POWDER

FIG. 4: VACUUM ATOMIZATION PROCESS FOR PRODUCING SUPERALLOY POWDER

1 BILLET

2 COMPLEX SHAPES

TYPICAL HIP CONDITIONS
(TITANIUM/SUPERALLOYS)

TEMPERATURE 1500-2300°F
PRESSURE 1000-15000 PSI
TIME ½-4 HOURS

FIG. 5: HOT ISOSTATIC COMPACTION PROCESS (HIP)

TYPICAL CYCLE

PRESSURE - 1000 PSI
TEMPERATURE - 1750°F
TIME - 1 HOUR

FIG. 6: VACUUM HOT PRESS (VHP) FOR THE PRODUCTION
OF NET P/M SHAPES IN TITANIUM

FIG. 7: STRAIN-RATE DEPENDENCE OF A FLOW STRESS

FIG. 8: FLOW DIAGRAM OF DIFFUSION BONDING TECHNIQUES FOR THE MANUFACTURE OF W FIBER-Ni ALLOY MATRIX MONOTAPES

FIG. 9: FLOW CHART OF THE WESTINGHOUSE CANADA COMPOSITE AIRFOIL MANUFACTURING PROCESS

FIG. 10: UNCONSTRAINED FAILURE MODES FOR A FIBER COMPOSITE

FIG. 11: CONSTRAINED FAILURE MODE FOR A FIBER COMPOSITE

**FIG. 12: AUGMENTATION OF TENSILE-STRESS
DISTRIBUTION AROUND A PARTICLE
BY PLASTIC FLOW**

FIG. 13: EFFECT OF SUPERIMPOSED
HYDROSTATIC PRESSURE ON
DUCTILE-FRACTURE BEHAVIOUR

FIG. 14: VARIATION OF NATURAL STRAIN
TO FRACTURE WITH PRESSURE
FOR ALUMINUM ALLOY

FIG. 15: TYPICAL FORMING LIMIT DIAGRAM FOR SHEET METAL

(a)

(b)

(c)

FIG. 16: MODEL ELEMENTS

FIG. 17: PLOT OF EXPERIMENTAL MEASUREMENTS OF $\mu L/h$ AND CORRESPONDING VALUES OF $2x/L$ AT THE INNERMOST VOID IN COMPOSITE SPECIMEN FORGED TO 20% REDUCTION

FIG. 18: As-HIPed COMPOSITE ANVIL

FIG. 19: LAYOUT OF INDUCTION HEATING COMPRESSION SYSTEM, SHOWING
 (A) MTS LOAD FRAME, (B) ENVIRONMENTAL CHAMBER, (C) MTS CONTROL
 SYSTEM, (D) EXPONENTIAL FUNCTION GENERATOR, (E) LEVEL INDUCTION
 GENERATOR, (F) TEMPERATURE CONTROLLER, RECORDERS

FIG. 20: SCHEMATIC CROSS SECTION OF THE INDUCTION HEATING COMPRESSION TRAIN

KEY TO FIGURE 20

1. Displacement transducer
2. Flipper shaft handle
3. Top anvil support
4. Water cooling coil
5. Top plate
6. Top flange
7. Flipper shaft
8. Top ceramic block
9. Copper shield
10. Top asbestos plate
11. Collar
12. RF flange
13. RF leads
14. Clamping nut
15. RF port
16. Water cooling coil
17. Bottom plate
18. Bottom anvil support
19. Vacuum door clamp
20. Inconel sheathed thermocouple
21. Transducer extension probe
22. Transducer extension tube
23. Compression chamber
24. Induction coil
25. Bottom die
26. Susceptor
27. Bottom anvil
28. Vacuum door
29. Cooling chamber
30. Vacuum door handle

FIG. 21: SCHEMATIC DIAGRAM OF THE TEMPERATURE CONTROL SYSTEM

FIG. 22: DETAILS OF THE COOLING CHAMBER SHOWING THE ACTUATING SHAFTS OF THE VACUUM DOOR

FIG. 23: SCHEMATIC DESCRIPTION OF THE GAS SUPPLY AND VACUUM SYSTEM

- A DCDT
- B DCDT CHAMBER
- C ROTARY SHAFTS
- D MICROMETER SCREWS
- E PROBES
- F TOP ANVIL

FIG. 24: DETAILS OF THE STRAIN MONITORING APPARATUS
AND UPPER ANVIL ASSEMBLY

FIG. 25: RAM POSITION CONTROL SYSTEM

FIG. 26: SPECIMEN TEMPERATURE AS A FUNCTION OF TIME FOR GRAPHITE AND STAINLESS STEEL SUSCEPTORS

FIG. 27: FLOW CHART OF DATA ACQUISITION AND RECORDING SYSTEM

GROOVE GEOMETRY - ALL DIMENSIONS IN mm.
 - HEIGHT TO DIAMETER RATIO ~ 1.5
 - END FACES GROOVED TO RETAIN LIQUID LUBRICANT

FIG. 28: SPECIMEN GEOMETRY FOR THE MATRIX MATERIAL

FIG. 29: SPECIMEN GEOMETRY FOR THE COMPOSITE MATERIAL

FIG. 30: TEMPERATURE CALIBRATION CURVES CORRELATING SET POINT WITH SPECIMEN TEMPERATURE AND DISPLAYED CHAMBER TEMPERATURE (ORIGINAL SYSTEM)

FIG. 31: TEMPERATURE CALIBRATION CURVES CORRELATING SET POINT WITH SPECIMEN TEMPERATURE AND DISPLAYED CHAMBER TEMPERATURE (MODIFIED SYSTEM)

*ALL DIMENSIONS IN mm.

FIG. 32: HYDROSTATIC EXTRUSION BILLET

FIG. 33: REPRODUCIBILITY OF FLOW CURVES

MARBLE'S ETCH ; 500 X

INCO ETCH ; 2400 X

FIG. 34: MICROSTRUCTURES IN As-HIPed MAR M200 COMPACTS

4750 X

FIG. 35: EFFECT OF SOAKING PERIOD ON THE MICROSTRUCTURE OF THE MATRIX MATERIAL

12 X

MARBLE'S ETCH ; 100 X

FIG. 36: CROSS SECTIONS OF COMPOSITE MATERIAL HIPed AT 1050°C

a: 21 X

b: 21 X

FIG. 37: CROSS SECTIONS OF COMPOSITE MATERIAL HIPed AT 1150°C

700 X

FIG. 38: ELECTRON IMAGE OF CRACKED TUNGSTEN FIBER

360 X

FIG. 39: X-RAY SCAN FOR HAFNIUM
AT THE LOCATION OF THE
HORIZONTAL TRACE

250 X

FIG. 40: X-RAY IMAGE FOR HAFNIUM
SHOWING CONCENTRATION IN THE CRACK

FIG. 41: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 950°C

FIG. 42: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 1000°C

FIG. 43: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 1050°C

FIG. 44: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 1100°C

FIG. 45: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 1150°C

FIG. 46: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF MAR M200 AT 1180°C

3 3

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FIG. 47: TEMPERATURE DEPENDENCE OF THE PEAK FLOW STRESS OF MAR M200 FOR VARIOUS STRAIN RATES

FIG. 48: STRAIN RATE DEPENDENCE OF THE PEAK FLOW STRESS OF MAR M200 AT DIFFERENT TEMPERATURES

FIG. 49: TEMPERATURE DEPENDENCE OF THE STRAIN RATE SENSITIVITY EXPONENT $m = (\partial \ln \sigma / \partial \ln \dot{\epsilon})_{T, \text{Structure}}$

FIG. 50: PEAK FLOW STRESSES OF THE MAR M200 AT 1150°C AND SIX DIFFERENT STRAIN RATES

FIG. 51: COMPARISON OF THE PEAK FLOW STRESSES OF THE SOLUTION TREATED
 AND AS-HIPed MAR M200 COMPACTS AT 1150°C

FIG. 52: EFFECTS OF MESH SIZE ON THE PEAK FLOW STRESS OF MAR M200

FIG. 53: EXTRUDED BILLET - 15% REDUCTION

FIG. 54: EXTRUDED BILLET - 20% REDUCTION

FIG. 55: EXTRUSION PRESSURES FOR SEVERAL SUPERALLOYS

FIG. 56: MICROHARDNESS MEASUREMENTS ACROSS EXTRUDED BILLETS

FIG. 57: HIGH TEMPERATURE FLOW CURVES OF EXTRUDED BILLETS

FIG. 58: FLOW CURVES OF MAR M200 COMPACTS PREFORGED AT 1100°C TO DIFFERENT AMOUNTS OF STRAIN

FIG. 59: FLOW CURVES OF MAR M200 COMPACTS PREFORGED AT 1150°C TO DIFFERENT AMOUNTS OF STRAIN

FIG. 60: REDUCTIONS OF PEAK FLOW STRESS OF PRESTRAINED MAR M200
COMPACTS AT DIFFERENT TEMPERATURES

FIG. 61: SECONDARY FLOW CURVES OF MAR M200 COMPACTS
PREFORMED AT 1100°C AND $8.8 \times 10^{-2} \text{ s}^{-1}$

FIG. 62: SECONDARY FLOW CURVES OF MAR M200 COMPACTS
PREFORGED AT 1100°C AND $2.1 \times 10^{-1} \text{ s}^{-1}$

FIG. 63: MICROSTRUCTURES DEVELOPED IN MAR M200 COMPACTS FORGED AT 1100°C AND 10^{-1} s^{-1} TO DIFFERENT STRAINS

MARBLE'S ETCH; 500X

**FIG. 64: MICROSTRUCTURE DEVELOPED IN OVER-AGED MAR M200
COMPACTS DURING HOT WORKING TO A STRAIN OF 0.6 AT
1050°C AND $3.0 \times 10^{-4} \text{ s}^{-1}$**

2400 X

2400 X

**FIG. 65: MICRODUPLEX NATURE OF DYNAMICALLY RECRYSTALLIZED
AREAS IN MAR M200 COMPACTS ETCHED TO SHOW GRAIN BOUNDARIES**
(a) INITIALLY FINE GRAINED COMPACT,
(b) INITIALLY COARSE GRAINED COMPACT

1000°C ; 500 X

950°C ; 500 X

1100°C ; 500 X

1150°C ; 500 X

FIG. 66: EFFECTS OF TEMPERATURE ON THE MICROSTRUCTURE DEVELOPED DURING FORGING

1000 °C ; 300 X

950 °C ; 300 X

1100 °C ; 300 X

1150 °C ; 300 X

FIG. 67: EFFECTS OF TEMPERATURE ON THE HOT DUCTILITY OF
MAR M200 COMPACTS DEFORMED TO 0.6 STRAIN AT 10^{-3} s^{-1} ;

$1.0 \times 10^{-2} \text{ s}^{-1}$; 300 X

$0.88 \times 10^{-1} \text{ s}^{-1}$; 300 X

$1.4 \times 10^{-3} \text{ s}^{-1}$; 300 X

$9.6 \times 10^{-4} \text{ s}^{-1}$; 300 X

**FIG. 68: EFFECTS OF STRAIN RATE ON THE HOT DUCTILITY OF MAR M200
COMPACTS DEFORMED TO 0.6 STRAIN AT 1100°C**

$\epsilon = -0.2$; 400 X

$\epsilon = 0.1$; 400 X

$\epsilon = 0.8$; 400 X

$\epsilon = 0.4$; 400 X

FIG. 69: DAMAGE DEVELOPED IN THE MAR M200 COMPACTS DURING FORGING TO VARIOUS STRAIN LEVELS, AT 1100°C AND 10^{-1} s^{-1}

10^{-2} s^{-1} ; 300 X

10^{-1} s^{-1} ; 300 X

10^{-3} s^{-1} ; 300 X

FIG. 70: EFFECTS OF COLD WORKING BY EXTRUSION PRIOR
TO FORGING ON THE HOT DUCTILITY OF MAR M200
COMPACTS FORGED TO 0.6 STRAIN AT 1100°C

FIG. 71: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF THE COMPOSITE MATERIAL AT 1050°C

FIG. 72: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF THE COMPOSITE MATERIAL AT 1100°C

FIG. 73: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF THE COMPOSITE MATERIAL AT 1150°C

FIG. 74: EFFECTS OF STRAIN RATE ON THE FLOW CURVE OF THE COMPOSITE MATERIAL AT 1180°C

FIG. 75: STRAIN RATE DEPENDENCE OF THE PEAK FLOW STRESS OF THE COMPOSITE MATERIAL AT DIFFERENT TEMPERATURES

MURAKAMI'S ETCH; 300 X

MARBLE'S ETCH; 100 X

MURAKAMI'S ETCH; 400 X

**FIG. 76: LONGITUDINAL AND TRANSVERSE SECTIONS SHOWING FIBER
CRACKING IN COMPOSITE MATERIAL FORGED TO
0.8 STRAIN AT 1100°C AND $1.4 \times 10^{-3} \text{ s}^{-1}$**

**FIG. 77: TRANSVERSE SECTION OF FORGED COMPOSITE SHOWING
EXTENSIVE CAVITIES DEVELOPED NORMAL TO THE LOADING
DIRECTION AT THE FIBER MATRIX INTERFACE. THE ARROWS
INDICATE THE DIRECTION OF FIBER MOVEMENTS.**

(100 X)

800 X

400 X

**FIG. 78: TRANSVERSE SECTIONS OF COMPOSITE MATERIAL SHOWING
MATRIX CRACKING DEVELOPED DURING FORGING TO 0.1 STRAIN
AT 1180°C AND $0.88 \times 10^{-1} \text{ s}^{-1}$**

500 X

FIG. 79: TRANSVERSE SECTIONS OF COMPOSITE MATERIAL SHOWING MICROVOIDS DEVELOPED AT POWDER PARTICLE BOUNDARIES DURING FORGING TO 0.3 STRAIN AT 1100°C AND $0.88 \times 10^{-1} \text{ s}^{-1}$

PHOTOGRAPHS REDUCED 50%

FIG. 80: TRANSVERSE SECTIONS OF COMPOSITE MATERIAL SHOWING VOID INITIATION AND GROWTH BETWEEN THE HAFNIUM NITRIDE COATING AND THE TUNGSTEN FIBERS

4X

100 X

FIG. 81: TRANSVERSE SECTION THROUGH THE As-HIPed COMPOSITE

FIG. 82: TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED TO 0.03 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1100°C

FIG. 83: TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED TO 0.06 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1100°C

FIG. 84: TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED TO 0.10 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1100°C .

FIG. 85: TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED TO 0.30 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1100°C

10^{-1} s^{-1} ; 5 X

10^{-4} s^{-1} ; 5 X

**FIG. 86: EFFECTS OF STRAIN RATE ON THE HOT DUCTILITY OF
COMPOSITE BLANK DEFORMED TO 0.3 STRAIN AT 1100°C**

6X

100 X

100 X

FIG. 87:- TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED
TO 0.10 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1180°C

MARBLE'S ETCH; 700 X

FIG. 88: TRANSVERSE SECTION THROUGH A COMPOSITE BLANK DEFORMED TO 0.10 STRAIN AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1180°C SHOWING MICROSTRUCTURE OF γ^1 DENUDED REGION

200 X

300 X

400 X

100 X

FIG. 89: CONCENTRATION OF γ^1 DENUDED REGIONS IN AREAS CONTAINING
 (a) CAVITIES AT FIBER/MATRIX INTERFACE, (b) MATRIX CRACKING
 (c) FIBER CRACKING, (d) HIGHLY CONCENTRATED PLASTIC DEFORMATION

FIG. 90: X-RAY SCAN FOR ALUMINUM AT THE LOCATION OF THE HORIZONTAL TRACE SHOWING (a) CONCENTRATION IN THE CRACK, (b) NO EVIDENCE OF CONCENTRATION IN VOID FREE REGION

FIG. 91: X-RAY SCAN FOR CHROMIUM AT THE LOCATION OF THE HORIZONTAL TRACE SHOWING (a) CONCENTRATION IN THE CRACK, (b) NO EVIDENCE OF CONCENTRATION IN VOID FREE REGION

MARBLE'S ETCH; 800 X

MARBLE'S ETCH; 800 X

FIG. 92: MICROSECTIONS OF AS-RECEIVED MAR M200 POWDERS

INCO ETCH ; 4750 X

MARBLE'S ETCH ; 500 X

FIG. 93: MICROSTRUCTURES OF MAR M200 COMPACTS
SUBJECTED TO DIFFERENT HEAT TREATMENTS

FIG. 94: EFFECTS OF TEMPERATURE ON THE FLOW CURVES OF MAR M200 COMPACTS AT CONSTANT TRUE STRAIN RATE

FIG. 95: RATIO OF PEAK FLOW STRESS OF SOLUTION TREATED (ANNEALED) AND AS-HIPed MAR M200 COMPACTS

FIG. 96: FLOW CURVES OF EXTRUDED AND As-HIPed MATERIALS DEFORMED AT 1100°C AND DIFFERENT STRAIN RATES

FIG. 97: EFFECT OF STRAIN RATE ON PEAK FLOW STRESS OF PREFORGED AND As-HIPed COMPACTS

FIG. 98: EFFECT OF TEMPERATURE ON FLOW BEHAVIOUR AND STEADY STATE MICROSTRUCTURES OF MAR M200 COMPACTS.

FIG. 99: STRAIN RATE DEPENDENCE OF THE APPARENT MODULUS OF ELASTICITY OF MAR M200 COMPACTS AT DIFFERENT TEMPERATURES

UNETCHED ; 300 X

MARBLE'S ETCH ; 300X

**FIG. 100: FRACTURE DEVELOPED AROUND COARSE PARTICLE IN MAR M200
COMPACT DURING HOT WORKING TO A STRAIN OF 0.6 AT
950°C AND $1.4 \times 10^{-3} \text{ s}^{-1}$**

FIG. 101: FLOW CURVES FOR MATRIX AND COMPOSITE MATERIALS
DEFORMED AT $9.6 \times 10^{-5} \text{ s}^{-1}$ AND 1100°C

FIG. 102: FLOW CURVES FOR MATRIX AND COMPOSITE MATERIALS
DEFORMED AT $3 \times 10^{-4} \text{ s}^{-1}$ AND 1100°C

FIG. 103: FLOW CURVES FOR MATRIX AND COMPOSITE MATERIALS
DEFORMED AT $9.6 \times 10^{-5} \text{ s}^{-1}$ AND 1050°C

FIG. 104: FLOW CURVES FOR MATRIX AND COMPOSITE MATERIALS
DEFORMED AT $3 \times 10^{-4} s^{-1}$ AND $1050^{\circ}C$

FIG. 105: DEFORMATION MODES IN (a) MATRIX AND (b) COMPOSITE MATERIAL

FIG. 106: PEAK FLOW STRESSES OF MATRIX AND COMPOSITE MATERIALS AT 1150 AND 1180°C AND DIFFERENT STRAIN RATES (semi log. plot)

FIG. 107: PEAK FLOW STRESSES OF MATRIX AND COMPOSITE MATERIALS AT 1050 AND 1100°C AND DIFFERENT STRAIN RATES (semi log. plot)

FIG. 108: PEAK FLOW STRESSES OF MATRIX AND COMPOSITE MATERIALS AT 1150 AND 1180°C AND DIFFERENT STRAIN RATES (log. plot)

FIG. 109: PEAK FLOW STRESSES OF MATRIX AND COMPOSITE MATERIALS AT 1050 AND 1100°C AND DIFFERENT STRAIN RATES (log. plot)

a

b

FIG. 110: FLOW PATTERNS OF (a) SUPERPLASTIC EUTECTIC OF LEAD-TIN AFTER 58% REDUCTION IN HEIGHT DURING PLANE STRAIN COMPRESSION AND (b) COMPOSITE MATERIAL FORGED TO 53% REDUCTION IN HEIGHT

(A)

$T = 1100^{\circ}\text{C}$

$\dot{\epsilon} = 0.088 \text{ s}^{-1}$

$\epsilon = 0.26$

(B)

$T = 1050^{\circ}\text{C}$

$\dot{\epsilon} = 0.0001 \text{ s}^{-1}$

$\epsilon = 0.30$

(C)

$T = 1050^{\circ}\text{C}$

$\dot{\epsilon} = 0.0014$

$\epsilon = 0.24$

(D)

$T = 1100^{\circ}\text{C}$

$\dot{\epsilon} = 0.088 \text{ s}^{-1}$

$\epsilon = 0.33$

FIG. 111: SCHEMATIC SHOWING FRACTURE PATTERNS OBSERVED AT DIFFERENT TEMPERATURES AND STRAIN RATES IN COMPOSITE BLANKS FORGED TO HIGH STRAINS

(a) $b = h$

(b) $b = 2h$

FIG. 112: IDEALIZED SLIP LINE FIELDS FOR PLANE STRAIN COMPRESSION

FIBER ARRANGEMENT

DEFORMED MODEL TO
THE CRITICAL STRAIN

FIG. 113: MODEL (A) FOR COMPOSITE MATERIAL

FIBER ARRANGEMENT

DEFORMED MODEL TO
THE CRITICAL STRAIN

FIG. 114: MODEL (B) FOR COMPOSITE MATERIAL

FIG. 115: EFFECTS OF VOLUME FRACTION OF CIRCULAR FIBER ON CRITICAL STRAIN

FIBER ARRANGEMENT

COMPOSITE CELL

FIG. 116: MODEL (C) FOR COMPOSITE MATERIAL

FIG. 117: EFFECTS OF VOLUME FRACTION OF HEXAGONAL FIBER ON CRITICAL STRAIN.

FIG. 118: COMPUTED EFFECTIVE STRAIN DISTRIBUTION AT
20% REDUCTION IN HEIGHT
(Ref. 124)

FIG. 119: MAXIMUM DAMAGE DISTRIBUTIONS IN A FORGED COMPOSITE
AT 20% REDUCTION IN HEIGHT

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