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HIGH TEMPERATURE MECHANICAL PROPERTIES AND BEHAVIOUR OF PLATINUM-BASE ALLOYS

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ABSTRACT

The need to reduce fuel consumption aboard spacecraft and jets has initiated the development of platinum-base superalloys, which are intended to raise the firing temperatures of such engines by being able to withstand the arduous environments within the hot zones at temperatures at least 200°C higher than the most advanced nickel-base superalloys. The platinum-base superalloy development approach is based on mimicking the $\gamma/\gamma'$ microstructures of the well-established nickel-base superalloys, with the intention of potentially exploiting the high melting point and environmental inertness of the platinum base.

This project reports on the high temperature mechanical properties and behaviour of three platinum-base alloys, which forms part of the larger platinum-base superalloy development initiative. High temperature uniaxial compression testing is employed to investigate the variation in the high temperature strength with temperature and strain rate as well as to assess the effects of the deformation variables on the microstructural evolution of these materials during a laboratory simulated thermo-mechanical forming operation.

The starting microstructures obtained after homogenisation are different for each material. The homogenisation treatment produces a high volume fraction of $\gamma'$ arranged in a dendritic-like network in Pt$_{86}$Al$_{14}$Cr$_3$Ru$_4$; a low volume fraction of irregularly shaped $\gamma'$ in Pt$_{86}$Al$_{10}$Cr$_4$; and a platinum-rich solid solution devoid of $\gamma'$ in Pt$_{86}$Al$_{10}$Ru$_4$. The change in the high temperature strength with deformation temperature for Pt$_{86}$Al$_{10}$Cr$_4$ shows an abrupt transition, which is attributed to the different phase transformations that the alloy undergoes during compression testing above and below the homogenisation temperature. In contrast, Pt$_{86}$Al$_{14}$Cr$_3$Ru$_3$ and Pt$_{86}$Al$_{10}$Ru$_4$ resist microstructural changes during compression testing and consequently do not display abrupt transitions in the changes in high temperature strength with deformation temperature.

Pt$_{86}$Al$_{10}$Cr$_4$ and Pt$_{86}$Al$_{10}$Ru$_4$ show extensive grain refinement during hot compression testing. This is due to the metal softening events that occur during the deformation experiments. Based upon metallographic analysis and the interpretation of the laboratory flow data, the mechanism of restoration is provisionally identified as dynamic recrystallisation. In contrast, Pt$_{86}$Al$_{14}$Cr$_3$Ru$_3$ did not show a significant reduction in grain size, which is primarily due to the inability of the alloy to be deformed to significant strains before the onset of cracking.
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LIST OF ABBREVIATIONS

FCC face-centered cubic
APB anti-phase boundary
SFE stacking fault energy
\( \gamma \) FCC matrix phase of superalloys, which must be coherent with \( \gamma' \)
\( \gamma' \) FCC intermetallic primary precipitate phase of superalloys, which must be coherent with \( \gamma \)
\( \gamma'' \) FCC intermetallic secondary precipitate phase of superalloys, which must be coherent with \( \gamma \)
Pt\(_3\)Al FCC intermetallic precipitate phase of platinum-base alloys, which may either be primary or secondary in nature and need not be coherent with \( \gamma \)
L\(_12\) high temperature FCC form of Pt\(_3\)Al, which is required for \( \gamma/\gamma' \) superalloy coherency
D\(_{0'}\)c or D\(_{0_{24}}\) low temperature body-centered tetragonal martensitic form of Pt\(_3\)Al, which is not coherent with \( \gamma \)
(Pt) platinum-rich solid solution phase
Pt-Al-Ru platinum-aluminium-ruthenium alloying system
d\(_o\) original diameter of the compression specimen before compression
h\(_o\) original height of the compression specimen before compression
d\(_f\) final outer diameter of the compression specimen after compression
h\(_f\) final height of the compression specimen after compression
d\(_c\) contact diameter of the compression specimen after compression
d\(_{fa}\) actual measured final outer diameter of the compression specimen after compression
d\(_{fa}\) FEM simulated final outer diameter of the compression specimen after compression
d\(_{cs}\) FEM simulated final outer diameter of the compression specimen after compression
d\(_{ca}\) actual contact diameter of the compression specimen after compression
\( \varepsilon_u \) true uniaxial strain
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$\sigma_u$</td>
<td>true uniaxial stress</td>
</tr>
<tr>
<td>$\dot{\varepsilon}_i$</td>
<td>mean nominal strain rate</td>
</tr>
<tr>
<td>$\dot{\varepsilon}$</td>
<td>mean strain rate</td>
</tr>
<tr>
<td>$F_i$</td>
<td>instantaneously measured load during uniaxial compression</td>
</tr>
<tr>
<td>$A_i$</td>
<td>instantaneous cross-sectional area that corresponds to the instantaneous load</td>
</tr>
<tr>
<td>$\dot{\varepsilon}_u$</td>
<td>true uniaxial strain rate</td>
</tr>
<tr>
<td>$\dot{\varepsilon}$</td>
<td>strain rate</td>
</tr>
<tr>
<td>$v_i$</td>
<td>instantaneous velocity of the deformation platens during uniaxial compression testing</td>
</tr>
<tr>
<td>$\Delta \dot{\varepsilon}_i$</td>
<td>instantaneous strain increment</td>
</tr>
<tr>
<td>$\Delta t$</td>
<td>time increment corresponding to the instantaneous strain increment</td>
</tr>
<tr>
<td>$\varepsilon_\theta$</td>
<td>hoop strain</td>
</tr>
<tr>
<td>$\sigma_\theta$</td>
<td>hoop stress</td>
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<tr>
<td>$\alpha$</td>
<td>slope of the plot of $\varepsilon_\theta$ against $\varepsilon_u$</td>
</tr>
<tr>
<td>$\sigma_h$</td>
<td>hydrostatic stress</td>
</tr>
<tr>
<td>$\sigma_r$</td>
<td>representative stress</td>
</tr>
<tr>
<td>$\mu$</td>
<td>coefficient of friction</td>
</tr>
<tr>
<td>$\tau$</td>
<td>Tresca coefficient of friction</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Coulombic coefficient of friction</td>
</tr>
<tr>
<td>$\tau_i$</td>
<td>average frictional shear stress</td>
</tr>
<tr>
<td>$p$</td>
<td>interfacial pressure or axial stress</td>
</tr>
<tr>
<td>$A_a$</td>
<td>apparent area of contact</td>
</tr>
<tr>
<td>$P$</td>
<td>force normal to $F$</td>
</tr>
<tr>
<td>$\sigma_f$</td>
<td>material flow stress</td>
</tr>
<tr>
<td>$k$</td>
<td>value of 0.577 $\sigma_f$</td>
</tr>
<tr>
<td>$\sigma_f$</td>
<td>flow stress corrected for friction</td>
</tr>
<tr>
<td>$B$</td>
<td>barrelling coefficient</td>
</tr>
<tr>
<td>$m$</td>
<td>Tresca coefficient of friction</td>
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</table>
List of Abbreviations

Λ: interpolation function dependent on material-specific parameters

FEA: Finite Element Algorithm

$\varepsilon_{ij}$: strain rate tensor

$\sigma_{ij}$: deviatoric tensor

$\Delta T_{\text{adiabatic}}$: temperature rise due to deformation heating

$\Delta T_{\text{adiabatic}}$: temperature rise due to adiabatic heating only

$T_{\text{actual}}$: actual temperature of the specimen obtained from the sum of the apparent specimen temperature and the temperature due to deformation heating

$\eta$: adiabatic heat factor

$\gamma$: fraction of work dissipated as heat

$\int \varepsilon d\sigma$: area under the flow curve

$C_p$: specific heat

$\rho$: material density

$\sigma_{\text{apparent}}$: apparent flow stress without considering the effects of deformation heating

$\sigma_{\text{actual}}$: actual flow stress of the material due to deformation heating

FEM: Finite Element Model

LG: logarithmic strain

PEEQ: plastic equivalent strain

EDE: energy of deformation

RX-GS: estimated recrystallised grain size ranking

$\sigma_{\text{hps}}$: high temperature proof strength

MFSE: mean flow stress estimate

$\varepsilon_p$ or $\varepsilon_{\text{peak}}$: peak strain

$\sigma_p$ or $\sigma_{\text{peak}}$: peak stress

$\varepsilon_c$: critical strain

$\sigma_c$: critical stress

$\varepsilon_s$ or $\varepsilon_{\text{ss}}$: steady state strain
<table>
<thead>
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<th>Abbreviation</th>
<th>Description</th>
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<tr>
<td>$\sigma_{ss(e)}$</td>
<td>steady state flow stress</td>
</tr>
<tr>
<td>MDRX</td>
<td>metadynamic recrystallisation</td>
</tr>
<tr>
<td>$Z$</td>
<td>Zener-Hollomon parameter</td>
</tr>
<tr>
<td>$K$</td>
<td>slope of $\ln(\sigma)$ versus $1/T$ plots at various strain levels</td>
</tr>
<tr>
<td>$m$</td>
<td>slope of $\ln(\sigma)$ versus $\ln(\varepsilon)$ plots at various strain levels</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>flow stress</td>
</tr>
<tr>
<td>$Q$</td>
<td>activation energy</td>
</tr>
<tr>
<td>$R$</td>
<td>universal gas constant</td>
</tr>
<tr>
<td>$T$</td>
<td>temperature of deformation</td>
</tr>
<tr>
<td>$\partial\sigma/\partial\varepsilon$</td>
<td>work-hardening rate</td>
</tr>
<tr>
<td>LVDT</td>
<td>linear variable differential transformer</td>
</tr>
<tr>
<td>rf</td>
<td>radio frequency</td>
</tr>
<tr>
<td>$T_o$</td>
<td>actual measured temperature of the specimen</td>
</tr>
<tr>
<td>$T_s$</td>
<td>set-point temperature</td>
</tr>
<tr>
<td>$W$</td>
<td>actual power output</td>
</tr>
<tr>
<td>$P$</td>
<td>proportional bandwidth control/P-constant/p-gain</td>
</tr>
<tr>
<td>$I$</td>
<td>integral control/I-constant</td>
</tr>
<tr>
<td>$D$</td>
<td>derivative control/D-constant</td>
</tr>
<tr>
<td>TEM</td>
<td>transmission electron microscopy</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscopy</td>
</tr>
<tr>
<td>EBSD</td>
<td>electron backscattered detection</td>
</tr>
<tr>
<td>SED</td>
<td>secondary electron detection</td>
</tr>
<tr>
<td>EDS</td>
<td>electron diffraction spectroscopy</td>
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CHAPTER 1

INTRODUCTION

1.1 Background and motivation

Platinum-base superalloys are currently being developed for use as structural materials in components intended for ultra-high temperature applications. The platinum-base superalloy development is an attempt to expand the industrial applications of platinum and in so doing, improve the exporting capacity of the local platinum market in South Africa\(^1\). This approach has evolved as a result of the persistent pressure that fuel emissions legislations have been enforcing on industry as well as from the continuous need to conserve fuel aboard spacecraft and jets\(^2\). Furthermore, the well-established series of nickel-base superalloys, which are the most common group of metallic materials employed in the hot zones of aerospace and jet engines, are reaching a limit in operating temperatures\(^3\),\(^4\),\(^5\),\(^6\),\(^7\). This is a direct consequence of the relatively low melting point that is inherent to the nickel base and it is therefore felt that any advances in the quest for higher service temperatures would have to be gained through an alloy development approach\(^4\),\(^8\).

The platinum superalloy development approach is thus based on mimicking the microstructures of the nickel-base superalloys with the benefit of potentially exploiting the higher melting point and superior high temperature oxidation resistance of the platinum base\(^8\),\(^9\),\(^10\). The platinum-superalloy design involves the development of a high volume fraction of ordered FCC L\(_{12}\) cubic precipitates (intermetallic phase) finely dispersed in a more ductile FCC matrix, analogous to the nickel-base superalloys\(^3\),\(^4\),\(^5\),\(^6\),\(^8\),\(^9\),\(^10\). The dispersion should be coherent\(^7\), meaning that although there is a high surface area associated with the precipitates, the actual lattice mismatch with respect to the matrix and hence the interfacial and elastic energies are very small\(^1\). This presents the system with a very small driving force for precipitate coarsening, thereby enabling the material to maintain mechanical integrity after prolonged exposure at high temperatures\(^1\).

It is thought that the high density of the raw platinum metal would be compensated by the improvements made in fuel conservation during operation\(^7\). Moreover, it should be viable for scrapped components to be processed so as to recover the platinum economically, therefore the actual cost is not reflected in the high initial price of the precious metal itself, but rather by the sum of the refining costs and the charges in interest incurred on the capital investment in the platinum consumed\(^11\). Notwithstanding the potential
benefits mentioned above, it is unlikely that platinum-base superalloys would replace nickel-base superalloys other than in critical components that have to operate at the maximum possible temperature.\textsuperscript{1,3}

1.2 Scope for platinum-base superalloys

The improvement in the service efficiency of gas turbine materials for both land-based and jet engines is driven by the demand for increased thrust-to-weight ratios, superior fuel efficiency and reduced fuel emission, all of which are governed by greater firing temperatures during operation.\textsuperscript{2,3} Materials selection should be based on the ability to withstand hot corrosive attack from sulphate and sulphite fluxing at low to intermediate temperatures and oxidation at high temperatures; high specific strength and modulus over a wide temperature range; and good fatigue resistance to thermal and vibrational stress fluctuations.\textsuperscript{21,23} Platinum materials have already found an application in the aerospace industry where they are currently being employed in the nozzles of spacecrafts\textsuperscript{12} to meet the arduous service requirements, particularly during re-entry to the earth's atmosphere. A very small increase in operating temperature is considered a significant advancement, for example, it is possible to increase the output of land-based gas turbines by up to 13% by raising the service temperature by only 55°C, which represents a significant financial gain.\textsuperscript{23} The platinum-base alloys currently under investigation are intended to operate at least 200°C higher than the maximum operating temperature of the most advanced commercially available nickel-base superalloy grades.\textsuperscript{1,5}

Platinum made an early debut in structural materials intended for high temperature applications in the early 1960's as a platinum-modified aluminide coating, which was later patented in the 1970's.\textsuperscript{2,13} These materials are currently being used to protect nickel-base superalloy surfaces from premature environmental degradation, through the improved adherence of the protective oxide to the superalloy substrate; the prevention of void formation at the oxide-coating interface; and the promotion of the selective oxidation of aluminium that causes a reduction in the oxide growth rate thereby delaying spallation. More recently, the use of platinum as an alloying constituent to nickel-base superalloys has been investigated, where the role of platinum is to impart superior environmental resistance without compromising the high temperature mechanical integrity of the component itself.\textsuperscript{4,15} Platinum partitions strongly to the ordered phase, which is considered the weaker link in the corrosion resistance of such materials,\textsuperscript{14} and so improves the hot corrosion of the material as a whole.\textsuperscript{11} The high temperature oxidation resistance is also improved through the accelerated growth of the continuous alumina scale and the chromium enriched subscale region.\textsuperscript{15,16} A new class of dispersion hardened platinum materials is currently being developed. These materials are alloyed with oxidisable additions of zirconium, yttrium and cerium, which upon annealing in an oxidising medium leads to the internal oxidation of the platinum and the formation of finely dispersed oxide particles.\textsuperscript{17} The dispersion strengthening, however, leads to such excessive brittleness that these materials
are ultra-sensitive to thermal shock and severe particle coagulation also manifests during rapid cooling, which results in a complete loss in high temperature strength and creep resistance\textsuperscript{17}.

Platinum-base superalloys would benefit engine components and provide added advantages that the above mentioned materials cannot offer. The $\gamma$ phase is a ductile FCC matrix that serves to toughen the material, making it much more capable of accommodating plastic strain than the dispersion hardened materials. In addition, since the superalloy is platinum-rich, the entire component would experience superior oxidation and creep resistance, unlike the platinum-modified aluminide coatings, which impart enhanced properties only to the surface of the component. The high temperature properties of the platinum-base superalloys would also be superior to the nickel-base superalloys containing small additions of platinum but at the expense of a much higher cost. It is clear that the material choice is governed by the application of the end-product and therefore it seems as if platinum-base superalloys would probably be employed in high-tech applications where the best possible high temperature properties are required throughout the entire volume of the component and where cost is not a major consideration.

1.3 Project objectives

The design engineer is primarily interested in the high temperature properties of the end product material, particularly the high temperature strength, for the development and mechanical design of structural components intended for high temperature applications. Fifty percent of the total weight of superalloys is still being employed in disk and blade components of gas turbines\textsuperscript{18}, which are largely produced via a multi-step hot forging schedule, during which careful control of the processing parameters is central to the evolution of a fine-grained microstructure\textsuperscript{19,20}. Such a microstructure is critical to the development of the mechanical properties required for these components, including low-cycle fatigue resistance, room temperature strength and high temperature ductility\textsuperscript{9,21,22,23}. Enabling a superalloy to flow during a forging operation is not a trivial exercise as often superalloys are brittle and hot working operations generally have to be performed above the $\gamma'$ ageing temperature to achieve a significant reduction in strength and increase in ductility\textsuperscript{18}.

The primary objective of the current study is to investigate the high temperature mechanical properties of three platinum base alloys that have been manufactured and supplied by Mintek. This includes a comparative study between the high temperature strength values of the different alloys as well as an assessment of the hot formability of the experimental material during high temperature compression testing. The assessment of the flow behaviour of the material by means of the analysis of laboratory flow data obtained during hot working in order to gauge the hot formability of the alloys is emphasised. Microstructural analysis of the hot worked state is also conducted in an attempt to explain the observed
flow behaviour. At present, there is little or no published literature concerning the hot formability of any platinum-base material, as platinum has not made an appearance as a structural material to date.
CHAPTER 2

LITERATURE REVIEW

2.1 Phase transformations and precipitate morphologies in platinum-base alloys

2.1.1 Mimicking the γ/γ' microstructures of nickel-base superalloys

(a) The basic microstructure of nickel-base superalloys

Nickel-base superalloys are a commercial series of alloys designed specifically to resist creep deformation and environmental degradation at high operating temperatures\textsuperscript{11}. The good creep resistance and high strength is derived from a high volume fraction of cuboidal shaped γ' Ni₃Al precipitates embedded finely and coherently in a γ matrix (see figure 2.1), which serve as obstacles to the movement of dislocations when the material undergoes deformation and/or restoration processes\textsuperscript{8,14}. The evolution of the γ' precipitates, which is an FCC ordered intermetallic phase, is driven by a lowering of the chemical free energy, the γ/γ' interfacial energy and the elastic distortion energy associated with the γ/γ' lattice mismatch\textsuperscript{25}. Equilibrium γ' shapes develop as a consequence of a balance between the elastic and interfacial energies\textsuperscript{26}. The elastic energy is proportional to the volume of γ' as well as the extent of the shielding interaction between γ' precipitates and favours faceting along (001), whilst the interfacial energy is proportional to the γ/γ' interfacial area and promotes compact, isotropic γ' shapes\textsuperscript{26}. The γ matrix, which is a ductile FCC phase, is strengthened by solid solution strengthening mechanisms and serves to toughen the material as a whole.
Figure 2.1: Scanning electron micrograph (SEM) showing the typical microstructure of a nickel-base superalloy, consisting of $\gamma'$ (the larger precipitates) and $\gamma$ (the smaller precipitates) embedded in a $\gamma$ matrix (after reference 27).

(b) $\gamma'$ coarsening and dissolution in nickel-base superalloys

The spherical to cubic structural transformation (see figure 2.2(A)) of the $\gamma'$ precipitates of nickel-base superalloys is indicative of the competition between the elastic and interfacial energies\textsuperscript{28}. Spherical or quasi-spherical shapes are present when the average size in the $\gamma'$ precipitates is small, which causes the interfacial energy to dominate\textsuperscript{28}. However, the elastic contribution increases as the precipitate continues to grow in an attempt to minimise the associated interfacial energy\textsuperscript{27}, which drives the transformation in shape from spherical to cubic\textsuperscript{29}. The spherical precipitate deforms in isotropic elasticity towards a cuboidal shape with $\{100\}$ planes and the shear deformation on $\{100\}$ approaches equal extensions in $[010]$ and $[001]$ with twice the compression experienced along $[100]$\textsuperscript{28}. The result is that the strain energy in the matrix is less than that associated with the spherical inclusion. In practice, precipitates actually transform beyond a cuboidal shape and are therefore observed as being slightly concave on the $\{100\}$ faces\textsuperscript{28}. The elastic energy associated with the octocubical shape is less than that associated with the cubic morphology\textsuperscript{29}. This forms the basis of the driving force for the transformation from a cube to an octocube (see figure 2.2(B)) by means of single precipitates splitting when the $\gamma'$ size is above some critical value\textsuperscript{29}.

The final shape change is a solid-state transformation from an octocubic to an octo-dendritic morphology (see figure 2.2(C)) that occurs in a highly saturated matrix\textsuperscript{28}. The coherency of the $\gamma'/\gamma$ interface is maintained, therefore the formation of the dendritic arms serves to accommodate elastic strain along $\{001\}$, despite the growth mechanism that forces the eight branches of the octodendritic structure to extend in $\langle 111 \rangle$\textsuperscript{29}. Secondary and tertiary branching sequences could take place when the dendritic arms are sufficiently separated from one another\textsuperscript{29}. The rate of precipitate growth is progressively reduced as it becomes controlled by the solute diffusion from the increasingly depleted matrix\textsuperscript{28}. The $\gamma'/\gamma'$ interface is smoothed by the loss in coherency associated with the action of advanced dislocation trapping\textsuperscript{28}. The fully
dendritic structure is obtained upon prolonged ageing, which facilitates the coarsening of the octo-dendritic branches\textsuperscript{36}. The $\gamma'$ precipitates coarsen according to the Ostwald Ripening process, where the larger precipitates grow at the expense of the smaller ones\textsuperscript{27}, thereby giving rise to wider $\gamma$ channels which results in a loss in strength and creep properties as dislocations are able to bow between the precipitates\textsuperscript{24}. In contrast, it has been observed that smaller precipitates do not necessarily dissolve in favour of the growth of larger ones, which is attributed to a preferential coarsening behaviour that is determined by the coherency strains of $\gamma'$ precipitates\textsuperscript{22}. The dissolution of $\gamma'$ is governed by the differences in the local stability that is associated with the elastic interactions and therefore it is often observed that precipitates do not dissolve by a continuous thinning process but are rather linked by a successive disappearance of individual precipitates as they become more unstable in relation to one another\textsuperscript{35}. The octodendrites finally transform to fully dendritic structures (see figure 2.2(D))\textsuperscript{30}.

![Schematic illustration showing the sequence of successive shape changes during the structural evolution of a $\gamma'$ precipitate that grows freely (after reference 30).](image)

Figure 2.2: Schematic illustration showing the sequence of successive shape changes during the structural evolution of a $\gamma'$ precipitate that grows freely (after reference 30).

(c) **Platinum-base superalloys as microstructural analogues of nickel-base superalloys**

The design of platinum-base superalloys is based on developing $\gamma/\gamma'$ microstructures analogous to those of the nickel-base superalloys\textsuperscript{24}. Platinum has excellent resistance to environmental degradation and an extremely high melting point which is thought to provide the basis for superior resistance to high temperature oxidation and creep respectively\textsuperscript{29}. The addition of aluminium to platinum has been found essential not only to precipitate the ordered Pt$_3$Al phase ($\gamma'$) but also to limit the extent of loss in the high temperature oxidation resistance that is associated with the pure platinum\textsuperscript{8}. Figure 2.3 shows the equilibrium binary phase diagram for the platinum-aluminium system\textsuperscript{24}. The following reactions are noteworthy:
Reaction 1 is the general eutectic reaction that starts at 1507°C at about 20 atomic percent of aluminium. It gives rise to an aluminium eutectic composition during which the high temperature $L_1$ structure of Pt$_3$Al is formed, which eventually transforms to $D_0_{19}$ at lower temperatures, as well as the platinum-rich solid solution (Pt)$^5$. Reaction 1 can be subdivided into reactions 2 and 3. Reaction 2 is a congruent transformation that takes place at 1290°C and results in the formation of the low temperature $D_0_{19}$ structure of Pt$_3$Al. This transformation is believed to be similar to the martensitic transformation observed in the platinum-iron system that occurs in the vicinity of 25 atomic percent of platinum$^4$. It is uncertain whether the mechanism of the martensitic transformation in Pt$_3$Al proceeds via the classical shear mechanism observed in the iron-carbon system or by a mechanism of pure ordering followed by a twinning process$^{35}$. In addition, the enthalpy changes can be very small in non-ferrous martensitic transformations and non-ferrous martensites can even be softer than the parent phase$^{35}$. Reaction 3 occurs at 1280°C and is responsible for the formation of the platinum-rich solid solution (Pt)$^5$.

Figure 2.3: The current binary platinum-aluminium equilibrium phase diagram at the platinum-rich end (after reference 34).
2.1.2 The effects of ruthenium additions to platinum-rich Pt-Al

Ruthenium has been shown to partition nearly exclusively to (Pt) with extremely low solubility in Pt$_3$Al when added in quantities of about 5 atomic percent to alloys of the platinum-rich Pt-Al system$^{5,4,38}$. At platinum contents greater than 73 atomic percent, a c labelled mixture of Pt$_3$Al and (Pt) solid solution containing up to 10 atomic percent of aluminium and 58 atomic percent of ruthenium exists$^{39}$. Small additions of ruthenium (below 5 atomic percent) increase the hardness of the material significantly (see table 2.1)$^{39}$, probably by a solid solution strengthening mechanism$^{5,9}$. Larger additions of ruthenium do not necessarily increase in the overall hardness of Pt-Al-Ru further as the higher contents of ruthenium tend to influence the volume fraction and morphology of the Pt$_3$Al phase (see table 2.1), which is predominantly responsible for the hardening of Pt-Al-Ru$^{39}$.

The limited solubility of ruthenium in Pt$_3$Al also means that it is unlikely that this element would affect the transformational behaviour of the precipitate phase$^{5}$. Therefore, ruthenium is seen to have little influence on the ability of the alloying system to maintain the L1$_2$ form of Pt$_3$Al at low temperatures$^{4,24,38}$. This has been confirmed by the XRD analysis conducted by Hill et al., where the experimental XRD traces of Pt-Al-Ru at room temperature display peaks that correspond to the D0$_{24}$ form of Pt$_3$Al when compared to modelled XRD patterns obtained using Crystallographica$^6$ (see figure 2.4)$^{34,36}$. The transformation to the D0$_{24}$ form of Pt$_3$Al results in a loss in the matrix-precipitate coherency which ultimately diminishes the creep resistance of the material$^6$.

The "Maltese Cross"-like Pt$_5$Al$_3$Ru$_4$ (see alloy 9 in table 2.1) is related to the tetragonally distorted structure of the Pt$_3$Al phase$^6$. Evidently, the distortion of D0$_{24}$ Pt$_5$Al vertically along the unit cell is very slight, therefore the XRD traces of the L1$_2$ and D0$_{24}$ forms are very similar, with the only distinguishable difference being some peak distortion and splitting of the D0$_{24}$ form at the high diffraction angles of interest$^4$. In addition, it has been alluded to by Süss that texturing of the samples could manifest during the homogenisation treatment of the cast form of Pt$_{46}$Al$_{54}$Ru$_{10}$$^{37}$. This often renders the split peaks, which are rather low in intensity and thus difficult to detect, even more difficult to discern$^{34,37}$. The presence of twins$^9$ and/or martensitic$^{35}$ lathes in the Pt$_3$Al phase is an indication of the displactic transformation from the cubic to the tetragonal structure. Alternatively, this precipitate morphology could be due to the relatively high lattice misfit at both room temperature (-1.2% at 24°C) and at elevated temperature (-0.97% at 800°C) in Pt$_{46}$Al$_{54}$Ru$_{10}$. The effect of ruthenium in Pt$_{46}$Al$_{54}$Ru$_{10}$ is predominantly to impart solid solution strengthening to the (Pt) phase$^{5,6}$, however, it does not alter the L1$_2$-to-D0$_{24}$ transformation since it shows such limited solubility in the Pt$_3$Al phase$^{34,36}$. Hence, the addition of ruthenium to the Pt-Al system is unsuccessful in retaining the L1$_2$ form of Pt$_3$Al, which is needed for developing a microstructure analogous to nickel-base superalloys.
<table>
<thead>
<tr>
<th>alloy</th>
<th>composition of Pt-Al-Ru</th>
<th>microstructural observations</th>
<th>hardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pt$<em>{75}$Al$</em>{25}$Ru$_2$</td>
<td>lightly twinned Pt$_3$Al matrix and thick grain boundary phase fine second phase Pt in a eutectoid mixture</td>
<td>398±15</td>
</tr>
<tr>
<td>2</td>
<td>Pt$<em>{90}$Al$</em>{10}$Ru$_4$</td>
<td>distinctly cored two-phase lightly twinned Pt$_3$Al matrix</td>
<td>454±11</td>
</tr>
<tr>
<td>3</td>
<td>Pt$<em>{75}$Al$</em>{25}$Ru$_4$</td>
<td>lightly twinned Pt$_3$Al matrix fine second phase Pt in a eutectoid mixture third twinned phase</td>
<td>443±8</td>
</tr>
<tr>
<td>4</td>
<td>Pt$<em>{75}$Al$</em>{25}$Ru$_5$</td>
<td>unidentified matrix phase large unidentified second phase</td>
<td>492±9</td>
</tr>
<tr>
<td>5</td>
<td>Pt$<em>{85}$Al$</em>{15}$Ru$_5$</td>
<td>primary and discrete Pt$_3$Al twinned precipitates Pt matrix</td>
<td>438±10</td>
</tr>
<tr>
<td>6</td>
<td>Pt$<em>{75}$Al$</em>{25}$Ru$_6$</td>
<td>fairly coarse dendritic mixture of Pt$_3$Al and Pt twinned Pt$_3$Al with high volume fraction</td>
<td>489±9</td>
</tr>
<tr>
<td>7</td>
<td>Pt$<em>{75}$Al$</em>{25}$Ru$_7$</td>
<td>twinned Pt$_3$Al matrix discrete Pt</td>
<td>454±6</td>
</tr>
<tr>
<td>8</td>
<td>Pt$<em>{80}$Al$</em>{20}$Ru$_8$</td>
<td>untwinned Pt$_3$Al matrix Widmanstätten-like Ru platelets</td>
<td>531±12</td>
</tr>
<tr>
<td>9</td>
<td>Pt$<em>{90}$Al$</em>{10}$Ru$_9$</td>
<td>fine two-phase mixture with Pt matrix discrete twinned Pt$_3$Al forming “Maltese Cross” structures</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 2.1: Hardness numbers and microstructural traits for various Pt-Al-Ru ternary alloys at the platinum-rich end (reconstructed from references 5, 6 and 36).
2.1.3 The effects of chromium additions to Pt-Al

Chromium partitions preferentially to (Pt) but also shows limited solubility in Pt-Al when added in quantities of about 5 atomic percent to platinum-rich alloys of the Pt-Al system. The addition of chromium reduces the aluminum content in (Pt) and also serves to shift the platinum-rich boundary of the Pt-Al phase to higher platinum contents compared to that of the binary platinum-aluminum system and in so doing, reduces the width of the (Pt)-Pt,-Al two-phase field. Previous experimental work conducted on the Pt-Al-Cr ternary alloy by Hill et al. has shown that the Pt-Al precipitates display a bimodal distribution in size, containing both major Pt-Al, which are coarse precipitates between 200nm and 1000nm in size with angular and cubic morphologies, and minor Pt-Al, which are fine, cuboidal precipitates located in the (Pt) matrix between the major Pt-Al. The size of the minor Pt-Al varies depending on their distance from the major Pt-Al and can be as small as 10nm closest to the major Pt-Al. Three separate studies by Hill et al. has also shown that Pt-Al-Cr, Pt-Al-Cr, and Pt-Al-Cr comprise of small primary Pt-Al surrounded by a two-phase mixture of (Pt) solid solution and Pt-Al.

The Pt-Al precipitates are absent from twins and/or martensitic laths and is therefore considered as the desirable L12γ phase. Furthermore, Selected Area Diffraction patterns (SADs) taken along the (112) axis at room temperature show superlattice spots that correspond to the L12 variant of Pt-Al. The lattice mismatch is reported as 0.72% at both room and elevated temperature (800°C), meaning that the alloy is near-coherent. The cuboidal morphologies observed are consistent with that of the nickel-base
superalloys, which also have cuboidal precipitates with lattice misfits less than 1%. It has been suggested that the major PtAl form during the homogenisation treatment at 1350°C for 96 hours, whilst the minor precipitates form during the slow, furnace cooling from the homogenisation temperature, which is consistent with that observed for nickel-base superalloys. The smaller size of the minor PtAl is attributed to a reduction in the concentration of aluminium in the immediate surroundings of the precipitates.

2.2 Inhomogeneous metal flow and flow stress corrections

2.2.1 Barrelling

(a) The physical phenomena of barrelling

During uniaxial compression testing, a solid cylindrical specimen of initial height \( h_0 \) and initial diameter \( d_0 \) is reduced along its height between a pair of open, overhanging platens in a process that is commonly termed “upsetting”\(^{41,42,43,44}\). The interaction between the unavoidable surface asperities of the deformation platens and that of the test specimen gives rise to frictional constraints at the contact surfaces\(^{45,46,47}\), which retard the flow of metal directly beneath the loading face in the dead zones (see zones 1 and 2 in figure 2.5)\(^{48,49,50,51,52,53,54,55}\). As the extent of deformation is increased, so the specimen spreads laterally, causing the material in the free deformation region (see zone 4 in figure 2.5) to extend along the free surface, which is the point of least resistance. At very high strains, further lateral extension is primarily due to the folding over of the sides of the specimen (see figure 2.5), \(^{51,54,55,56,57,58,59,60}\). The accumulation of material is greatest at the centre of the specimen (see zone 3 in figure 2.5), whilst the material remains relatively undeformed in the region closest to the loading face. \(^{51,54,55,56,57,58}\) Friction leads to an inhomogeneous distribution of strain through the cross-section of the compression specimen.\(^{51,54,55,56,57,58}\).
Figure 2.5: Schematic representation of the inhomogeneous strain distribution throughout the cross-section of a barrelled specimen. The extent of deformation experienced in each zone is related to the distance separating the lines in the respective zones (redrawn from reference 51).

Figure 2.6: Schematic representation of end face expansion due to the lateral spread and folding over at the specimen sides (redrawn from reference 47).
(b) Geometrical changes during barrelling

During frictional uniaxial compression the specimen bulges outward to form a barrel shape of new dimensions (see figure 2.5). By applying the principle of volume constancy between the initial cylindrical specimen and the barreled specimen and by assuming that the barreled radius fits a circular arc, equation 2.1 and equation 2.2 can be written for conditions of homogeneous and inhomogeneous deformation respectively:

$$d_i h_i = d_f h_f$$

Equation 2.1

$$\frac{\pi}{12} (2d_i^2 + d_f^2) h_f = \frac{\pi}{4} (d_i h_f)$$

Equation 2.2
Equation 2.3 is an analytical solution for the specimen contact diameter after compression, which according to experimentalists such as Banerjee\textsuperscript{42} and Schey\textsuperscript{47}, is extremely difficult to measure physically within an acceptable degree of accuracy. It relates the contact diameter ($d_c$) to the radius of curvature ($R_c$), the original height ($h_o$), the original diameter ($d_o$) and the final height ($h_f$) of the specimen\textsuperscript{41,47}. Equation 2.4 is known as the new geometrical barrel shape factor and can be used to validate the initial assumptions that the volume of the specimen is conserved and that the radius of the barrelled specimen fits a circular arc. The expression relates $R_c$ in terms of $h_o$, $d_o$, $d_f$ and $d_c$\textsuperscript{41,48,49}.

$$d_c = \frac{2}{3} \left[ -2x \pm \sqrt{\frac{9h_o d_o^2}{h_f} - 8x^3} \right]$$

and

$$x = R_c \pm \sqrt{R_c^2 - \frac{h_f^2}{4}}$$

*Equation 2.3*

$$R_c^{eq} = \left( \frac{h_f}{2(d_f - d_c)} \right) \left( \frac{3d_c^2}{2d_f^2 + d_c^2} \right)$$

*Equation 2.4*
2.2.2 Buckling

In order to maintain an axisymmetric mode of deformation during uniaxial compression testing, it is vital that the test specimen be aligned centrally with respect to the deformation platens, in the direction of the applied load, and that the specimen remains in this position throughout the course of the compression event. Any misalignment that arises during the test procedure could cause the metal to deform asymmetrically and the specimen would buckle along its height.

Metal flow is highly dependent of the temperature of deformation. An inhomogeneous temperature distribution within the test specimen will cause an inhomogeneous distribution of flow stress and hence strain. Since the inhomogeneous distribution in temperature is likely to be asymmetric about the central axis of the cylindrical specimen, the mode of deformation suffered by the specimen would also be asymmetric in nature. A phenomenon commonly termed as die-chilling in the field of metalworking is often the cause of temperature-inflicted specimen buckling. Die-chilling is brought about when the temperature of the two platens are different, causing the specimen to deform differently according to the local temperatures at the respective loading faces. This gives rise to chill caps and chill bands and local shear bands develop inhomogeneously through the corners of the specimen.

Under conditions of sliding friction (as defined in section 2.3.1(c)), the work that is required to overcome the frictional forces at the interface to continue deformation is transformed into heat. The heat is evolved in a very thin film, meaning that the temperatures developed are sufficiently high to result in lubrication breakdown locally at specific points at the loading face, which cause significant changes in the properties of the test material at the interface. Such circumstances can lead to severe specimen buckling with associated asymmetric flow. Under conditions of sticking friction (as defined in section 2.3.1(c)), however, temperature rises at the interface do not exceed local rises within the bulk of the test material. The material would therefore deform asymmetrically and the interfacial temperature rises would only promote an increase in free surface expansion or barrelling.
2.2.3 Flow stress correction due to frictional effects

(a) Stress evaluation

During uniaxial compression testing a solid cylindrical specimen is inserted between a set of flat platens. The platens are driven toward each other at an initial velocity to produce an initial strain rate. The force required for this operation is measured and logged as a function of strain. The true uniaxial strain ($\varepsilon_u$) experienced by the material during this process is given by equation 2.5, where $h_o$ is the original/initial height of the specimen and $h_f$ is the final height. The true uniaxial stress ($\sigma_u$) is given by equation 2.6, where $F_i$ is the instantaneously measured load that is needed to strain the metal and $A_i$ is the instantaneous cross-sectional area that corresponds to the instantaneous load measurements over which the load is exerted in perfect uniaxial compression\textsuperscript{43,44}. Equation 2.6 is a simplified expression of equation 2.8, which can also be used to determine the true uniaxial stress. The original specimen height is given by $h_o$, the original specimen diameter by $d_o$, the final specimen height after deformation by $h_f$ and the final specimen diameter after deformation by $d_f$\textsuperscript{42}.

Equation 2.7 is used to determine $A_i$, where $d_i$ is the instantaneous diameter of the specimen and $h_i$ is the instantaneous height. Note that both equation 2.5 and equation 2.6 are used to compute the true uniaxial strain and stress respectively on the assumption that the cylindrical geometry is perfectly maintained throughout the course of the compression cycle. The true uniaxial strain rate ($\dot{\varepsilon}_u$) of the metal is calculated by the expression given by equation 2.9, where $V_i$ is the instantaneous velocity of the platens, $h_i$ is the instantaneous height of the specimen, $\Delta \varepsilon$ is the strain increment and $\Delta t$ is the corresponding time increment. Note that $\dot{\varepsilon}_u$ is an instantaneous quantity and describes the strain rate at any level of strain during non-constant strain rate compression\textsuperscript{43,44}.

The hoop strain, which is given by $\varepsilon_o$ in equation 2.10, is defined as the strain experienced along the circumference of the specimen during uniaxial compression, where $d_o$ is the original/initial outer diameter of the specimen; $d_f$ is the final outer diameter of the specimen; and $d_c$ is the contact diameter of the specimen\textsuperscript{48}. The hoop stress, which is given by equation 2.11, is the stress exerted along the circumference of the compression specimen, where $\alpha$ is the slope of the plot of $\varepsilon_o$ against $\varepsilon_u$. The hoop stress in tensile in nature because the outer diameter of the specimen barrels due to the action of secondary tensile stresses during uniaxial compression\textsuperscript{48}. The representative and hydrostatic stresses, denoted as $\sigma_r$ and $\sigma_m$ respectively, are given by equation 2.13 and equation 2.14 respectively. The representative strain is given by equation 2.12 and is the strain experienced along a plane through the centre of the barrelled specimen in the direction of the applied compression force\textsuperscript{48}.
\[ \varepsilon_s = \ln \left( \frac{h_2}{h_1} \right) \]

*Equation 2.5*

\[ \sigma_s = \left| \frac{F}{A} \right| \]

*Equation 2.6*

\[ A = \frac{\pi \left( \frac{d_i}{2} \right)^3}{h_1} \]

*Equation 2.7*

\[ \sigma_s = \frac{8F_t}{\pi} \left( \frac{3h_2d_x^2}{h_1} - d_f^2 \right) \]

*Equation 2.8*

\[ \varepsilon_s = \left| \frac{\Delta u}{h} \right| = \frac{\Delta \varepsilon_s}{\Delta t} \]

*Equation 2.9*

\[ \varepsilon_s = \ln \left( \frac{2d_f + d_x}{3d_x^2} \right) \]

*Equation 2.10*

\[ \sigma_s = \sigma_s \left( \frac{1 + 2\alpha}{2 + \alpha} \right) \]

*Equation 2.11*
\[ \varepsilon_s = \frac{2}{\sqrt{3}} (1 + \alpha + \alpha^2)^3 \]

*Equation 2.12*

\[ \sigma_s = \sigma_s \left( 0.5 + \alpha \right) \left[ 3(1 + \alpha + \alpha^2) \right]^3 \]

*Equation 2.13*

\[ \sigma_s = \frac{1}{3} (\sigma_s + \sigma_s) \]

*Equation 2.14*

(b) Stress correction factors

One method of interpreting the deformation behaviour of a metal is to analyse the material flow stress\textsuperscript{56}. There are a number of expressions that have been proposed to correct flow data according to a particular friction condition. Gleeble\textsuperscript{9} recommends that equation 2.15 and equation 2.16 be used to adjust the flow stress according to a friction coefficient\textsuperscript{57}. No mention is made of whether the coefficients of friction (\( \nu \)) for use in these expressions are Tresca or Coulombic in nature. Evans and Scharning\textsuperscript{43} as well as Chovet et al\textsuperscript{6} describe the von Mises yield criterion by the expression given in equation 2.17. This equation is used to approximate the local equivalent flow stress, where \( \tau \) is the Tresca coefficient of friction. Similarly, Zhang et al\textsuperscript{88} describes the von Mises yield criterion by the expression given in equation 2.18, which is also used to adjust the flow stress for a Tresca coefficient of friction. Banerjee\textsuperscript{42} describes the Siebel corrective function by the expression given in equation 2.19. This expression is used to correct the flow stress at low coefficients of friction, where \( \mu \) is the Coulombic coefficient of friction. Partender and Bünten\textsuperscript{69} describe the Siebel corrective function by the expression given in equation 2.20. This equation is used to adjust the flow stress, where \( \tau \) is the Tresca coefficient of friction.
\[
\sigma^c = \sigma_a \left( 1 + \frac{\nu d_i}{3h_i} \right)^2
\]

*Equation 2.15*

\[
\sigma^c = \frac{\sigma_a}{2} \left( \frac{vd_i}{h_i} \right)^2 \left[ \exp \left( \frac{vd_i}{h_i} \right) - \frac{vd_i}{h_i} - 1 \right]^{-1}
\]

*Equation 2.16*

\[
\sigma' = \sigma_s \left[ 1 + \frac{\tau d_i}{h_i} \exp \left( \frac{3\epsilon}{2} \right) \right]^{-1}
\]

*Equation 2.17*

\[
\sigma^c = \sigma_a \left[ 1 + \frac{\mu d_i}{3\sqrt{3}h_i} \right]^{-1}
\]

*Equation 2.18*

\[
\sigma^c = \sigma_a \left( 1 + \frac{\mu d_i}{3h_i} \right)
\]

*Equation 2.19*

\[
\sigma' = \sigma_s \left( \frac{\mu d_i \sqrt{h_i}}{3h_i^2} \right)
\]

*Equation 2.20*
(c) The nature of friction

Friction is most commonly characterised by equation 2.21, which is an expression used to define the Coulombic coefficient of friction in terms of relevant forces and stresses. $F$ is the force that is required to move the body of mass; $P$ is the force normal to $F$; $\tau_i$ is the average frictional shear stress which is also given by $F/A_A$ where $A_A$ is the apparent area of contact; $p$ is the interfacial pressure (also known as the axial stress) normal to $F$ which is also given by $P/A_A$; and $\mu$ is the Coulombic coefficient of friction.

$$\mu = \frac{F}{P} = \frac{\tau_i}{p}$$

*Equation 2.21*

Equation 2.21 embodies the two most basic of Amonton’s laws of friction, that is, the frictional force is proportional to the normal force and that it is independent of the size of the apparent contact area. The relationship is used to describe a sliding motion and is most commonly employed in the characterisation of friction in metal forming operations. However, the expression is not necessarily a realistic model for metalworking processes because when $\tau_i$ reaches a critical value $k$, less energy will be required for metal-shearing within the actual body of the material with the surface remaining stationary, as opposed to end-face sliding against the platen surface as described by equation 2.21. This sub-surface shearing phenomenon is known as sticking friction, despite there not being any actual sticking to the platen surface. Sticking friction is described by equation 2.22, which defines the breakdown of sliding friction in terms of a critical value of frictional shear stress, given by $k$.

$$\tau_i = \mu p > k$$

*Equation 2.22*

According to von Mises, the value of $k$ is given by $0.577\sigma_f$, where $\sigma_f$ is the material flow stress. This value remains constant for metallic materials, however, during most metal forming operations, the normal interfacial pressure reaches high flow stresses such that the Coulombic coefficient of friction calculated from equation 2.21 drops off. This poses a practical limitation that is often ignored and hence the frequent reports of unrealistically low values of friction coefficients. Equation 2.23 is a more realistic approximation of the coefficient of friction under sticking friction conditions and is the friction model that is most commonly employed during investigations of metal working operations. In this expression, $\tau$ is the interfacial shear factor or the Tresca coefficient of friction; which is zero for a frictionless interface.
and unity for perfect sticking friction; and $\sigma$ is the flow stress as described by the von Mises yield criterion$^{55,56,61}$.  

$$\tau = \tau k \text{ or } \tau = \tau \frac{\sigma^c}{\sqrt{3}}$$

Equation 2.23

The major drawback with the relationship given in equation 2.23 is the assumption that the material of the specimen at the specimen/platen interface behaves closely like the bulk portion of the specimen. This is hardly true as often the specimen is lubricated with a third material and/or the chemistry of the interface changes significantly during the course of testing due to a combination of specimen oxidation and the incorporation of platen and/or lubricating material into the specimen surface under severe sticking conditions$^{55}$.  

The effects of friction on the deformation behaviour of a solid cylindrical specimen during uniaxial compression testing may be summarised as follows:

1. When the interfacial friction is zero ($\tau = 0$), end-face expansion proceeds freely and the specimen retains its cylindrical shape as the specimen expands along the free-surface. The flow stress of the material during uniaxial compression is then given by the axial stress, which is a global pressure averaged over the total cross-sectional area of the specimen$^{55}$.  

2. When the interfacial friction is low ($\tau < k$), end-face expansion is opposed by the frictional shear stress. The specimen barrels somewhat but the end-face spreads outward away from the central point by a sliding motion. The flow stress of the material is slightly lower than the axial stress$^{55}$.  

3. When the interfacial friction is sticking at some point ($\tau = k$), a small dead-zone is formed immediately beneath the loading-face and sliding is confined to an annulus. The frictional drag should be modelled either by $\tau$ or by a mixture of $\tau$ and $\mu$$^{55}$.  

4. When the interfacial friction is sticking at all points ($\tau > k$), the specimen sticks to the platen from the edge inward. A specimen with a low initial d/h ratio deforms highly inhomogeneously and end-face growth is predominantly due to folding over of the specimen sides. At higher d/h ratios, the dead-zones interpenetrate thereby resulting in end-face expansion and a steep rise in interfacial axial stresses$^{55}$.  

22
(d) Solving the coefficient of friction

The barrelling coefficient is a ratio defined by the expression given in equation 2.24, where $B$ is the barrelling coefficient; $h_f$ is the final specimen height after deformation; $d_f$ is the final specimen diameter after deformation; $h_o$ is the initial specimen height; and $d_o$ is the initial specimen diameter \(^{43,57}\).

$$B = \frac{h_f d_f^2}{h_o d_o^2}$$

*Equation 2.24*

For a fixed specimen volume, $d_o/h_o$ ratio, deformation temperature and strain rate, the barrelling coefficient is related to the Tresca coefficient of friction by the relationship given in equation 2.25, where $m$ is the Tresca coefficient of friction; $\Lambda$ is an interpolation function; and $B$ is the barrelling coefficient \(^{43}\).

$$m = \Lambda \left(1 + \sqrt{B}\right)$$

*Equation 2.25*

It has been shown that $\Lambda$ is strongly dependent on the $d_o/h_o$ ratio and that it also forms a linear relationship with this ratio, whilst being only weakly dependent on the specimen volume, deformation temperature and strain rate \(^{43}\). $\Lambda$ may be solved by interpolating from a host of material-specific parameters that have to be determined empirically \(^{43}\). Also, the relationship between $B$ and $m$ is mainly affected by the initial dimensions of the specimen, $d_o/h_o$ (see figure 2.8) and only slightly influenced by the specimen volume, deformation temperature and strain rate \(^{43}\).
From figure 2.8, the square root of the barrelling coefficient appears to take a straight-line relationship with \( m \), with all the curves seemingly converging to a particular point\(^\text{43}\). This is rather convenient because if \( m \) could somehow be determined for a single value of \( B \) then all the other values of \( m \) could be solved by extrapolating back on the basis that the straight-line function represents a valid relationship\(^\text{43}\). The coefficient of friction may be determined by simulating the compression test numerically and reiterating the input of \( m \) into the simulation until the simulated outer diameter of the specimen at the end of the compression event fits that obtained physically in the laboratory\(^\text{50}\).

The deformation behaviour of solid cylindrical specimens during uniaxial compression testing has been modelled by Evans and Schaming\(^\text{43,44}\) using a Finite Element Algorithm (FEA), which has a fully coupled viscoplastic formulation\(^\text{40}\). The deformation process is mapped in two dimensions and the specimen geometry is defined and discretised into nodal isoparametric elements, which are structured axisymmetrically\(^\text{43,44}\). The numerical procedure employs updated Lagrangian meshes to formulate nodal tracking and elemental distortion through the duration of the simulation, whilst volume conservation is attained by a penalty function\(^\text{43,44}\). The nodal positions are updated on the basis of the nodal velocities and a single-step Euler scheme and automatic algorithms allow for nodal attachment and detachment to the platen surfaces at any given stage in the compression cycle\(^\text{43,44}\). The metal is assumed to behave completely isotropically as deformation proceeds according to the relationship given in equation 2.26\(^\text{44}\). Equation 2.26 gives the numerical relationship between the strain rate tensor and the deviatoric tensor used to define the simulated metal deformation process\(^\text{44}\). The specimen surfaces that make contact with the platens are subject to boundary conditions in the form of velocities, which are equal to that of the platen movement in the direction of loading\(^\text{43}\). End-faces are given Tresca frictional tractions as defined by the sticking friction laws. The specimen surfaces at the free-ends can deform and bulge freely in the absence
of boundary constraints\textsuperscript{43,44}. Figure 2.9 shows the mesh distortion that occurs during a simulated FEM uniaxial compression experiment.

\[
\varepsilon'_{ij} = \frac{3\varepsilon_{ij}}{2\sigma}
\]

\textit{Equation 2.26}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure2_9}
\caption{A Finite Element mesh showing the specimen geometry and associated strain fields before (left) and after (right) compression (after reference 44).}
\end{figure}

2.2.4 Flow stress correction due to deformation heating effects

During the deformation of a metal, most of the work that is generated is dissipated in the form of adiabatic heat, whilst the remaining work is converted into energy that is used to drive microstructural changes within the material\textsuperscript{62,63}. At relatively low deformation rates (generally at strain rates less than 10\textsuperscript{-3}/s\textsuperscript{44}), there is sufficient time available for the heat to be transferred away from the specimen before it is able to cause significant temperature rises locally within the specimen\textsuperscript{64,65}. At higher deformation rates (generally at strain rates greater than 10\textsuperscript{-3}/s\textsuperscript{64}), the amount of heat generated is much greater and there is also insufficient time for effective thermal transfer away from the specimen, thereby resulting in a significant increase in temperature within the specimen\textsuperscript{64,65}. Under such circumstances, the thermal inertia of the thermocouple could result in a significant lag in the transfer of the electrical signal, which means that at high strain rates there could be a considerable difference between the actual temperature of the specimen and that which is measured\textsuperscript{66}. During a formability investigation, it is important that the actual temperature of deformation be known as it provides additional quantitative information concerning the temperature.
dependence of the flow stress of a material\textsuperscript{62}. Even in a qualitative study such information is crucial in deducing whether a particular flow response is indicative of an actual softening phenomenon due to true isothermal behaviour or merely an artefact of one due to the effects of deformation heating\textsuperscript{65,67}.

Several approaches have been proposed for the generation of isothermal flow data. The most common of these approaches involves the approximation of the temperature rise with the aid of the thermal transfer model given by equation 2.27\textsuperscript{52}. The temperature due to deformation heating is given by $\Delta T_{\text{actual}}$ and that due to adiabatic heating alone by $\Delta T_{\text{adiabatic}}$\textsuperscript{64}. The adiabatic correction factor is denoted by the symbol $\eta$ and is zero for strain rates less and equal to $10^{-3}/s$ and unity for strain rates greater than and equal to $10/s$\textsuperscript{64}. The fraction of work that is dissipated as heat is given by $\gamma$\textsuperscript{68}, where $0.80 \leq \gamma \leq 0.95$\textsuperscript{18}. The integral function $\int \sigma \epsilon d\epsilon$ gives the area under the flow curve, which is representative of the total energy required for the deformation process\textsuperscript{62,64}. $C_p$ is the specific heat and $\rho$ is the density of the material\textsuperscript{62,63}.

$$
\Delta T_{\text{actual}} = \eta \Delta T_{\text{adiabatic}} \quad \text{and} \quad \Delta T_{\text{adiabatic}} = \frac{\gamma \int \sigma \epsilon d\epsilon}{\rho C_p}
$$

\textit{Equation 2.27}

After the value of $\Delta T_{\text{actual}}$ is computed from equation 2.27, a straight-line plot of $\ln(\sigma_{\text{apparent}})$ against $1000/T_{\text{actual}}$ is then constructed, where $\sigma_{\text{apparent}}$ is the apparent flow stress without considering the effects of deformation heating and $T_{\text{actual}}$ is the actual temperature of the specimen obtained from the sum of the apparent specimen temperature and temperature rise due to deformation heating\textsuperscript{64}. The actual flow stress of the material due to deformation heating ($\sigma_{\text{actual}}$) is obtained by extrapolating back to the initial test temperature from the plot described above\textsuperscript{62}. Similarly, a plot of $\sigma_{\text{apparent}}$ against $T_{\text{actual}}$ would yield an exponentially decaying curve, which could also be used to extrapolate values of $\sigma_{\text{actual}}$ (see figure 2.10)\textsuperscript{62}. The entire flow curve can be reconstructed\textsuperscript{67} by reiterating this procedure at an appropriately chosen strain increment\textsuperscript{62}. The temperature rise due to deformation increases with increasing strain, strain rate and decreasing deformation temperature\textsuperscript{19}.
The adiabatic correction factor $\eta$ is a numerical fraction that is representative of the amount of adiabatic heat that is retained within the specimen after heat loss to the deformation platens by conduction. It may only be used between the isothermal conditions that exist at deformation rates $\leq 10^{-3}/s$ (where $\eta = 0$) and the adiabatic conditions that arise at deformation rates $\geq 10^{-3}/s$ (where $\eta = 1$). In general, $\eta$ had been considered to vary linearly with the function $\log(\dot{\varepsilon})$, meaning that for deformation rates of $10^{-3}$, $10^{-2}$, $10^{-1}$, $10^0$ and $10^1$/s, $\eta$ would assume values of 0.00, 0.25, 0.50, 0.75 and 1.00 respectively. However, several more recent studies have independently provided evidence suggesting that $\eta$ is dependent on strain and deformation temperature.
2.3 The interpretation of softening phenomena from flow data analysis

2.3.1 The fundamentals of softening mechanisms

(a) Recovery

Recovery is a restoration process that lowers the stored energy in a deformed microstructure by a series of micromechanisms that involve a rearrangement and change in the internal dislocation structure and density\textsuperscript{70}. The process is predominantly governed by a material property known as the stacking fault energy (SFE) and when favourable conditions prevail, SFE controls the extent to which dislocations are capable of dissociating, thereby determining the rate of dislocation climb and cross-slip within the material\textsuperscript{70}. Materials with face-centred cubic (FCC) crystallographic structures have low SFE, which means that the stacking fault or the separation distance between partial dislocations itself is large, thereby making it exceedingly easy for dislocations to dissociate into partial dislocations\textsuperscript{71}. Under such conditions, climb and slip processes are hindered to a large extent and subsequent recovery is limited. Consequently the activation energies observed for the hot deformation of materials with low SFE are substantially higher than those for self-diffusion and creep processes\textsuperscript{71}. In ordered structures such as $\gamma'$ in nickel-base superalloys, the SFE is very close in magnitude to the anti-phase boundary energy (APB), which is high and therefore the likely softening mechanism in such structures would be recovery\textsuperscript{72}.

Recovery proceeds via two primary micromechanisms; namely, the rearrangement of dislocations into stable configurations and the subsequent annihilation of dislocations, both of which are achieved by the glide, climb and slip of dislocations\textsuperscript{71}. Dislocations of opposite polarity on the same crystallographic plane annihilate by means of gliding towards each other, whilst dislocations of opposite sign on different planes annihilate by a combined glide/climb process\textsuperscript{70}. Screw dislocations, on the other hand, would annihilate by cross-slip\textsuperscript{70}. The lowering in the dislocation density by a gliding mechanism can occur at relatively low temperatures, whilst glide/climb is only possible at relatively high temperatures\textsuperscript{70}. Cross-slip takes place at high temperatures for materials of low SFE. The activation energy decreases with increasing dislocation density and decreasing annealing times when recovery is controlled by a thermally activated glide process\textsuperscript{70}. Dislocations with low angle misorientation (less than 15°) rearrange into lower energy configurations so as to reduce the stored energy originally brought about by the deformation process, and in so doing, annihilate under favourable conditions\textsuperscript{70}.


(b) Recrystallisation

Recrystallisation is the dominant softening mechanism occurring in materials with low SFE and involves the nucleation and growth of new strain-free grains within a highly deformed structure. Recovery in such materials is delayed as glide and climb processes are difficult to achieve and the resultant accumulated strain provides the driving force for softening by recrystallisation. A minimum level of stored energy is required for the initial nucleation process and to sustain subsequent growth. Nucleation occurs when a crystallite of low internal energy grows into a deformed portion of the material that is separated by a high angle crystallographic misorientation (greater than 15°). The micromechanisms that govern recrystallisation are thermally activated and diffusion controlled, therefore the temperature at which recrystallisation takes place is reduced as the annealing time is increased. Clearly, recrystallisation and recovery are competing processes. Recovery is not possible once the completion of recrystallisation has been attained, which is characterised by the consumption of the entire deformed substructure.

(c) Dynamic recrystallisation and flow characteristics

Dynamic recrystallisation is a restoration process that occurs during the hot deformation of materials of low SFE. It involves the birth of new strain-free grains, which are nucleated at pre-existing grain boundaries (see figure 2.11(a)) of grains that are already in the growth stage within the deformed material (see figure 2.11 (b)). At relatively high initial grain sizes and low strain rates, a band of recrystallised grains thickens at prior grain boundaries (see figure 2.11(c)). A necklace-like structure may be developed when there is a large difference between the sizes of the initial and the recrystallised grains (figure 2.11(b) and figure 2.11(c)) and eventually the material becomes fully recrystallised (see figure 2.11(d)). Thus new grains originate at old grain boundaries but since the material is continuously being deformed, the dislocation density of the new grains increases, thereby reducing any driving force for further growth. The final effect is a cease in the overall growth of the newly formed grains and therefore the size of the dynamically recrystallised grains does not vary with strain when the rate of work hardening is balanced by that of dynamic recrystallisation.
A superalloy exhibits limited dynamic recovery during continuous plastic deformation due to its low SFE. The state of recrystallisation within a grain during deformation is referred to as being under alternative variation, meaning that at the onset of deformation, there is a distinct variation in dislocation density and corresponding structure in every grain within the material, which causes an increase in stored energy within a grain that is unique to every grain. The general profile of the flow curve in figure 2.12(b) is typical for a material that undergoes dynamic recrystallisation. The dislocation density increases until some peak stress \( \sigma_p \) is attained, which corresponds to a peak strain \( \varepsilon_p \), at which stage the accumulation in strain is sufficient to provide a driving force for dynamic recrystallisation to occur by creating an embryo which acts as a nucleation site. Important to note is the critical strain \( \varepsilon_c \), which corresponds to the critical stress \( \sigma_c \), required for the onset of dynamic recrystallisation. During continuous deformation, the dynamically recrystallised grains would again increase the stored energy and revert to unrecrystallised grains iteratively until deformation ceases. Since the extent and mode of deformation is unique to each crystallographic orientation of every grain (referred to as texture), several alternative variations may coexist within the material.

The commencement of dynamic recrystallisation causes a substantial drop in flow stress, which is associated with dislocation annihilation, however, the effect of dynamic recrystallisation on the actual shape of the flow curve can only be viewed visually after the peak strain. Figure 2.12(a) is representative of a typical flow curve observed at low Zener-Hollomon parameters (high temperature and low strain rate) when the critical strain for recrystallisation is greater than the strain needed for the completion of recrystallisation. Under such conditions, sufficient time is provided by the relatively low deformation rate for effective dislocation rearrangement and material softening prior to the onset of a subsequent hardening cycle, thereby giving rise to the oscillating flow stress pattern observed. Figure 2.12(b), on the other hand, is a typical flow curve obtained at high Zener-Hollomon parameters (low temperature and high strain rate), where insufficient time is available for alternating cycles of hardening and softening to be
clearly visible and the material remains in a partially recrystallised state as subsequent cycles of recrystallisation start before previous ones have completed. The strain required for the completion of recrystallisation is therefore greater than the critical strain for recrystallisation and a single broad peak of flow stress, which is indicative of the average flow stress condition, is observed at and beyond the steady state strain, denoted $\varepsilon_c$. At this stage, the rate of hardening is balanced by the rate of softening.

Figure 2.12: Schematic representations of the shapes of flow curves under conditions that favour dynamic recrystallisation. Figure 2.12A illustrates the cyclic peak stress when $\varepsilon_r$ is less than $\varepsilon_c$ whilst figure 2.12B shows the single peak stress behaviour when $\varepsilon_r$ is greater than $\varepsilon_c$. The inserts in both figures represent magnified portions of the flow curve (after reference 70).

(d) Static and metadynamic recrystallisation

Static recrystallisation takes place in a previously hot deformed material when subjected to a subsequent annealing treatment. The amount of stored energy, which is controlled by the hot deformation parameters, along with the annealing variables, determines the kinetics of the static restorative process. Metadynamic recrystallisation occurs during the cooling process after deformation in the presence of recrystallised nuclei when the critical strain for the initiation of dynamic recrystallisation is exceeded. Some grains, which were favourably orientated, would naturally have restored to a certain extent and aligned so as to facilitate easy mobility of subgrain boundaries during the hot deformation process. During a post-deformation annealing treatment, the nuclei which have already formed during the deformation process would grow in an attempt to consume the heterogeneous, partially dynamically recrystallised surroundings without the incubation period that is usually required for the initiation of recrystallisation (see figure 2.13). The kinetics of a metadynamic recrystallisation process is also a function of the rate of cooling from the deformation temperature to room temperature. A material which has undergone partial
dynamic recrystallisation will possess a microstructure that is extremely heterogeneous in nature and would comprise of small dynamically recrystallised grains which are nearly dislocation-free; large dynamically recrystallised grains with a moderate dislocation density; and large unrecrystallised grains with a relatively high dislocation density\textsuperscript{20,45}. Certain regions within the material would grow during the initial stages of a post-deformation annealing treatment according to the mechanism of metadynamic recrystallisation, whilst other regions may recover metadynamically or statically if the level of deformation is either below or above the critical strain for recrystallisation respectively\textsuperscript{70}.

![Figure 2.13: Schematic illustration of the extent of fractional recrystallisation against annealing time, showing the onset of metadynamic recrystallisation (MDRX) (after reference 70).](image)

2.3.2 The identification of recrystallised grains

Notwithstanding the effects of static restoration on the overall softening response of the material, it is the dynamic restoration events that govern the flow of metal during the blow of a forging process. Dynamic recrystallisation, in particular, is a very important consideration during the forging of superalloys as it lowers the mean flow stresses significantly, thereby reducing the upsetting force required for deformation\textsuperscript{74,78}. Although flow data analysis provides valuable information concerning the occurrence of dynamic recrystallisation, it is considered ideal practise to attempt to validate such analysis with microstructural observations\textsuperscript{79}.

Generally, recrystallised grains are distinguishable from unrecrystallised grains on the basis of the shape and size as well as by the internal dislocation density\textsuperscript{18,19,20,63,72,75,80,81}. However, since deformation is carried out during the nucleation and growth of new grains that have recrystallised dynamically, the variation in grain size and shape is not obvious and consequently the task of visually identifying recrystallised grains is extremely complicated\textsuperscript{20}. Furthermore, the softening kinetics at high temperature often makes it very difficult to distinguish those grains that recrystallise dynamically from those that
recrystallise statically and/or metadynamically; although it has been suggested that metadynamically recrystallised grains often differ in appearance from those that recrystallise dynamically on the basis of more well defined recrystallised grain boundaries\textsuperscript{77}. In some cases, a time delay of merely 1 second between the end of the deformation cycle and the cooling of the specimen to room temperature would cause the metal to recrystallise statically and/or metadynamically\textsuperscript{26,77}. In addition, the evolution of new phases during cooling from the deformation temperature could at times make it difficult to relate the frozen (or quenched) microstructure to the actual hot-worked state of the material\textsuperscript{65,81,82}. Under certain deformation conditions the dislocation structures and densities are very similar between different grains, despite these grains possessing different times of recrystallisation-state variation, which further complicates the exercise of visually identifying dynamically recrystallised grains\textsuperscript{78}.

Sometimes dynamically recrystallised grains can be identified on the basis of the large amounts of residual deformation displayed by individual grains and by the grain elongation observed in the direction of the dominant metal flow\textsuperscript{62}. Dynamically recrystallised grains are often serrated along the edges prior to a subsequent nucleation event, which usually occurs at prior grain boundaries that give rise to distinctive necklace-like structures\textsuperscript{19,80,81}. This is a direct result of dislocation sub-boundaries with medium to high misorientations that are developed during the transient state between newly generated grains and initial grains during the nucleation stage\textsuperscript{80}. Also, in the early stages of deformation before the peak stress is reached, grain boundaries can be unstable and may appear irregular, wavy and bulged or can bow outward due to the gradient in the local strain and the inhomogeneous substructure that is developed by the mechanism of grain boundary sliding or shearing\textsuperscript{63,80}. It has been suggested that when small recrystallised grains are observed in the vicinity of the tips of the larger elongated grains then the softening mechanism could be metadynamic recrystallisation as the growth of the smaller grains is “pinched off” or hindered by the recrystallisation process\textsuperscript{72}.

### 2.3.3 Zener-Hollomon parameter

The Zener-Hollomon parameter ($Z$) (see equation 2.28) captures the effects of both deformation temperature and strain rate on the microstructural evolution during hot working and represents a mathematical expression for a temperature-compensated strain rate. The activation energy may be estimated by the slope ($K$) of the $\ln(\sigma)$ versus $1/T$ plots at various strain (see equation 2.29)$^{58}$. The strain rate sensitivity in turn may be estimated from the slope ($m$) of the $\ln(\sigma)$ versus $\ln(\dot{\varepsilon})$ plots at various strain (see equation 2.30)$^{58}$. The benefit of the Zener-Hollomon parameter is discussed in detail at a later stage (see section 2.3.4).
Expression for the Zener-Hollomon parameter:

\[ Z = \varepsilon' \exp \frac{Q}{RT} \]

where:
- \( Z \) = Zener-Hollomon parameter
- \( \varepsilon' \) = strain rate
- \( T \) = deformation temperature
- \( Q \) = activation energy of the material
- \( R \) = universal gas constant

\[ Q = \frac{KR}{m} \]

Expression for the activation energy of the material:

\[ m = \frac{\partial \ln(\sigma)}{\partial \ln(\varepsilon')} \]

Expression for the strain rate sensitivity of the material:

\[ 2.3.4 \quad \text{Effect of deformation parameters, annealing variables and initial grain size on the size of dynamically and statically recrystallised grains} \]

The deformation parameters, strain, strain rate and deformation temperature have a direct influence on the flow stress of the material. The well-known Hall-Petch relationship indicates that a particular recrystallised grain size is a function of both the material flow stress as well as the initial grain size.

The kinetics of dynamic recrystallisation are accelerated by a smaller initial grain size because there are many high angle grain boundaries, which serve as favourable sites for the nucleation of dynamically recrystallised grains. The amount of stored energy in a deformed material, which provides the driving force for dynamic recrystallisation, also increases with a smaller initial grain size. The rate of nucleation is thus accelerated, dynamic recrystallisation proceeds more rapidly and can therefore be initiated at a lower deformation temperature. The critical strain for the onset of recrystallisation steadily decreases with decreasing flow stress and Zener-Hollomon parameter, and the size of the dynamically recrystallised grains decreases with increasing flow stress, manifested by decreasing deformation temperature and increasing strain rates (increasing Zener-Hollomon parameter).
At intermediate to high strain rates, flow softening in low SFE metals is often attributed to dynamic recrystallisation which arises due to the effects of deformation heating. However, in certain circumstances, adiabatic heating can lead to flow instabilities in the form of adiabatic shear band formation. Dynamic recrystallisation is also favoured by an increase in strain, which would promote a smaller recrystallised grain size. The size of the recrystallised grains will approach a minimum at the strain required for a steady state flow stress and will not change during further deformation. Often the metal finds itself in a partially dynamically recrystallised state and the microstructure is heterogeneous, comprising of both recrystallised and unrecrystallised grains. In such a case, the unrecrystallised and recrystallised grain sizes increase with an increase in the deformation temperature, whilst the unrecrystallised grain size decreases and the recrystallised grain size increases with increasing strain rate. At some critical strain rate the size of the unrecrystallised grains approaches the size of the recrystallised grains and the material becomes homogeneous. A similar trend is observed for an increase in strain, that is, the unrecrystallised grain size decreases and the recrystallised grain size increases with increasing strain until some critical strain is attained where the microstructure becomes homogeneous.

The process of dynamic recovery tends to be hindered at low deformation temperatures, which raises the internally stored energy of the material and thus reduces the annealing temperature required for the initiation of static recrystallisation. The micromechanisms that control static recrystallisation are thermally activated processes and therefore the relationship between the annealing temperature and the rate of static recrystallisation is given by the Arrhenius equation. The annealing temperature at which static recrystallisation starts is thus reduced as the time of the annealing treatment is increased. The stored energy of the deformed material, which represents the driving force for any form of recrystallisation, is a direct function of the amount of deformation or strain. Therefore, both the nucleation and the growth mechanisms are accelerated and the nuclei generated per unit volume of material are greater at a lower annealing temperature for a higher strain, owing to a smaller statically recrystallised grain size.

2.3.5 Kocks-Mecking plots

Figure 2.14 is a schematic representation of a flow curve for a metal that typically undergoes dynamic recrystallisation. The work-hardening rate is given by the slope of the flow curve up to the peak stress. The slope of the flow curve initially increases linearly due to work-hardening, which is associated with an increase in dislocation density, after which the slope gradually changes to a slower linear decline before it rapidly decreases to zero at the peak stress. Initially, it is the dynamic recovery restorative events that are the main causes for the changes in the shape of the flow curve, however, dynamic recrystallisation also commences within this strain regime, which results in a much more rapid and obvious change in the slope of the flow curve. The point defined as the strain required to achieve a steady
state flow stress ($\varepsilon_{\text{ss}}$) is indicative of a stage where the flow stress becomes invariant with further straining, reflecting the balance in the rates of hardening and restoration. The flow stress of a material that only recovers dynamically during deformation is given by $\sigma_{\text{ss}(\varepsilon)}$. Such a material would undergo dynamic recrystallisation without displaying any visible signs of a peak stress. Instead, the flow curve remains smooth before and after the critical strain for dynamic recrystallisation, which suggests that the material undergoes dynamic recovery only. The flow stress continues to increase until such a point that the progress of dynamic recrystallisation is balanced by the ongoing work-hardening events that occur in the unrecrystallised portions of the material, marked by the peak flow stress ($\sigma_{\text{peak}}$) which is attained at the peak strain ($\varepsilon_{\text{peak}}$).

![Schematic diagram of the flow curves for a material that typically undergoes dynamic recovery only and for one that typically undergoes dynamic recrystallisation only during hot deformation (after reference 84).](image)

The Kocks-Mecking plot (see figure 2.15) is of paramount importance as it serves as the only reliable indication of the initiation of subgrain formation associated with dynamic recrystallisation. The procedure involves the plot of the work-hardening rate ($\partial\sigma/\partial\varepsilon$) against the flow stress ($\sigma$). The point of sudden inflection from the linear dynamic recovery regime is indicative of the critical stress ($\sigma_c$), which corresponds to the critical strain required for the initiation of dynamic recrystallisation ($\varepsilon_c$). This noticeable change in the work-hardening rate is associated with the large reduction in flow stress with increasing strain due to the annihilation of dislocations during the nucleation and growth of new strain-free grains within the material. The point where the work-hardening rate intersects the flow stress axis is indicative of the peak stress, beyond which the work-hardening rate is negative as the flow stress begins to drop off significantly due to the rate of dynamic recrystallisation surpassing that of strain-hardening. A plot of $\ln(\partial\sigma/\partial\varepsilon)$ against $\varepsilon$ can also be used to yield a point of inflection that is representative of the
critical strain values$^{73,84,85}$. Alternatively, $-(\partial \varepsilon / \partial s) / \partial \sigma$ may be plotted against $\sigma$ to reveal a more accurate value for the critical strain for dynamic recrystallisation, which is given by the minima in these plots$^{73,85}$.

**Figure 2.15:** Schematic illustration of a Kocks-Mecking plot, showing the work-hardening rate against flow stress for a material that would recrystallise dynamically and for one that would only recover dynamically (after reference 84).

Flow stress tends to follow the variations in strain rate during constant platen velocity uniaxial compression testing due to the high strain rate sensitivity of the flow stress at elevated deformation temperatures$^{84}$. The peak stress, which depicts the balance between restoration and work-hardening events, is very complex under such conditions$^{80,84,85}$. At relatively low strains, the work-hardening of the material is balanced by both dynamic restoration processes and the progressive strain rate softening that occurs as a result of the continuous reduction in strain rate during constant velocity upsetting$^{84}$. At higher strains, the work-hardening is initially complemented by the strain rate hardening response caused by the continuously increasing strain rate, whilst strain rate softening due to decreasing strain rate becomes dominant towards the end of the compression cycle$^{84}$. The interactions between the strain and the strain rate hardening and softening events complicate the peak stress values as the work-hardening rate undergoes a series of sudden increases and decreases in flow stress and the scatter in the work hardening rate is increased (see figure 2.16)$^{73,84,85}$. 
2.3.6 Aspects of nickel-base superalloy forging

It is a general misconception that the development and production of superalloy materials are solely directed at the manufacture of components that are intended to have good creep resistance during service. The capability to withstand creep deformation is the primary function of superalloys but these materials do need to meet other criteria depending on their intended application. When creep is the primary mode of deformation, then the material should be as course-grained as possible. This is often achieved either by a directional solidification technique to produce single crystal materials or by a post-deformation heat treatment in order to facilitate grain growth at high temperature. The aim in both techniques is to maximise the diffusional distance that atoms have to travel as they migrate through the grain itself toward the grain boundaries. Grain boundaries represent regions of high energy that are able to accommodate the accumulation of matter during creep deformation (that is, under conditions of relative low loads and high temperatures). However, a fine-grained material is the more desirable when the loading conditions in service are arduous. This is most commonly achieved by some thermo-mechanical process at the manufacturing stage of the component itself.

Various forming schedules have been developed for the forging of superalloy turbine disks. One such processing route involves the initial ageing/homogenisation treatment of a cast billet followed by a complex multiple-stroke forging operation. The forging process serves to convert the billet into the appropriate shape as well as to control the microstructural evolution and hence mechanical property development of the final item, that is, a fine grained material with \( \gamma' \) dispersed finely and coherently in a \( \gamma \) matrix. In order to achieve this, the processing conditions have to facilitate efficient metal flow during the blow without cracking as well as allow for the generation of the appropriate phases and
microstructure that will yield the desired properties. The γ' solvus temperature, for example, has to be exceeded in order to achieve sufficient ductility and flow stress reduction during the blow. In the same instance, forging either above or below the γ' solvus temperature has a profound affect on the creep ductility of the superalloy material. The conventional forging operation of turbine disk involves a series of initial strokes to reduce the cylindrical billet vertically along its height followed by several subsequent blows and finally a machining operation to form the final article.

During an industrial superalloy turbine disk forging schedule, a cylindrical starting billet is forged in two separate stages known as the preform forge and the finish forge. During performing the upsetting process has to achieve an appropriate mass distribution within the forging mould and the microstructural evolution of the material should be carefully controlled with regard to subsequent forging strokes. During the finishing stage, a disc close to the ultrasonic contour is forged within small tolerances and the forging parameters are controlled so as to avoid any modifications to the microstructure (for example, the suppression of grain growth) that had been developed at the end of the performing stage. The temperature, strain rate and upsetting strain during deformation as well as the lubricant, cooling rates, temperature of the dies and interstroke times and temperatures are carefully controlled to produce the required properties. The material undergoes various softening processes during the forging schedule including dynamic recrystallisation during the strokes, which is the crucial restorative mechanism responsible for large flow stress reductions that facilitate substantial billet reduction without the possibility of crack formation in the article itself. Unavoidable surface frictional forces and inhomogeneous temperature distribution throughout the volume of the billet result in an inhomogeneous distribution in strain (see figure 2.17), which in turn results in an inhomogeneous distribution in microstructural features and mechanical properties. In addition, the deformation parameters are carefully controlled so as to enable good control over the microstructural evolution of the material during the strokes. Since the cooling rates in industrial forging operations are almost always slow, control of the microstructure during the stroke also influences the microstructural evolution during interstroke periods, where static and/or metadynamic recrystallisation are prevalent and often override the effects of dynamic recrystallisation. The refined grain size is attained by achieving a balance between the benefits of billet height reductions and power consumption by the forging tools in overcoming large flow stresses and surface friction as well as the complex relationship between the processing parameters of different forming steps and their respective effects on the microstructural evolution.
Figure 2.17: Finite Element Model output, showing the inhomogeneous strain distribution that arises during the hammer forging of a turbine disk (after reference 88).
CHAPTER 3

EXPERIMENTAL PROCEDURES

3.1 The high temperature compression dilatometer

3.1.1 General set-up and components

During the course of the current study, the Theta® Deformation Dilatometer was used extensively to conduct high temperature deformation experiments. The apparatus utilizes the principle of induction heating to heat a metallic test specimen to a preset temperature and deformation is carried out in uniaxial compression. The high temperature deformation tests that were conducted on the experimental material all adhered to the following procedure:

1. The testing environment was evacuated to achieve a high vacuum of $10^{-5}$ torr.
2. The specimen was heated at a chosen rate to a preset deformation temperature.
3. The temperature of the specimen was held at the deformation temperature for 10 minutes in order to allow the temperature to equilibrate throughout the volume of the specimen.
4. After soaking for 10 minutes at the holding/deformation temperature, the specimen was deformed under variable strain rate uniaxial compression to a preset level of strain.
5. At the end of the deformation cycle, the induction heating process was ceased and helium gas was injected onto the specimen immediately to cool the specimen down to room temperature.

Figure 3.1 is a full view of the high temperature deformation testing apparatus that shows the general setup of the Theta® Deformation Dilatometer. Figure 3.2 shows the interior of the main housing of the Theta® Deformation Dilatometer in greater detail and figure 3.3 is a photograph of the highlighted region in figure 3.2.
Experimental Procedures

The rf (radio frequency) solenoid coil (see figure 3.2 and figure 3.3) is doubly wound. The outer coil is a water-cooled rf coil that is directly connected to the rf generator, which is housed in the main console (see figure 3.1). The inner coil has one closed end and small holes machined over the entire length that allow for the injection of the quenching gas directly onto the specimen. The rf generator feeds the rf coil with an alternating current at high frequency to produce a varying magnetic field, which induces eddy currents in the specimen. The power associated with the eddy currents provides the source of heat to the specimen. The maximum power output of the rf generator is 1kW and the operating frequency is 400kHz.

The thermocouple (see figure 3.4(a)) used to monitor the temperature of the specimen is a type K thermocouple, which has platinum wires and is thus capable of withstanding the harsh experimental environment and hence maintain reliable temperature readings at testing temperatures as high as 1600°C. The thermocouple is spot welded to the surface of the compression specimen in the middle along its height. In order for thermocouple to be spot welded effectively and to ensure that the weld is not damaged significantly during the course of the experiment it is necessary to remove as much of the surface oxide from the specimen as possible before welding.

The platens (see figure 3.3 and figure 3.4(b)) used for deformation are inserted into platen holders that are held against the platen supports with platen clips. The platens are manufactured from quartz glass and are therefore capable of resisting heating by induction (note that the platen surfaces in contact with the specimen are located under the rf coil (see figure 3.2)). The front-end of the fore pushrod (see figure 3.3) rests on the surface of the left platen that is also in contact with the specimen and the front-end of the rear pushrod (see figure 3.3) rests on the lip of the right platen.

During compression, the fluid flow rate through the hydraulic pump (see figure 3.1) is adjusted to move the deformation arm (see figure 3.2) at a desired rate. The deformation arm causes the left platen support (see figure 3.3) to move toward the right whilst the retaining pins hold the right platen support (see figure 3.3) stationary. This movement in turn causes the left platen to move the fore pushrod towards the right and in so doing, the core of the LVDT (Linear Variable Differential Transformer) (see figure 3.2) is disturbed. This disturbance causes the piezoelectric in the LVDT to provide a voltage signal that is related to the amount of displacement that the pushrod sustained during the compression cycle. In the event of the right platen moving due to the platen twisting within the platen holder, the movement of the rear pushrod will compensate for any discrepancy in the displacement measurement. The LVDT has a displacement limit of 2mm from the rest position of the core, which implies that a specimen may be compressed by a maximum of 4mm. If the set displacement is not within this limit, then the deformation arm will, in theory, continue to compress the specimen indefinitely. During compression, the load cell (see figure 3.1), which is located on the deformation arm, provides a voltage signal that is representative of the resistance of specimen to the deformation process. Several compression tests were performed on mild steel specimens.
and excellent correlation was established in the load displacement measurements obtained for separate tests conducted under the same set of deformation conditions.

The specimen, with the thermocouple attached, is carefully inserted between the pair of platens (see figure 3.2, figure 3.3 and figure 3.4(b)). The specimen/platen contact surface is lubricated with circular tantalum foil pieces in order to reduce the surface friction during compression. The foil also limits the extent of pitting as the specimen surfaces tend to stick to the platens at high temperatures. Once the specimen is properly in place, the thermocouple is plugged into the power source provided (see figure 3.2) and the thermocouple wires are carefully aligned flat along the grooves in the left platen (see figure 3.4(a)).

The temperature and time are entered into the computer programme and the temperature control feeds a voltage signal to a thyristor, which in turn sends the amplified signal to the rf generator. The controller then regulates the temperature according to the temperature signal that is read from the thermocouple. Since the rf coil is water cooled and therefore at a much lower temperature than the specimen, care is taken to prevent the thermocouple wires from making contact with the rf coil. When contact is made the voltage signal from the sensor is disturbed due to the large difference in temperature that the thermocouple has to read within a very short period of time. Therefore, a Cu-Be clip was made especially to hold the thermocouple wires firmly in place along the grooves found in the left platen in order to prevent such contact. The weights pulley (see figure 3.2) is adjusted so as to hold the specimen firmly in place between the two platens by means of pulling the two platen supports (see figure 3.3) toward each other. At the end of deformation, the rf generator is switched off and helium gas is pumped directly onto the specimen to quench down to room temperature.

All compression tests are performed under high vacuum at a pressure of about $10^{-5}$ torr. A high vacuum is needed to remove as many oxygen atoms from the testing environment as possible so as to limit the oxidation of the surface of the specimen, thereby preventing any interference with the signal from the thermocouple, and to limit the oxidation of the stainless steel interior of the main housing. This pressure is achieved in a multi-step pump-down sequence, which utilises a standard suction pump and a diffstak pump, which is equivalent to a high pressure diffusion pump. The first stage is known as a roughing phase, which involves the pumping down of the testing chamber (ie. the main housing). When the roughing pressure is about $10^{-2}$ torr then the backing phase is commenced. This involves the pumping down of the bottom section of the diffstak pump. At this point the diffstak pump is heated. After approximately 30 minutes, once the diffstak pump has heated sufficiently, the backing process is stopped and the high vacuum diffusion pump-down is started. Testing can commence when the high vacuum pressure reaches about $10^{-5}$ torr.
Figure 3.1: Photograph showing a full view of the entire apparatus used for high temperature compression testing. The Theta® Deformation Dilatometer is housed at the top of the main console with the data-capturing unit and the control centre located just outside of the field of view toward the left of the main unit.
Figure 3.2: Photograph showing the interior of the Theta® Deformation Dilatometer and the main components located within.

Figure 3.3: Magnified portion of the region within the interior of the Theta® Deformation Dilatometer highlighted in the figure 3.2.
3.1.2 Programme control and data acquisition

(a) System requirements

- control heating rate to a maximum temperature of 1400°C
- hold temperature during soaking period up to temperatures as high as 1400°C
- hold temperature constant during deformation up to temperatures as high as 1400°C
- read voltage signals instantaneously from thermocouple
- perform real-time conversions of voltage signals to temperature values in deg
- record and store temperature values during deformation
- shut down rf generator to commence cooling at the end of overshoot period
- control deformation to a maximum displacement of 4000 microns
- read voltage signals instantaneously from LVDT
- perform real-time conversions of voltage signals to displacement values in microns
- record and store displacement values during deformation
- read/recognise and store negative displacement values due to thermal expansion (during heating and soaking periods)
- provide a displacement reference based on the voltage signal from the LVDT after thermal expansion to initiate the start of deformation
- read, record and store displacement values due to overshoot
• Control deformation up to a maximum of 4000 microns, taking into account negative displacement due to thermal expansion, positive displacement due to the desired nominal displacement input and the excess positive displacement due to overshoot.

• Create a safety measure to abort deformation when maximum displacement exceeds 4000 microns.

• Read voltage signals instantaneously from the load cell.

• Perform real-time conversions of voltage signals to load values in newtons.

• Record and store load values during desired displacement and overshoot periods to a maximum of 5000 newtons.

(b) Programme code architecture

A computer programme was written in Visual C® code to control the operation of the Theta® Deformation Dilatometer, which met all the system requirements (refer to section 3.1.2(a)). Figure 3.5 is a flow chart illustrating the basic code architecture of the programme developed to control the operation of the instrument and to acquire the relevant data during such operation.

The set-up stage involves the definition of the system variables such as the displacement, temperature, load, sampling size; constant parameters such as the displacement safety margin, scaling factors required for measured system variables, sampling rate, overshoot default averaging factors; and system arrays required for the reading and storing of the measured system variables.

The user is required to input three system variables to the mainframe; i.e. heating rate, holding temperature and desired displacement.

Based on the user input and the set-up information, a cut-off displacement is calculated based on the sum of the desired displacement and the safety margin taking into account an estimated overshoot displacement minus an estimated displacement due to thermal expansion of the specimen during heating.

The display in the LVDT window in the control centre shows the amount of displacement that the LDVT can accommodate. This varies with every compression test as the adjustment of the core of the LDVT is extremely sensitive to the positioning of the specimen between the platens. If the cut-off displacement is greater than the LDVT range display, then the test will be aborted otherwise the controller would not have a final displacement as a reference and the instrument will compress the specimen indefinitely until the deformation arm is reset manually. This is to ensure that a platinum specimen, which is extremely expensive and difficult to manufacture, is not wasted in an unsuccessful test. If the cut-off displacement is less than the LVDT range display, then the test proceeds.
The system is commanded to output 5 volts to the deformation arm to commence the compression event. The timer is started and the counter begins to clock.

The voltage signals obtained from the thermocouple, the load cell and the LVDT are sampled and scaled to provide values of temperature, load and displacement.

At this point the counter clicks at increment of one. If the counter exceeds 60 000 clicks then the test is aborted as it is unlikely that, at the given built-in sampling rate, a compression cycle would exceed 60 000 samples. Once again, this is to ensure that the deformation arm can be reset manually as it was likely to have compressed the specimen beyond the level of the desired displacement. If the counter is less than 60 000 clicks then the test proceeds.

The system is commanded to output 0 volts to the deformation arm in order to deactivate the compression. The timer is stopped and the counter ceases to click.

At this point a status command is established. If the status is "2" then the desired displacement was not achieved and the test is aborted. If the status is "1" then the desired displacement was achieved and the programme proceeds to ascertain the overshoot. During the overshoot period, the rate of increase in the displacement decreases with increasing sampling count. If the rate of increasing displacement is greater than the default overshoot setting (set at zero) then the load, displacement and temperature values are recorded and stored as temporary values. The record of temporary values halts when the rate of increasing displacement starts to become negative (that is, the rate at which displacement accumulates decreases) due to a springback effect of the deformation arm as compression halts.

At this point the mainframe is exited, which triggers the deactivation of the rf generator.

The following information is displayed as arrays:

- number of samples
- sampling time
- number of overshoot samples
- sampling time for overshoot
- starting displacement
- cutoff (desired) displacement
- final (actual) displacement
Figure 3.5: Flow chart showing the code architecture of the programme used to control the Theta Deformation Dilatometer.
3.1.3 Temperature control

The following problems with regard to the temperature control were experienced upon the commencement of testing:

1. Heating was achieved without any temperature control (i.e., the rf generator appeared to feed the specimen with either maximum power or zero power).

2. Temperature was not controlled during the holding/soaking period (i.e., temperature fluctuated drastically about the set holding temperature).

3. Temperatures above 1250°C were not attainable.

In order to solve these problems, knowledge had to be gathered on the phenomena of induction heating and the fundamentals of temperature control.

(a) Heating phenomena in the Theta® Deformation Dilatometer

The initial stage of heating is predominantly by induction. The rf generator provides the rf coil with an alternating current at a frequency of 400kHz to produce a varying magnetic field. The magnetic field threads through to the specimen to induce a voltage therein. Eddy currents flow in the specimen and power is consumed in the resistance to form the basis of heat generation. Skin effect is a phenomenon inherent to induction heating. It involves the generation of eddy currents at the surface of the specimen, within the specimen itself. The heat does not have to pass through a surface layer of high thermal resistance and therefore the induction heating process is much more rapid than any form of direct electrical resistance heating. As time progresses during heating, the heat is conducted from the surface of the specimen toward the centre. The heat probably does not equilibrate within the specimen before it can be conducted from the specimen to the platens and radiated to the environment. In order words, heat is continually and dynamically being supplied to the specimen surface by induction and conducted to the centre of the specimen and toward the platens. Therefore, the rate at which heat has to be generated by induction decreases with time as the thermal gradient between the surface and the centre of the specimen levels off and as the heat is slowly conducted to the platens and radiated to the environment.
(b) Temperature control

The temperature control must:

1. Receive a voltage signal provided by the thermocouple representing the actual measured temperature.

2. Compare this signal with another voltage signal representing the desired temperature.

3. Initiate a corrective action based on this comparison.

Set-point

The basic function of the temperature controller is to adjust the power that is output from the rf generator to the rf coil by means of comparing the actual temperature of the specimen measured (Tₐ) to that of the set-point (Tₛ), which is the comparative voltage signal that must be provided by the controller. The set-point temperature in this system is a manual setting, which is the most simple and inexpensive way of achieving a set-point signal.

On-off control

On-off control involves an electronic response whereby the power output is either completely on (representing the maximum power output) or completely off (representing the minimum power output), which is the simplest form of control action. When thermal losses are steady state, then on-off control manifests in equally spaced intervals of on and off cycles, but thermal lag often results in a periodic temperature response (see figure 3.6).
The power output is temporarily turned off when $T_s$ reaches $T_c$. Then, it continues to rise until a point is reached whereby the heat that has already been delivered to the specimen is sufficiently dissipated to the platens and environment so as to cause the temperature of the specimen to drop, during which the power output remains off. At this point, the power is turned on maximum output again and $T_s$ increases until $T_i$ is reached again, thereby initiating another cycle. The period of a cycle is dependent on the time constant of the heating and cooling processes and the respective slopes that represent the heating and cooling rates, which are always very different.

**Proportional bandwidth control (p-constant/gain)**

The proportional bandwidth is the fractional percentage of the instrument over which the full range of power control acts. This control action attempts to achieve better response than on-off control by means of applying power to the difference in $T_i$ and $T_c$. Equation 3.1 is the expression that is used to determine the actual power output delivered ($W$) when incorporating the effects of proportional bandwidth control, where $P$ is the proportional gain (in percent) of the temperature programme controller.

$$W = P(T_i - T_c)$$

*Equation 3.1*
The effect of widening the proportional bandwidth is demonstrated in the temperature versus time outputs shown in figure 3.7(a) to figure 3.7(f). The curves show that widening the proportional bandwidth reduces the tendency for overshoot in $T_o$. This is achieved by means of allowing for faster responses to the changes in $T_o$. However, the curves also indicate that this reduces the sensitivity of the temperature control and the response is underdamped progressively and eventually reaches instability.

![Temperature versus time curves illustrating the effect of the proportional bandwidth control action on the heating response of the metal specimen.](image)
**Integral control (I-constant)**

The error signal that exists after proportional bandwidth control is amplified and integrated until $T_e$ is reached. Equation 3.2 is the expression used to determine the actual power output delivered (W) by incorporating the corrective effects of both proportional bandwidth control and integral control, where $I$ is the integral gain parameter (in percent).

$$W = P[(T_s - T_o) + I](T_e - T_o)dt$$

*Equation 3.2*

The effect of increasing the integral control is shown in figure 3.8(a) to figure 3.8(d). The integrated error signal is then used to provide an additional output control signal upon heating in order to hold the process at the desired temperature and so doing, the peak temperature is reduced with increasing $I$.

![Figure 3.8](image-url)  
*Figure 3.8: Temperature versus time curves illustrating the effect of the integral control action on the heating response of the metal specimen.*
Derivative control (I)-constant

The overshoot and instability associated with proportional bandwidth control is compensated by derivative control. The signal is differenti ated and provides an additional output control action upon cooling. Figure 3.3 is the expression used to determine the actual power output delivered (W) by incorporating the corrective effects of proportional bandwidth control, integral control and derivative control, where D is the derivative gain parameter (in percent).

\[
W = P \left[ T_s - T_o \right] + \int \left[ T_s - T_o \right] dt + D \frac{\partial}{\partial t} \left( T_s - T_o \right)
\]

Equation 3.3

The effect of derivative control is shown in figure 3.9(a) and figure 3.9(b). Derivative control opposes the corrective action of proportional bandwidth and integral control by assisting in dampening the oscillations that accompany narrow proportional bandwidths.

![Figure 3.9](image_url)

Figure 3.9: Temperature versus time curves illustrating the effect of the derivative control on the heating response of the metal specimen.

(c) Final formulation

PID (proportional bandwidth, integrated time, derivative time) constants were used to address the aforementioned problems associated with the heating capabilities of the instrument, which seemed characteristic of on-off control.

Finally, given the complexity in the sequence of heating events taking place in the specimen as previously described, it was found necessary to use more than one PID group to achieve an optimum heating
response. Initially, three PID groups with two PID boundary points were used. The boundary points represent the transitions in temperature at which the PID constants changed according to their settings.

Generally, extremely wide proportional bandwidths (between 200 and 350) and very short integral and derivative constants (between 1 and 3 and 3 and 9 respectively) had to be programmed. This was based on the rapid heating rates involved in induction heating. The proportional bandwidths were increased with increasing PID group, given the relatively slower heating rates associated with equilibration of heat within the specimen as time progresses. Accordingly, the time constants (both integral and derivative) were reduced with increasing PID group. Table 3.1 is a summary of PID constants programmed for the system (with an additional PID group that was added for a finer tuning to the heating response).

<table>
<thead>
<tr>
<th>PID group</th>
<th>PID boundary</th>
<th>P</th>
<th>T</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>200</td>
<td>1</td>
<td>9</td>
</tr>
<tr>
<td>2</td>
<td>600°C</td>
<td>250</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>800°C</td>
<td>300</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>1100°C</td>
<td>350</td>
<td>4</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 3.1: Final PID constants for achieving optimum heating response.

The PID constants gave an excellent response in the heating of platinum specimens in the Theta® Deformation Dilatometer. The resultant control includes:

1. Near-perfect temperature tracking in T, with respect to T, between 800°C and 1400°C
2. A maximum overshoot of 5°C for a period of 5 seconds
3. A holding capability of ±0.5°C during soaking
4. Up to 1300°C in 120 seconds and 1400°C in 190 seconds with the response mentioned in 1, 2 and 3 above.
3.2 Material

Three platinum-base alloys were provided by Mintek with the following percentage atomic compositions:

1. Pt\textsubscript{10}Al\textsubscript{10}Ru\textsubscript{4}
2. Pt\textsubscript{10}Al\textsubscript{10}Cr\textsubscript{4}
3. Pt\textsubscript{10}Al\textsubscript{24}Cr\textsubscript{3}Ru\textsubscript{4}

3.2.1 Material history

The individual metal elements were arc melted and cast into a mould. The molten cast was stirred and allowed to solidify in an inert environment. The solid cylindrical specimens were then manufactured from the cast billets using specialised tooling equipment.

3.2.2 Heat treatments

The cast alloys were homogenised at 1300°C for 96 hours under argon gas, after which they quenched in water. Post-stress annealing heat treatments were performed at 1200°C under argon gas at varying times, followed by a water quench.

3.3 Microscopy

3.3.1 Specimen sectioning

The compressed barrel-shaped specimens were sectioned in the centre along the axes parallel to the applied load using a microslicer. Buckled specimens were sectioned as close to the centre as possible.

3.3.2 Specimen preparation

The sectioned specimens were hot mounted. The hot mounted specimens were ground to a 1200 grit finish and polished using diamond paste to a 1 micron finish. Note that the contact ends of homogenised compression specimens were ground on 600 grit paper to remove as much of the oxide as possible. However, grinding was not particularly successful in removing all the oxide as excessive grinding would
easily have introduced bevels into the contact ends. Bevelled ends result in specimen buckling during uniaxial compression testing.

The sectioned specimens were electro-etched in a solution containing 25g NaCl crystals dissolved in 20ml of 33% dilute HCl with 90ml of distilled H2O at 6-9V using alternating current and a graphite counter-electrode for 45-90 seconds. This etching technique gave satisfactory etched surfaces for optical microscopy and scanning electron microscopy.

### 3.3.3 Microscopy techniques

Optical microscopy was conducted on the Leica® microscope using Nomarski interference contrast for phase and grain boundary enhancement. Scanning Electron Microscopy (SEM) with electron backscattered detection (EBSD) and secondary electron detection (SED), Electron Diffraction Spectroscopy (EDS) with EBSD and x-ray mapping with EBSD was conducted on the Leica® 440 microscope.
CHAPTER 4

RESULTS I

ANALYSIS OF LABORATORY FLOW DATA

4.1 Introduction

A series of high temperature compression tests has been performed on the experimental materials using the Theta® Deformation Dilatometer. This chapter deals with the analysis of the laboratory flow data gathered during the compression testing exercise, in an attempt to assess the value of such data in providing insight to the hot deformation behaviour of the material. This is in view of the fact that often microstructural observations cannot alone provide conclusive details concerning certain aspects of laboratory simulated metal forming operations and hence the analysis of flow data becomes the only reliable means of assessing the flow behaviour of the material. The manner in which the flow data analysis is presented in this chapter allows the reader to assess the appropriateness of the specific apparatus employed in providing useful information concerning the hot formability of platinum-base alloys.

4.2 Calculation of strain rates

In order to show more quantitative results concerning the effects of the compression testing variables on the microstructural evolution of the material, it is imperative firstly to determine the strain rates during each compression test. This task is relatively simple under conditions of constant strain rate testing, during which the velocity of the deformation ram decreases at an exponential rate so as to provide a constant true strain rate throughout the course of deformation. Under such conditions, the strain rate of interest is simply given by the final ram velocity divided by the nominal strain input. However, in the current study, calculation of the strain rate is slightly more complex, as the strain rate changes continuously with strain. The change in the strain rate increases exponentially with increasing strain, which is a consequence of the exponential increase in true strain with the increasing reduction of the specimen height during the upsetting process.
Ideally, an individual strain rate should be computed and quoted for every strain increment as sampled from the LVDT during constant velocity compression testing. However, this would make subsequent calculations (such as the calculation of the Zener-Hollomon parameter (see section 4.6), which requires a single value of strain rate and temperature), nearly impossible. Consequently, it was decided to quote a single value of strain rate for each compression test which would best describe the actual strain rate condition. The displacement data that was logged from the LVDT was converted to true strain data using equation 2.5 and plotted against the respective time increments (see figure 4.1 and figure 4.2). Figure 4.3 represents a magnified portion of the curve shown in figure 4.1(a) at 0.06/s. Since individual values of
strain rate are determined by taking the gradient at each time increment, it is important that the scatter in the raw data shown in figure 4.3 be reduced as the incremental values of gradients are very sensitive to the exact values of true strain. It for this reason that the raw data was filtered so as provide better quality in the true strain data used to compute the incremental gradients of strain rates. Once incremental values of strain rates were determined from the filtered true strain data, the values were summed and divided by the number of samples to produce a quantity that can best be described as a mean nominal strain rate (\( \dot{\varepsilon}_n \)).

When conducting an investigation on the influence of the testing variables on the high temperature strength values, it is necessary to once again determine suitable values of strain rates. This is done by means of employing the same method that was used to determine the mean nominal strain rate except that incremental gradients are of course computed up to the level of true strain that corresponds to the high temperature strength value (i.e. the yield point). This value of strain rate, which is described as the mean strain rate (\( \dot{\varepsilon}_m \)), is quoted as an approximate value because of the unavoidable inaccuracies that arise when using the small quantities of elastic strain in the computation of the gradients required for the determination of \( \dot{\varepsilon}_n \). The mean nominal strain rates (\( \dot{\varepsilon}_n \)) determined from this procedure and the corresponding rounded-off values (\( \dot{\varepsilon}_m \)) as well as the mean strain rates (\( \dot{\varepsilon}_m \)) that correspond to the strain rates at the high temperature strength values have been quoted in table 4.1 for easy reference.

![Plots of true strain against sampling time for PtAl_{13}Ru_4 deformed at a deformation temperature of 1400°C. The plots represent a magnified portion of the data shown in figure 4.1(a) at 0.06s.](image-url)
## Table 4.1: Tabulation of deformation temperature \( T \), mean nominal strain rate \( \dot{\varepsilon} \) and mean nominal strain rate estimate \( \dot{\varepsilon} \) for each compression test for Pt<sub>90</sub>Al<sub>10</sub>O<sub>4</sub> and Pt<sub>80</sub>Al<sub>10</sub>Cr<sub>10</sub>. \( \dot{\varepsilon} \) is the exact value determined from the true strain-against-time plots at the maximum level of strain experienced during the compression test and \( \dot{\varepsilon} \) is the value of \( \dot{\varepsilon} \) rounded off. \( \dot{\varepsilon} \) is an estimate of the strain rate experienced at the level of strain corresponding to the high temperature strength.
Strain rates of 0.01/s, 0.1/s, 0.5/s and 1/s for Pt<sub>0.1</sub>Al<sub>0.1</sub>Ru<sub>0.1</sub>, and 0.1/s and 1/s for Pt<sub>0.1</sub>Al<sub>0.1</sub>Cr<sub>0.1</sub> were the initial targets at the outset of the series of compression tests. The relatively poor displacement control of the hydraulic pump made it impossible to achieve these exact values of strain rates, however, it was possible to obtain values of 0.06/s, 0.1/s, 0.5/s and 1.2/s for Pt<sub>0.1</sub>Al<sub>0.1</sub>Ru<sub>0.1</sub>, and 0.1/s and 1.1/s for Pt<sub>0.1</sub>Al<sub>0.1</sub>Cr<sub>0.1</sub> (see table 4.1) by adjusting the rate of fluid flow through the pump. The excess strain accumulated during compression at 1400°C (at 0.5/s and 1.2/s) for Pt<sub>0.1</sub>Al<sub>0.1</sub>Ru<sub>0.1</sub> and at 1400°C (at 1.1/s) for Pt<sub>0.1</sub>Al<sub>0.1</sub>Cr<sub>0.1</sub> is a result of the unavoidable overshoot in displacement that occurs at higher ram velocities where displacement control is increasingly compromised.
4.3 Fundamental Stress Analysis

The load-displacement data that was logged during compression testing is shown in figure 4.4 and figure 4.5 under the different deformation conditions. This information is used to compute basic mechanical stresses, namely the axial, hoop, hydrostatic and representative stresses according to equation 2.6, equation 2.11, equation 2.12 and equation 2.13 respectively. Figure 4.6 and figure 4.7 are plots of hoop strain against axial strain, which give slopes that are necessary for the computation of the hydrostatic and representative stresses according to equation 2.12 and equation 2.13 respectively. These stress quantities, which form the basis for subsequent analysis, are plotted against the axial strain and are shown in figure 4.8 and figure 4.9 for Pt65Al30Ru5 and in figure 4.10 for Pt65Al30Cr5.

![Load-displacement plots](image1)

**Figure 4.4:** Plots of load against displacement for each compression test conducted for Pt65Al30Ru5 at 1400°C and varying strain rates (a) 0.1/s and (b) varying deformation temperatures.

![Load-displacement plots](image2)

**Figure 4.5:** Plots of load against displacement for each Pt65Al30Cr5 specimen compressed to significant strain (i.e., $\varepsilon > 0.3$) at varying deformation conditions.
Figure 4.6: Plots of hoop strain against axial strain for each compression test for Pt$_6$Al$_{13}$Ru$_4$ at varying strain rates (a) and deformation temperatures (b). The values of $a$, which are used in the calculations of the hydrostatic and representative stresses, are given by the respective slopes of the curves.

Figure 4.7: Plots of hoop strain against axial strain for each Pt$_6$Al$_{13}$Cr$_4$ specimen compressed to significant strain (i.e. $e>$0.3) at varying deformation conditions. The values of $a$, which are used in the calculations of the hydrostatic and representative stresses, are given by the respective slopes of the curves.
Figure 4.8: Plots of axial, hoop, hydrostatic and representative stresses against axial strain for PtωM13Ru, at varying strain rates at 1400°C. Note that the compression axis is used as the reference for stress exerted in the positive direction.
Figure 4.9: Plots of axial, hoop, hydrostatic and representative stresses against axial strain for Pt$_{20}$Al$_{10}$Ru$_{4}$ at varying deformation temperatures at 0.1 s. Note that the compression axis is used as the reference.
Figure 4.10: Plots of axial, hoop, hydrostatic, and representative stresses against axial strain for Ti6Al4V at varying deformation conditions. Note that the compression axis is used as the reference for stress exerted in the positive direction.

4.4 Friction

4.4.1 Inhomogeneous deformation

Unavoidable friction at the loading face during uniaxial compression testing inhibits the free flow of metal directly beneath the platens, thereby forcing the compression specimen to bulge outward into a barrel shape. The result of this change in specimen geometry is an inhomogeneous accumulation of metal and an inhomogeneous distribution in grain morphology throughout the cross-section of the material as depicted in figure 4.11. Metal flow is heavily retarded in zone A, which known as the dead zone, and the material remains relatively undeformed with a microhardness of nearly that of the original material. In zone D, the
material is free to flow outward and accumulates to a certain extent. The microhardness is slightly higher than that of the original material and of that in zone A. Metal shear causes significant accumulation of material in zone B, where the microhardness is significantly higher than that in zones A and D. The measured microhardness is always highest in the centre of the specimen in zone C where the pileup of material is the most severe.

**Figure 4.11:** Light micrographs showing the variation in the microstructural profile over the cross-section of a deformed specimen due to the barrelling effect.
4.4.2 Finite Element Modelling (FEM)

(a) Introduction

Upon the initial inspection of the uniaxial stress data and from the variable microstructural profile observed across the specimen cross-section, it seems that the presence of friction at the loading interface between specimen and deformation platen gives rise to specimen barrelling and hence an inhomogeneous deformed microstructure. This means that any attempt to relate the deformed microstructure to its corresponding flow data would require an appropriate adjustment of the flow data according to the specific frictional condition experienced during the specific compression test. Such adjustments are often based on the use of flow stress correction models (see section 4.4.3) and require the input of a suitable coefficient of friction.

In addition, the deformed microstructure is dependent on the magnitude of strain. Barrelling results in an inhomogeneous strain distribution across the specimen cross-section such that the nominal strain input is not equal to the local strain where metallographic analysis of the deformed microstructure is conducted. It is therefore necessary to solve the local strain values so that an appropriate level of strain can be correlated to the friction-corrected flow data and deformed microstructure.

(b) Description

Finite Element Modelling (FEM) is a mathematical tool which is widely employed in solving complex multi-variable physical problems numerically. It was decided upon to develop an FEM using the software ABAQUS® 6.2 Standard in order to solve both the coefficient of friction and the local strain values for each compression test. The first step is the re-creation of the starting solid cylindrical specimen structure in two dimensions, which is represented as the rectangular structure as shown in figure 4.12(a).

The geometry is then discretised in a sequence of finite elements, by which each element is representative of a discrete portion of the actual continuous physical structure. The finite elements are joined by a series of nodes to form an enclosed mesh. ABAQUS® 6.2 Standard utilises an implicit analysis, meaning that a Newton-Raphson incremental-iterative method is used to output solutions to non-linear problems. Unlike a linear problem where a solution can be provided by solving a single system of equations, non-linear problems require either incremental times or load stepping. A convergence in the time/load increment is obtained within each iteration before proceeding to a subsequent iteration. Since the specimen is symmetrical, it is only necessary to model one eighth of the structure. This is less cumbersome than modelling the entire structure and also reduces the computational and simulation times substantially.

Boundary conditions are set at all the borders of the structure. The lower end of the structure is fixed and the upper end is subjected to a variable velocity, whilst the outer boundary is free to deform and expand laterally. The upper boundary condition, which is entered as a variable velocity, is based on the velocity
calculated from the displacement-against-sampling time plots as a function of reducing specimen height. The velocity data is then incorporated into the FEM via a short subroutine encoded in FORTRAN® so that as the specimen height is reduced by an increment the FEM assumes the value of velocity that corresponds to the new specimen height. The loading interface is also subjected to Tresca friction, which causes lateral nodal displacement. ABAQUS® 6.2 Standard uses the nodal displacements in its analysis and computation of the strain within each discrete element. The final input parameter is the uncorrected flow data, that is, the uniaxial stress and uniaxial strain that is calculated from the raw load-displacement data, which is specific to each compression test. The FEM is viscoplastic, meaning that deformation is plastic in nature. Although the FEM does require the input of Young's Modulus in order to run, the exact value has no bearing on the simulated results. It is for this reason that Young's Modulus was simply taken as the room temperature Young's Modulus for pure platinum.

(c) Solution and output procedure

The procedure for solving the coefficient of friction involves the iterative input of values for the Tresca coefficient of friction until the geometry of the simulated deformed structure fits that of the actual deformed specimen. The values of the Tresca coefficient of friction, the outer barred diameter of the deformed specimen actually measured, the outer barred diameter of the deformed structure simulated, the nominal strain input and the localised strain is given in table 4.2. Although the FEM is two-dimensional in its construction, it is possible to output strain in three dimensions. The local strain values quoted are the simulated strain values in the direction of the compression axis, which is consistent with the metallographic analysis conducted in the plane of the deformed specimen sectioned vertically along the compression axis. Note that all simulated strain outputs are logarithmic strain (LG) and not plastic equivalent strain values (PEEQ) as is frequently reported in similar works that have been conducted by other researchers such as Sheen® and Buchanan®. It seems more suitable to output LG strains from the FEM as the flow data input is based on the true axial strain, which is logarithmic in nature. Output of PEEQ would imply that the input and output data are not physically consistent. The development of localised strain within the simulated specimen during uniaxial compression is depicted in figure 4.12. As shown in figure 4.13, the specimen is seen to deform homogeneously in perfect uniaxial compression and the solid cylindrical shape of the specimen is maintained at the end of the compression cycle. The plots in figure 4.14 show the linear
dependence of the localised strain development at the specimen centre on the nominal strain under different deformations conditions. The similarity in the gradients of these plots at different deformation conditions indicates that the development of localised strain is independent of the deformation temperature and strain rate. The plots in figure 4.15 show the development of localised strain against the nominal strain under different conditions of friction. Clearly, the friction condition does influence the development of the localised strain at the specimen centre.

<table>
<thead>
<tr>
<th>test</th>
<th>conditions</th>
<th>( \tau )</th>
<th>( d_{o1} ) (mm)</th>
<th>( d_{o2} ) (mm)</th>
<th>( d_{cn} ) (mm)</th>
<th>( d_{cw} ) (mm)</th>
<th>( s )</th>
<th>( \varepsilon )</th>
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<tbody>
<tr>
<td>1. Pt(<em>{99})Al(</em>{1})Ru(_{4})</td>
<td>1400°C, 0.96/s</td>
<td>0.45</td>
<td>6.2</td>
<td>6.2</td>
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<td>5.0</td>
<td>0.51</td>
<td>1.38</td>
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<td>5.7</td>
<td>5.1</td>
<td>4.9</td>
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<td>1.49</td>
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<td>5.3</td>
<td>5.4</td>
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<td>5.3</td>
<td>0.69</td>
<td>1.96</td>
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<tr>
<td>5. Pt(<em>{99})Al(</em>{1})Ru(_{4})</td>
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<td>5.7</td>
<td>5.7</td>
<td>5.2</td>
<td>4.9</td>
<td>0.51</td>
<td>1.49</td>
</tr>
<tr>
<td>6. Pt(<em>{99})Al(</em>{1})Ru(_{4})</td>
<td>1250°C, 0.1/s</td>
<td>0.4</td>
<td>5.7</td>
<td>5.7</td>
<td>5.1</td>
<td>4.9</td>
<td>0.51</td>
<td>1.49</td>
</tr>
<tr>
<td>7. Pt(<em>{99})Al(</em>{1})Ru(_{4})</td>
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<td>5.5</td>
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<td>4.5</td>
<td>0.43</td>
<td>1.31</td>
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<tr>
<td>8. Pt(<em>{99})Al(</em>{1})Cr(_{4})</td>
<td>1400°C, 1.1/s</td>
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<td>6.3</td>
<td>5.3</td>
<td>5.1</td>
<td>0.63</td>
<td>1.87</td>
</tr>
<tr>
<td>9. Pt(<em>{99})Al(</em>{1})Cr(_{4})</td>
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<td>6.2</td>
<td>6.2</td>
<td>5.3</td>
<td>5.0</td>
<td>0.51</td>
<td>1.38</td>
</tr>
<tr>
<td>10. Pt(<em>{99})Al(</em>{1})Cr(_{4})</td>
<td>1300°C, 0.1/s</td>
<td>0.45</td>
<td>6.2</td>
<td>6.2</td>
<td>5.3</td>
<td>5.0</td>
<td>0.51</td>
<td>1.38</td>
</tr>
</tbody>
</table>

Table 4.2: Tabulation of the numerical output from the FEM for Pt\(_{99}\)Al\(_{1}\)Ru\(_{4}\) and Pt\(_{99}\)Al\(_{1}\)Cr\(_{4}\) compressed at varying deformation temperatures and strain rates, where:
- \( \tau \) - the Tresca coefficient of friction
- \( d_{o1} \) - outer barrelled diameter of the deformed specimen actually measured
- \( d_{o2} \) - outer barrelled diameter of the deformed structure simulated by the FEM
- \( d_{cn} \) - contact diameter of the deformed specimen actually measured
- \( d_{cw} \) - contact diameter of the deformed structure simulated by the FEM
- \( s \) - nominal input strain
- \( \varepsilon \) - effective local strain at centre of the structure simulated by the FEM
Figure 4.12: FEM simulations (the legend indicates the level of logarithmic strain) of $\text{Pb}_0.5\text{Al}_{0.5}\text{Ru}_4$ compressed at 1350°C at 0.1/s at increasing nominal strains.
Figure 4.13: FEA outputs showing the geometry of the simulated deformed structure for an input Tresca coefficient of zero and a specimen height reduction of 40%.

Figure 4.14: Plots of simulated localised logarithmic strain against nominal true strain input for P\textsubscript{w}M\textsubscript{w}Ra\textsubscript{d}, showing the independence of the localised strain on the deformation conditions.
Figure 4.13: Plots of the simulated localised logarithmic strain against the nominal true strain input for P₁₀₀₀₁₀₀Ru, upset by a 40% height reduction. The graph shows the effect of the magnitude of the Tresca coefficient of friction on the magnitude of the local strain developed in the centre of the simulated structure at the completion of the deformation cycle.

4.4.3 Flow stress correction due to friction

The flow stress of a metal is the most common and widely used material property that is assessed during the analysis of metal forming operations. It is given by the true axial stress, which is determined from the axial load (or the force that acts parallel to the principle loading axis in uniaxial compression) and the cross-sectional area over which the load is exerted. The cross-sectional area is calculated on the basis that the specimen deforms homogeneously during uniaxial compression, that is, the specimen retains its cylindrical geometry throughout the loading event. Friction, however, as previously mentioned, causes the specimen to barrel during deformation, meaning that the cross-sectional area is no longer given by equation 2.7. Furthermore, the continuous increase in load with strain suggests continuous work-hardening during the compression event, whereas it is actually an artifact of surface friction. Therefore, it is necessary to elucidate the effects of friction on the flow data as best as possible, in order to assess the actual behaviour of the material during hot working based on such data. The flow stress is corrected for friction as shown in figure 4.16 and figure 4.17 for P₁₀₀₁₀₀Ru, and in figure 4.18 for P₁₀₀₁₀₀Cr, according to the models described by Gleble, Gleble II, the von Mises yield criterion and the Siebel correction function, which are given by equation 2.15, equation 2.16, equation 2.17 and equation 2.20. The Tresca coefficients of friction that are solved by the FEM are used in the models to adjust the flow stress appropriately according to the particular compression test. The method of treating the problem of friction during uniaxial compression by adjusting flow stresses with the use of friction models is consistent with that proposed by Parted and Bünten. 39
Figure 4.16: Flow curves at 1400°C and varying strain rates for Pb61Al39Ru4, corrected for friction according to various friction models.
Figure 4.17: Flow curves at 0.1/s and varying deformation temperatures for PtAlRe, corrected for friction according to various friction models.
The output for the specimen outer diameter with strain for the various friction models is shown in figure 4.19 and figure 4.20 for Pt66Al16Cr4 and in figure 4.21 for Pt66Al16Cr4. The curves show that the Gleeble I, Gleeble II, the von Mises yield criterion and Siebel corrective models do not predict the values for the initial and final outer diameters of the compression specimen within close range of the actual diameters measured. However, the barrelling model (see equation 4.1) gives excellent correlation between the simulated and actual initial and final outer diameters of the compression specimen. Consequently, the entire flow curves are adjusted according to the corrected flow stress according to the barrelling model as shown in figure 4.22 and figure 4.23 for Pt66Al16Ru4 and in figure 4.24 for Pt66Al16Cr4.
\[ d_j = \left\lfloor 0.5 \left( 3d_0^2 \frac{h_0}{h_f} - d_0^2 \right) \right\rfloor^{1/3} \]

**Equation 4.1:** Expression for the barrelling model used to adjust flow data for the effects of friction, where:
- \( h_0 \): initial height of specimen
- \( h_f \): final height of specimen
- \( d_0 \): initial diameter of specimen
- \( d_c \): centre diameter of specimen
- \( d_f \): final outer diameter of specimen

**Figure 4.19:** Plots showing the variation of the specimen outer diameter with true strain for Pi60/Mg.Re4 as predicted for homogeneous deformation (homo), the barrelling model (bar I), the Gleebol \textsuperscript{®} I friction model, the Gleebol \textsuperscript{®} II friction model, the von Mises yield criterion and the Siebel corrective function at 1400°C and varying strain rates.
Figure 4.20: Plots showing the variation of the specimen outer diameter with true strain for P123Al0.85Ru4 as predicted for homogeneous deformation (homo), the barreling model (bar II), the Gleeb1 friction model, the Gleeb1 II friction model, the von Mises yield criterion and the Siebel corrective function at 0.1/s and varying deformation temperatures.
Figure 4.11: Plots showing the variation of the specimen outer diameter with true strain for Pt6Al14Cr as predicted for homogeneous deformation (homo), the barrelling model (bar I), the Gleeble\textsuperscript{TM} friction model, the Gleeble\textsuperscript{TM} II friction model, the von Mises yield criterion and the Siebel corrective function at varying deformation conditions.
Figure 4.22: Flow curves for PtAl₁₂Ru₄ as predicted for homogeneous deformation (homo) and the barrelling model (bar 1) at 1400°C and varying strain rates.

Figure 4.23: Flow curves for PtAl₁₂Ru₄ as predicted for homogeneous deformation (homo) and the barrelling model (bar 1) at 0.1/s and varying deformation temperatures.
4.5 Flow stress correction due to deformation heating

Deformation heating refers to the work that is generated in the form of heat during the deformation of metallic materials. During mechanical straining at intermediate to high strain rates (\( \dot{\varepsilon} \geq 0.1/s \)), there is insufficient time available for the full transfer of deformation heat away from the specimen. Consequently, the temperature of the specimen rises and in some instances, this temperature increase can significantly affect the flow behaviour of the material. Equation 2.27 is used to analytically determine the temperature rise due to deformation heating (\( \Delta T \)). Note that during the analysis, the temperature rise due to deformation heating is taken as adiabatic heating only, that is, the value of \( \Delta T_{\text{adiabatic}} \) determined from equation 2.27 is taken as \( \Delta T_{\text{actual}} \).

The flow stress values for each compression test are filtered so as to reduce the associated scatter. The area under each flow curve, which gives the value of EDE for a specific set of deformation conditions, is then estimated using the method of Riemann summing. This method involves the construction of rectangular units at small increments of strain as illustrated in figure 4.25. The area of each rectangular unit can then easily be calculated and the sum of the calculated areas for all the units provides an estimate of the total area under the flow curve (EDE). The values of EDE determined by this method are then used in conjunction with the room temperature values of specific heat (\( C_p \)) and density (\( \rho \)) for pure platinum along with the appropriate values for \( \gamma \) in the determination of \( \Delta T \). The change in \( \Delta T \) with strain is shown in figure 4.26 for the various deformation conditions.
Figure 4.25: Magnified portion of a filtered flow curve illustrating the procedure of Riemann summing that is used for determining the area under the flow curve, which is representative of the EDE. The rectangular constructs are used to approximate the area under the flow curve within a selected strain increment.

Figure 4.26: Plots of the rise in temperature due to adiabatic heating against true strain for $\dot{\varepsilon}_{1250^\circ C}$.

It was attempted to reduce the scatter in the $\ln(\sigma)$ versus $1000/T$ plots by excluding the data of the compression test that was conducted at $1250^\circ C$. The adiabatic stress values (i.e., the stress values that are not corrected for deformation heating effects) at $1250^\circ C$ are too large, in relation to the adiabatic stress values at the other deformation temperatures, to include in the $\ln(\sigma)$ versus $1000/T$ plots and would thus increase the scatter in such plots. The curves shown in figure 4.27 and figure 4.28 illustrate the scatter associated with such plots with and without including the data at $1250^\circ C$ respectively. The improvement in the scatter is demonstrated in the shape of the curves shown in figure 4.28. The isothermal curves that are
constructed using the values of flow stress that were extrapolated from figure 4.28 are shown in figure 4.29.

![Figure 4.27](image)

**Figure 4.27:** Plots of \(\ln(\sigma)\) against \(1/T\) at different strains for \(\text{Fe}_2\text{Al}\). Used to determine the values of isothermal stress at different strains. The isothermal stress is determined by extrapolating back to the actual temperature of the specimen when accounting for temperature rises due to deformation heating. The plots have been conducted at deformation temperatures of 1400°C, 1350°C, 1300°C and 1250°C.

![Figure 4.28](image)

**Figure 4.28:** Plots of \(\ln(\sigma)\) against \(1/T\) at different strains for \(\text{Fe}_2\text{Al}\). Used to determine the values of isothermal stress at different strains. The isothermal stress is determined by extrapolating back to the actual temperature of the specimen when accounting for temperature rises due to deformation heating. The plots have been conducted at deformation temperatures of 1400°C, 1350°C and 1300°C. The data at the deformation temperature of 1250°C has been omitted deliberately so as to improve the straight line fit of the curves.
Figure 4.29: Adiabatic (without correction for deformation heating effects) and isothermal (with correction for deformation heating effects) flow curves showing the effect of the deformation temperature on the isothermal flow behaviour of Pt$_{86}$Al$_{8}$Ru$_{6}$. The notation bar II refers to the flow stress that has been adjusted by the barrelled model.

4.6 Zener-Hollomon parameter

The microstructure of the hot worked material is governed dynamically by the deformation conditions. Microstructural evolution is driven thermodynamically by the effective strain and kinetically by the deformation temperature and strain rate during the hot deformation process. The Zener-Hollomon parameter ($Z$) is often used to relate the observed hot worked behaviour of the material in terms of the strain rate and deformation temperature with the flow stress experienced during deformation. The value of the $Z$ in context of the testing technique currently employed is invaluable. The overshoot in strain that occurs at $0.5/s$ and $1.2/s$ means that a direct comparison between the observed microstructures obtained at these strain rates cannot be conducted simply on the basis of strain rate and temperature. Instead, the difference in the effective strain and the influence of the effective strain on the $Z$ and hence on the flow stress also has to be taken into account.

When the material being investigated is reasonably well characterised and the activation energy is known at the specific deformation conditions being employed then $Z$ is usually determined directly from equation 2.28 by means of inserting the appropriate values of deformation temperature ($T$), strain rate ($\dot{e}$) and activation energy ($Q$). However, when the material is less well characterised as is Pt$_{86}$Al$_{8}$Ru$_{6}$, then it is necessary to determine the activation energy for each set of deformation conditions investigated. According to Zhang et al., $Q$ is dependent on the parameters $m$ and $K$ (see equation 2.29 and equation 2.30 respectively), which are solved directly from the slopes of the plots of ln($\sigma$) versus ln($\dot{e}$) (see figure 4.30 and figure 4.31) and ln($\sigma$) versus $1/T$ (see figure 4.27 and figure 4.28) respectively.
Figure 4.30: Plot of \( \ln(\sigma) \) against \( \ln(\varepsilon) \) at different strains for Pt\(_{100}\)Al\(_{10}\)Ru\(_{4}\) used to determine the strain rate sensitivity \( m \) at different strains. \( m \) is determined directly from the slopes of the respective curves. The plots have been conducted at true strains above and below 0.1.

Figure 4.31: Plot of \( \ln(\sigma) \) against \( \ln(\varepsilon) \) at different strains for Pt\(_{100}\)Al\(_{10}\)Ru\(_{4}\) used to determine the strain rate sensitivity \( m \) at different strains. \( m \) is determined directly from the slopes of the respective curves. The plots have been conducted at true strains only above 0.1. The data at true strains below 0.1 has not been included in the plots so to improve the straight line fit of the curves.
In order for $Q$ and hence $Z$ to be solved for a specific deformation test, it is necessary to determine $m$ and $K$ at the end of deformation, that is, at the highest level of strain experienced during a particular test (see table 4.3). However, overshoot at 1.2/s results in a nominal strain input of 0.69, which is a maximum level of strain and is not attained in any other test. This means that the \( \ln(\sigma) \) versus \( \ln(\dot{\varepsilon}) \) plot cannot be conducted at a strain of 0.69 as there is only one data point available (i.e. at 1.2/s). Similarly, overshoot at 0.5/s leads to a nominal strain input of 0.59, which is attained in only one other test (i.e. at 1.2/s). This implies that the \( \ln(\sigma) \) versus \( \ln(\dot{\varepsilon}) \) plot conducted at 0.59 strain would probably lead to an inaccurate determination of $m$ and thus $Q$ and $Z$ as there are only two sets of data points available at this level of strain. The benefit of solving $Z$ by the method employed by Zhang et al.\(^6\) is that $Z$ can be determined at various levels of strain such that a constitutive equation can be derived to describe the variation of $Z$ with strain at a particular deformation temperature and strain. This means that $Z$ at the end of the deformation cycles at 0.5/s and 1.2/s can be solved from the respective constitutive equations by extrapolating to strains of 0.59 and 0.69 respectively. Alternatively, a trendline that takes on the form of a straight-line function describes the variation of $Q$ with strain (see figure 4.33) and can be used to extrapolate values of $Q$ at strains of 0.59 and 0.69 for the tests conducted at strain rates of 0.5/s and 1.2/s respectively. It is imperative that the constitutive equations be used to describe all values of $Z$ in order for the extrapolated values to be comparable to the values determined directly from the \( \ln(\sigma) \) versus \( \ln(\dot{\varepsilon}) \) and \( \ln(\sigma) \) versus $1/T$ plots. The plots of $m$ and $K$ against true strain that are used in order to solve $Q$ against true strain (see figure 4.33) and hence $Z$ against true strain (see figure 4.34) are shown in figure 4.32. The $Z$ values of interest, that is, at the end of deformation, have been summarised in table 4.3. Note that the constitutive equations take the form of a third order polynomial function and in most cases cannot be used to accurately interpolate values of $Z$ below true strains of 0.1.

![Figure 4.32: Plots of $m$ against true strain (a) and $K$ against true strain (b) for Pr$_{90}$Al$_{10}$Ru$_{1}$ used to solve the variation of $Q$ with true strain.](image)
Figure 4.33: Plot of $Q$ against true strain for $\text{Pb}_3\text{Al}_{10}\text{Ru}_4$ used to solve the variation of $Z$ with true strain.
Figure 4.34: Plots of $Z$ against true strain for Pt$_{50}$Al$_{50}$Ru$_4$ at different deformation conditions.

Table 4.3: Summary of $Z$ values for Pt$_{50}$Al$_{50}$Ru$_4$ with considering the variation of the Zener-Hollomon parameter ($Z$) with strain.
4.7 Kocks-Mecking plots

The Kocks-Mecking plot shows the work hardening rate plotted against the corresponding flow stress values. Examples of plots of this nature are given by figure 4.35 to figure 4.37 for frictionless flow and by figure 4.38 to figure 4.39 for frictional flow. The relative critical stresses for the initiation of dynamic recrystallisation ($\sigma_c$) are given by the inflection points in the curves, which are marked by the arrows. The critical strains for the initiation of dynamic recrystallisation ($\varepsilon_c$) are obtained from the corresponding critical stresses in the flow curves (see figure 4.31 to figure 4.40), which are also marked by arrows. The relevant results have been summarised in table 4.4. No value of $\varepsilon_c$ could be determined for Pt$_{60}$Al$_{10}$Cr$_4$ at 1400°C (at 0.1/s) due to too large a scatter in the flow stress data and a value of $\varepsilon_c$ for Pt$_{60}$Al$_{10}$Ru$_4$ at 1250°C (at 0.1/s) could not be determined.

<table>
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<th>alloy</th>
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<th>$\varepsilon_c$ (frictionless)</th>
<th>$\sigma_c$ (frictional)</th>
<th>$\varepsilon_c$ (frictional)</th>
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<td>107</td>
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Table 4.4: Summary of the critical stresses ($\sigma_c$) and critical strains ($\varepsilon_c$) obtained from the Kocks-Mecking plots for the initiation of dynamic recrystallisation for Pt$_{60}$Al$_{10}$Ru$_4$ and Pt$_{60}$Al$_{10}$Cr$_4$ at different deformation conditions. Results for both frictional and frictionless conditions are presented.
Figure 4.35: Kocks-Mecking plot for Pt$_{60}$Al$_{40}$ deformed at 1400°C and 1.2/s for frictionless flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 178MPa.

Figure 4.36: Kocks-Mecking plot for Pt$_{60}$Al$_{40}$ deformed at 1400°C and 1.2/s for frictional flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 169MPa.
Figure 4.37: Kocks-Mecking plot for Pd3Al2Ru deformed at 1400°C and 0.5/s for frictionless flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 150 MPa.

Figure 4.38: Kocks-Mecking plot for Pd3Al2Ru deformed at 1400°C and 0.5/s for frictional flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 125 MPa.
Figure 4.39: Kocks-Mecking plot for Pt₀₆Al₁₉Ru₄ deformed at 1400°C and 0.1 s⁻¹ for frictionless flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 87.7 MPa.

Figure 4.40: Kocks-Mecking plot for Pt₀₆Al₁₉Ru₄ deformed at 1400°C and 0.1 s⁻¹ for frictional flow. The critical stress for dynamic recrystallisation is given by the inflection point that is marked by the arrow at 78.3 MPa.
Figure 4.41: Flow curves for frictionless conditions for $Pr_{60}Al_{13}Ru_{4}$ deformed at varying strain rates showing the critical strains for dynamic recrystallisation that are marked by the arrows.

Figure 4.42: Flow curves for frictional conditions for $Pr_{60}Al_{13}Ru_{4}$ deformed at varying strain rates showing the critical strains for dynamic recrystallisation that are marked by the arrows.
Figure 4.43: Flow curves for frictionless conditions for Pt66Al44Ru, deformed at varying temperatures showing the critical strains for dynamic recrystallisation that are marked by the arrows.

Figure 4.44: Flow curves for frictional conditions for Pt66Al44Ru, deformed at varying temperatures showing the critical strains for dynamic recrystallisation that are marked by the arrows.
Figure 4.45: Flow curves for frictionless conditions for each Pt_{80}Al_{10}Cr_{10} specimen compressed to significant strains (e > 0.3) deformed at varying deformation conditions showing the critical strains for dynamic recrystallisation that are marked by the arrows.

Figure 4.46: Flow curves for frictional conditions each Pt_{80}Al_{10}Cr_{10} specimen compressed to significant strains (e > 0.3) deformed at varying deformation conditions showing the critical strains for dynamic recrystallisation that are marked by the arrows.
4.8 Summary of flow data analysis

1. The calculation of the strain rate as a mean nominal strain rate according to the method described is beneficial in that the strain rate at the high temperature proof strength and at the end of the deformation cycle can be differentiated during non-constant strain rate compression testing.

2. The continuous increase in the measured load with strain and the inhomogeneous distribution in the grain morphology across the vertical section of the barrelled specimen suggest that friction at the loading face does influence the flow behaviour of the material when compressed to significant strains.

3. The FEM shows that friction causes the compression specimen to barrel during deformation such that the localised strain at the centre of the specimen is significantly greater than the nominal strain input.

4. The FEM predicts the outer diameter of the compression specimen with good accuracy and the contact diameter with less accuracy.

5. The FEM suggests that it is the friction condition and hence the surface finish of the compression specimen that determines the extent of barrelling and hence the extent of strain and microstructural inhomogeneities within the material during compression testing using tantalum lubrication.

6. The von Mises yield criterion and the Siebel corrective function do not predict the initial and final outer diameters, whilst the barrelling model gives good correlation between the actual and predicted outer diameters of the compression specimen.

7. Insufficient information with regards to the material density at the temperatures of deformation and the effects of strain on the adiabatic correction factor leads to inaccuracy during the extrapolation of isothermal stress values and consequently, no useful deductions may be inferred about the affects of deformation heating of the true flow behaviour of Pt55Al15Ru4.

8. The Zener-Holloman parameter has been determined for all the deformation conditions investigated for Pt55Al15Ru4 as a function of strain according to the method described by Zhang et al.

9. The Kocks-Mecking plots show that the critical strain (for metal flow adjusted for frictional effects) for Pt55Al15Ru4 increases with increasing strain rate and decreasing deformation temperature. The flow data used in the plots have been filtered to reduce the scatter associated with the work hardening rates obtained during non-constant strain rate compression testing and therefore the critical strain strain for dynamic recrystallisation should be viewed with caution. Nonetheless, it is possible to conclude that the presence of inflection points in the Kocks-Mecking plots at least suggest the occurrence of dynamic recrystallisation.
CHAPTER 5

RESULTS II

EXPERIMENTAL RESULTS

5.1 Starting state

The micrographs in figure 5.1(a), figure 5.2(a) and figure 5.3(a) illustrate the as-cast, inhomogeneous microstructures of Pt₆₅Al₆₆Ru₄, Pt₆₅Al₆₆Cr₄ and Pt₆₅Al₆₆Cr₄Ru₄ respectively. Figure 5.1(b), figure 5.2(b) and figure 5.3(b), which represent regions within the micrographs in figure 5.1(a), figure 5.2(a) and figure 5.3(a) respectively at higher magnification, show that the alloys comprise of two phases in the as-cast state, namely phase A and phase B. The elemental composition of the two phases were identified by EDS analysis, which showed that for Pt₆₅Al₆₆Ru₄ phase A is enriched in aluminium and depleted in ruthenium, whilst phase B is rich in ruthenium and deficient in aluminium; for Pt₆₅Al₆₆Cr₄ phase A is rich in aluminium and depleted in chromium, whilst phase B is rich in chromium and depleted in aluminium; and for Pt₆₅Al₆₆Cr₄Ru₄ phase A is enriched with aluminium and chromium and deficient in ruthenium, whilst phase B is enriched with ruthenium and deficient in aluminium and chromium.

![Micrographs showing the cast microstructure of Pt₆₅Al₆₆Ru₄ before the homogenization treatment at 1300°C for 96 hours.](image)

Figure 5.1: Light micrographs showing the cast microstructure of Pt₆₅Al₆₆Ru₄ before the homogenisation treatment at 1300°C for 96 hours.
After the homogenisation heat treatment at 1300°C for 96 hours, Pt-26Al-18Cr-14Ru is devoid of any dendritic features seen in the cast state (see figure 5.4(a) and figure 5.4(b)). The use of x-ray mapping (see figure 5.7) shows an even distribution in the concentration of the platinum, aluminium and ruthenium contents and thus indicates that Pt-26Al-18Cr-14Ru is single-phased in the homogenised condition. The single phase is believed to be a platinum solid solution which had been frozen in from the homogenisation temperature during quenching.
The micrograph in figure 5.5(a) shows the homogenised microstructure and the shape and size of the grains of Pt_{66}Al_{14}Cr_{1} after the homogenisation treatment. The microstructure has none of the dendritic structures evident in the cast state. Closer inspection at higher magnification (see figure 5.5(b)) shows that the homogenised structure is two-phased, comprising of a fine dispersion of small block-like precipitate structures (phase B) in a matrix phase (phase A). The elemental concentration of phases A and B were analysed using x-ray mapping (see figure 5.8), which showed that the precipitate structures (phase B) are rich in aluminium and depleted in chromium, whereas the matrix (phase A) is rich in chromium and depleted in aluminium. Consequently, phase A was provisionally identified as γ and phase B as γ′.

The homogenised microstructure of Pt_{66}Al_{14}Cr_{1}Ru_{3} after homogenisation at 1300°C for 96 hours is shown in the micrographs in figure 5.6(a) and figure 5.6(b). The two-phased microstructure is retained after the heat treatment and is characterised by a net-like second phase structure. X-ray mapping was employed in order to analyse the distribution in the elemental concentrations of phases A and B (see figure 5.9), which showed that the precipitate structures (phase B) are rich in aluminium and depleted in chromium, whereas the matrix (phase A) is enriched with ruthenium and chromium and deficient in aluminium. Consequently phase A was provisionally identified as γ and phase B as γ′.

**Figure 5.4:** Light micrographs showing the homogenised microstructure of Pt_{66}Al_{14}Cr_{1} after the heat treatment at 1300°C for 96 hours.
Results II: Experimental Results

Figure 5.5: Light micrographs showing the homogenised microstructure of Pt$_{70}$Al$_{30}$Cr$_5$ after the heat treatment at 1300°C for 96 hours.

Figure 5.6: Light micrographs showing the homogenised microstructure of Pt$_{70}$Al$_{30}$Cr$_5$ after the heat treatment at 1300°C for 96 hours.
Figure 5.7: X-ray maps of Pt$_{10}$Al$_{90}$Ru$_{4}$ in the homogenised state, showing the distribution in the concentration of elements across the mapped region. The x-ray maps are in back-scattered electron detection (BSED) mode, whilst the actual image is in the secondary electron detection (SED) mode. All images are of the same magnitude.

(a) SEM-SED image of the mapped region  
(b) concentration distribution of platinum  
(c) concentration distribution of aluminium  
(d) concentration distribution of ruthenium

Figure 5.8: X-ray maps of Pt$_{10}$Al$_{90}$Cr$_4$ in the homogenised state, showing the distribution in the concentration of elements across the mapped region. Blue indicates an area enriched in concentration of a particular element, whilst green indicates an area depleted in concentration of that element. The x-ray maps are in back-scattered electron detection (BSED) mode, whilst the actual image is in the secondary electron detection (SED) mode. All images are of the same magnitude.

(a) SEM-SED image of the mapped region  
(b) concentration distribution of platinum  
(c) concentration distribution of aluminium  
(d) concentration distribution of chromium
Figure 5.9: X-ray maps of Pt₉₅Al₁₅Cr₃Ru₃ in the homogenized state, showing the distribution in the concentration of elements across the mapped region. Blue indicates an area enriched in concentration of a particular element, whilst green indicates an area depleted in concentration of that element. The x-ray maps are in back-scattered electron detection (BSED) mode, whilst the actual image is in the secondary electron detection (SED) mode. All images are of the same magnitude.

(a) SEM-SED image
(b) concentration distribution of platinum
(c) concentration distribution of ruthenium
(d) concentration distribution of aluminium
(e) concentration distribution of chromium
5.2 Stress-strain and load-displacement data

The load-displacement data gathered during the series of compression tests were converted to true stress-true strain data and plotted in figure 5.10 and figure 5.11 for Pt86Al10Ru, and in figure 5.12 and figure 5.13 for Pt46Al10Cr4. Since Pt86Al10Cr4Ru4 could not be deformed to significant strains without cracking, it was not found necessary to present the true stress-true strain data on this alloy as the true stress-true strain curves were not needed for an analysis. Therefore only the load-displacement curves for Pt86Al10Cr4Ru4 are presented (see figure 5.14 and figure 5.15).

The true/flow stress data obtained for the compression testing of Pt86Al10Ru, were adjusted for surface friction at the loading face according to the barrelling model. These flow curves, corrected for friction, are shown in figure 5.10 for Pt86Al10Ru deformed at varying strain rates and in figure 5.11 for Pt86Al10Ru deformed at varying deformation temperatures. With the exception of Pt86Al10Ru deformed at 1250°C at 0.1/s, the flow stress increases until a peak is reached, after which it gradually decreases until the end of the deformation cycle. For Pt86Al10Ru deformed at 1400°C at 0.5/s, the flow stress increases until a peak is attained, then gradually decreases until a steady state in the flow stress is reached. This steady state represents a transient state during which the flow stress remains invariant with increasing true strain. For Pt86Al10Ru compressed at 1250°C at 0.1/s, the flow stress increases and then reaches a pseudo-peak where the flow stress shows a slight inflection point, at which point the flow stress decreases slightly followed by a gradual increase until the end of the deformation cycle.

The true/flow stress obtained for Pt86Al10Cr4 compressed at 1400°C at both 0.1/s and 1.1/s and at 1300°C at 0.1/s were adjusted for surface friction according to the barrelling model. These friction-corrected flow curves are shown in figure 5.13 for Pt86Al10Cr4. Note that under these deformation conditions, the flow stresses increase until peaks are reached, after which they gradually decrease until the end of the deformation event. The peak stress is particularly pronounced for Pt86Al10Cr4 compressed at 1400°C at 1.1/s. At 1100°C (at 0.1/s), the flow stress continues to increase throughout the compression cycle, displaying two inflection points (see figure 5.12). At 1200°C (at 0.1/s) and at 1300°C (at 1.1/s), the flow stresses increase throughout the relatively short deformation cycles (see figure 5.12).

The load-displacement curves for Pt86Al10Cr4Ru4 (see figure 5.14 and figure 5.15) show that this alloy could not be deformed to significant strains without cracking. The sudden fluctuations observed in the load data indicate the occurrence of cracking within the specimen. Note that the displacement never exceeds 900 microns as the desired displacement had to limited during testing in case the load was to reach values much greater than 5000N (that is, the upper limit of the load cell), thereby possibly inflicting damage to the load cell.
Figure 5.10: Flow stress against true strain at 1400°C and varying strain rates for Pt$_{80}$Al$_{10}$Ru$_{10}$.

Figure 5.11: Flow stress against true strain at 0.1/s and varying deformation temperatures for Pt$_{80}$Al$_{10}$Ru$_{10}$.
Figure 5.12: True stress against true strain at 0.1/s and varying deformation temperatures for $\text{Pt}_{80}\text{Al}_{10}\text{Cr}_2$.

Figure 5.13: True stress against true strain at 1.1/s and 0.1/s at 1400°C and 1300°C for $\text{Pt}_{80}\text{Al}_{10}\text{Cr}_2$. 
Figure 5.14: Load against displacement at varying deformation temperatures for Pt$_{80}$Al$_{14}$Cr$_6$Ru$_2$.

Figure 5.15: Load against displacement at varying strain rates at 1300°C and 1200°C for Pt$_{80}$Al$_{14}$Cr$_6$Ru$_2$. 
5.3 The deformed state

5.3.1 Pt$_{80}$Al$_{10}$Ru$_4$

Light micrographs were taken of Pt$_{80}$Al$_{10}$Ru$_4$ after hot compression testing to show the deformed microstructures. The micrographs shown in figure 5.16 were taken at the centre of the compressed specimens, which correspond to the effective localised strain values that were determined by the FEM analysis. All the micrographs at lower magnification (see figure 5.16), with the exception of 1400°C at 0.5/s and 1.2/s, show that duplex microstructures are obtained for Pt$_{80}$Al$_{10}$Ru$_4$. These duplex microstructures comprise of large, deformed grains with highly irregular grain boundaries elongated in the direction approximately normal to the principal compression axis. The larger grains are less well defined at 1400°C at 0.5/s and 1.2/s. All the micrographs at higher magnification (see figure 5.17) have been taken locally in the vicinity of the grain boundaries of the larger grains shown in the highlighted regions in figure 5.16 and show the smaller recrystallised grains at higher magnification.
Figure 5.16: Light micrographs showing deformed microstructures of Pt$_{60}$Al$_{10}$Ru$_4$ under different deformation conditions. The micrographs were taken at the centre of the compressed specimens which correspond to the effective localised strain values that were determined by the MEM analysis.
Figure 5.17: Light micrographs showing the deformed microstructures of Pd$_2$Al$_{10}$Ru$_4$ under different deformation conditions. The micrographs were taken within the regions highlighted in figure 5.16 at higher magnification.
Table 5.1 is a tabulation of the Zener-Hollomon parameters (Z) for Pt₆₆Al₁₆Ru₄ and the corresponding rankings in the sizes of the recrystallised grains (RX-GS) and microhardness measurements. It is important to realise that the task of determining the size of recrystallised grains by means of physical measurement is daunting, due to the elongated and irregular geometry of these grains. Instead, it is easier to simply identify or rank the comparative grain sizes with respect to the deformation conditions by visual inspection. This method suffices for a qualitative study of this nature. The tabulation in table 5.1 indicates that the RX-GS increases with decreasing values of Z. Conversely, the microhardness number decreases with decreasing values of Z. Therefore, the size of the recrystallised grains decreases with increasing deformation temperature and decreasing strain rate (indicated by increasing values of Zener-Hollomon parameter), which gives rise to larger values in the measured microhardness.

<table>
<thead>
<tr>
<th>test</th>
<th>Z (s)</th>
<th>RX-GS ranking</th>
<th>microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1406°C, 1.2/s</td>
<td>1.6×10¹⁰</td>
<td>1</td>
<td>612±25</td>
</tr>
<tr>
<td>1400°C, 0.3/s</td>
<td>3.7×10⁸</td>
<td>2</td>
<td>575±31</td>
</tr>
<tr>
<td>1250°C, 0.1/s</td>
<td>2.6×10⁷</td>
<td>3</td>
<td>566±38</td>
</tr>
<tr>
<td>1300°C, 0.1/s</td>
<td>1.7×10⁶</td>
<td>4</td>
<td>533±45</td>
</tr>
<tr>
<td>1350°C, 0.1/s</td>
<td>8.2×10⁵</td>
<td>5</td>
<td>515±43</td>
</tr>
<tr>
<td>1400°C, 0.1/s</td>
<td>4.1×10⁴</td>
<td>6</td>
<td>524±57</td>
</tr>
<tr>
<td>1400°C, 0.06/s</td>
<td>2.5×10³</td>
<td>7</td>
<td>496±49</td>
</tr>
</tbody>
</table>

Table 5.1: Tabulation of the recrystallised grain size (RX-GS) ranking from the smallest RX-GS to largest RX-GS under different deformation conditions for Pt₆₆Al₁₆Ru₄. The RX-GS indices indicate the ranking of the RX-GS from finest to coarsest in ascending order. The corresponding microhardness values, which have been taken within the region of metallographic interest, are also listed for the different deformation conditions.

5.3.2 Pt₆₆Al₁₆Cr₄

Light micrographs were taken of Pt₆₆Al₁₆Cr₄ after compression testing to show the deformed microstructures. The micrographs shown in figure 5.18(a), figure 5.18(b) and figure 5.18(c) were taken at the centre of the compressed specimens that were deformed at 1400°C (at 0.1/s), 1400°C (at 1.1/s) and 1300°C (at 0.1/s) respectively and correspond to the effective localised strain values that were determined by the FEM analysis. Under these deformation conditions, the microstructures obtained comprise of grains having irregular shaped grain boundaries. Figure 5.18(g), which is a micrograph at slightly higher
magnification of the microstructure shown in Figure 5.18(c), shows a large deformed grain, elongated in the direction normal to the applied compressive load, with highly irregular grain boundaries (see region A) and smaller grains also with irregular, wave-like grain boundaries (see region B).
Results II: Experimental Results

The micrographs shown in figure 5.18(d), figure 5.18(e) and figure 5.18(f) were taken as close as possible to the centre of the buckled compression specimen. The microstructure obtained for Pt₆₆Al₄₄Cr₄ after deformation at 1300°C at 1.1/s (see figure 5.18(d)) shows relatively large deformed grains, elongated in the direction roughly normal to the primary compression axis. In contrast, the microstructure obtained after deformation at 1100°C at 0.1/s (see figure 5.18(f)) shows relatively large undeformed grains. A duplex microstructure is obtained for Pt₆₆Al₄₄Cr₄ after deformation at 1200°C at 0.1/s (see figure 5.18(e)), comprising of large grains, some of which contain a second phase (see region A) and some of which are devoid from this second phase (see region B). The second phase is shown in figure 5.18(h) at slightly higher magnification. X-ray mapping (see figure 5.19) was performed within this magnified region in order to investigate the elemental distribution of the two-phase microstructure obtained at 1200°C at 0.1/s. The analysis revealed that the precipitate phase is rich in aluminium and slightly depleted in chromium (see figure 5.19(a)), whilst the matrix phase is slightly enriched in chromium and depleted in aluminium (see figure 5.19(c)). The distribution in the concentration of platinum is uniform across both phases (see figure 5.19(d)). Consequently, the precipitate phase was provisionally identified as γ' and the matrix as γ. Similarly, x-ray mapping was also employed to analyse the apparently single-phase nature of region B (see figure 5.18(c)). The x-ray maps showed a homogeneous distribution in the concentrations of platinum, aluminium and chromium within region B in figure 5.18(e). Note that other than at 1200°C, Pt₆₆Al₄₄Cr₄ remained single-phased after compression testing. The single-phase nature was confirmed by x-ray mapping.
Results II: Experimental Results

Figure 5.19: X-ray maps of Pt$_{80}$Al$_{10}$Cr$_4$ after deformation at 1200°C at 0.1/s, showing the distribution in the concentration of elements across the mapped region. Blue indicates an area enriched in concentration of a particular element, whilst green indicates an area depleted in concentration of that element. The x-ray maps are in back-scattered electron detection (BSED) mode, whilst the actual image is in the secondary electron detection (SED) mode. All images are of the same magnitude.

(a) SEM-SED image of the mapped region  (b) concentration distribution of platinum
(c) concentration distribution of aluminium (d) concentration distribution of chromium

5.3.3 Pt$_{80}$Al$_{10}$Cr$_4$Ru$_2$

The micrographs in figure 5.20 show the deformed microstructures of Pt$_{80}$Al$_{10}$Cr$_4$Ru$_2$ under different deformation conditions. All specimens buckled during compression and therefore the micrographs were taken as close to the centre of the buckled specimens as possible. The first feature of note is the presence of a second phase after deformation. X-ray mapping (see figure 5.21) was employed in order to determine whether the second phase was $\gamma'$. The analysis revealed that the second phase is rich in aluminium and slightly depleted in ruthenium (see figure 5.21(c)), whilst the matrix phase is slightly enriched in ruthenium and depleted in aluminium (see figure 5.21(d)). The distribution in the concentration of chromium (see figure 5.21(e)) and platinum (see figure 5.21(b)) is uniform across both phases. Consequently, second phase was provisionally identified as $\gamma'$ and the matrix as $\gamma$. The presence of $\gamma'$ is observed under all the deformation conditions explored, even at temperatures as high as 1400°C. The $\gamma'$ precipitates were initially expected to coarsen significantly and perhaps even dissolve at deformation temperatures exceeding the
homogenisation temperature but this is not observed (see figure 5.20(a)). After deformation at 1400°C, the material is intact at the centre of the specimen (see figure 5.20) but cracked towards the surface and edge regions (see figure 5.20). The cracking could be due to the high hoop stresses that accumulate in this region of the specimen due to severe metal shear.
Figure 5.20: Light micrographs showing the deformed microstructures of Pt10Al14Cr3Ru5 at different deformation conditions.
Figure 5.21: X-ray maps of Pt₈₀Al₄₀Cr₄Ru₂ after deformation at 1400°C, showing the distribution in the concentration of elements across the mapped region. Blue indicates an area enriched in concentration of a particular element, whilst green indicates an area depleted in concentration of that element. The x-ray maps are in back-scattered electron detection (BSED) mode, whilst the actual image is in the secondary electron detection (SED) mode. All images are of the same magnitude.

(a) SEM-SED image  
(b) concentration distribution of platinum  
(c) concentration distribution of ruthenium  
(d) concentration distribution of aluminium

After deformation at 1300°C (at 10²/s) (see figure 5.20(c)), the γ' precipitates appear (comparatively) undeformed, which is probably due to the small amount of input strain (refer to figure 5.15 for the small input displacement which is indicative of the small input strain). Cracking is observed mainly between the precipitates in γ, which is unexpected due to the more ductile nature of the matrix. It could be that the ultimate tensile strength of the matrix is significantly lower than the yield strength of the precipitate phase, which means that the matrix cracks as it takes up the load beyond the ultimate tensile strength whilst deformation in the precipitates is still in the elastic regime. Closer examination at much higher magnification is needed for a more confident explanation. Some cracks also appear to be assisted by
porous stress raisers within the material. Cracking is less severe after deformation at 1300°C (at 10^-7/s) (see figure 5.20(d)) than at 1200°C (at 10^-3/s) (see figure 5.20(g)) and could be attributed to the higher deformation temperature which allows for some recovery of strain. Similarly, cracking is less severe after deformation at 1200°C (at 10^-1/s) (see figure 5.20(c)) than at 1200°C (at 10^-2/s) (see figure 5.20(f)) and is probably as a result of the lower level of induced strain at the higher strain rate (refer to figure 5.15 for an indication of the respective strains at 1200°C (at 10^-1/s) and at 1200°C (at 10^-2/s), which are given by the respective input displacements). The material is unaltered after deformation at 1100°C (at 10^-3/s) (see figure 5.20(h)) due to the negligible amount of plastic strain input (see figure 5.14).

5.4 High temperature proof strength (σ_{htps})

High temperature proof strength values (σ_{htps}) were obtained for each compression test performed. The values of σ_{htps} are estimated at the appropriate level of true stress where the increase in true stress with increasing true strain deviates from linearity (see figure 5.22). This transition point, which indicates the change in the mode of deformation from elastic to plastic, is given by σ_{htps} as shown in figure 5.22. The values of σ_{htps} that are obtained by this method are given in table 5.2. Note that the strain rates corresponding to the values of σ_{htps}, which are quoted in table 5.2, are the mean strain rates (ε) that have been determined according to the procedure outlined in section 4.2. This has been done so that the values of σ_{htps} can be related to an appropriate value of strain rate that corresponds most closely to the actual strain rate experienced at a particular value of σ_{htps} during non-constant strain rate compression testing.

![Plot of true stress against true strain for Pm_3Al_{13}Cr_12 at a deformation temperature of 1400°C and a mean strain rate of 10^-7/s. The green line represents an extrapolation of the region where the increase in true stress with true strain is linear. The point at which the increase in true stress with true strain deviates from linearity is marked by the high temperature proof strength, denoted by the symbol σ_{htps}. In this case, σ_{htps} is determined as 28 MPa.](Image)
<table>
<thead>
<tr>
<th>Test</th>
<th>T (°C)</th>
<th>$\dot{e}$ (s$^{-1}$)</th>
<th>$\sigma_{npr}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. $Pt_{80}Al_{10}Ru_{10}$</td>
<td>1400</td>
<td>$10^3$</td>
<td>25</td>
</tr>
<tr>
<td>2. $Pt_{60}Al_{20}Ru_{20}$</td>
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<td>$10^2$</td>
<td>30</td>
</tr>
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<td>3. $Pt_{40}Al_{40}Ru_{20}$</td>
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<td>66</td>
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<td>$10^2$</td>
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<tr>
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<td>$10^2$</td>
<td>226</td>
</tr>
<tr>
<td>17. $Pt_{60}Al_{20}Cr_{20}Ru_{10}$</td>
<td>1100</td>
<td>$10^2$</td>
<td>291</td>
</tr>
<tr>
<td>18. $Pt_{80}Al_{10}Cr_{10}Ru_{10}$</td>
<td>1300</td>
<td>$10^2$</td>
<td>125</td>
</tr>
<tr>
<td>19. $Pt_{60}Al_{20}Cr_{20}Ru_{10}$</td>
<td>1200</td>
<td>$10^2$</td>
<td>103</td>
</tr>
<tr>
<td>20. $Pt_{80}Al_{10}Cr_{10}Ru_{10}$</td>
<td>1200</td>
<td>$10^2$</td>
<td>298</td>
</tr>
</tbody>
</table>

Table 5.2: Tabulation of high temperature proof strength ($\sigma_{npr}$) with corresponding deformation temperatures (T) and mean strain rates ($\dot{e}$).
The change in $\sigma_{\text{hyp}}$ with deformation temperature for Pt$_{86}$Al$_{14}$Ru$_1$, Pt$_{86}$Al$_{10}$Cr$_4$ and Pt$_{86}$Al$_{10}$Cr$_5$Ru$_1$ at a mean strain rate of $10^{-3}$/s is shown in figure 5.23. The change in $\sigma_{\text{hyp}}$ with deformation temperature for Pt$_{86}$Al$_{14}$Cr$_5$Ru$_1$ is linear. Initially, it was expected that this alloy would undergo $\gamma'$ coarsening and/or dissolution during the soaking period prior to the onset of deformation at deformation temperatures at and above 1300°C, which is based on the homogenisation temperature. $\gamma'$ coarsening causes a widening of the $\gamma$ channels between the $\gamma'$ precipitates, which provides less hindrance to dislocation movement and consequently gives rise to lower values of $\sigma_{\text{hyp}}$. However, the deformation microstructures in figure 5.20 show that Pt$_{86}$Al$_{14}$Cr$_5$Ru$_1$ resists $\gamma'$ coarsening and/or dissolution even at deformation temperatures as high as 1400°C. No transition point in $\sigma_{\text{hyp}}$ is observed as the alloy does not undergo any phase transformations at the deformation conditions investigated.

In contrast to Pt$_{86}$Al$_{14}$Cr$_5$Ru$_1$, a transition point in $\sigma_{\text{hyp}}$ is observed for Pt$_{86}$Al$_{10}$Cr$_4$ somewhere between deformation temperatures of 1200°C and 1300°C. This transition is partly attributed to the $\gamma'$ dissolution that occurs at 1300°C and 1400°C, meaning that the $\sigma_{\text{hyp}}$ at these deformation temperatures would be significantly lower than at 1100°C and 1200°C. By “significantly lower” it is implied that $\sigma_{\text{hyp}}$ at 1300°C and 1400°C would be lower than that predicted by a straight line function fitted and extrapolated through $\sigma_{\text{hyp}}$ at 1100°C and 1200°C. In addition, $\sigma_{\text{hyp}}$ at 1200°C is significantly higher than at 1300°C and 1400°C due to the partial reprecipitation of $\gamma'$ (figure 5.18(e)) and the evolution of $\gamma''$ (see figure 5.18(h)) that takes place during the soaking period at 1200°C prior to the onset of deformation during the hot compression test. In theory, $\sigma_{\text{hyp}}$ at 1100°C should be lower than at 1200°C due to the absence of $\gamma'$ at 1100°C. This is clearly not the case and it can be attributed to the extremely sluggish thermal kinetics that exist at deformation temperatures as low as 1100°C, which gives rise to slow dislocation movement, that exceeds the extent of dislocation hindrance due to the partial reprecipitation of $\gamma'$ and the evolution of $\gamma''$ at 1200°C.

Figure 5.24 shows the change in $\sigma_{\text{hyp}}$ with strain rate for Pt$_{86}$Al$_{14}$Ru$_1$, Pt$_{86}$Al$_{10}$Cr$_4$ and Pt$_{86}$Al$_{10}$Cr$_5$Ru$_1$. Note that $\sigma_{\text{hyp}}$ for Pt$_{86}$Al$_{14}$Cr$_5$Ru$_1$ is always greater than for Pt$_{86}$Al$_{10}$Ru$_4$ and Pt$_{86}$Al$_{10}$Cr$_4$ under the deformation conditions investigated. Also, $\sigma_{\text{hyp}}$ for Pt$_{86}$Al$_{14}$Ru$_1$ is always greater than for Pt$_{86}$Al$_{10}$Cr$_4$ at deformation temperatures at and above 1300°C, which means that the ability of a 4 atomic percent addition of ruthenium to strengthen the Pt-Al system by a solid solution strengthening mechanism is greater than that achieved by a similar addition of chromium (note that $\gamma'$ is absent from both Pt$_{86}$Al$_{10}$Ru$_4$ and Pt$_{86}$Al$_{10}$Cr$_4$ at and above 1300°C). Figure 5.25 illustrates the change in $\sigma_{\text{hyp}}$ with strain rate for Pt$_{86}$Al$_{10}$Cr$_5$Ru$_1$ and shows that the values of $\sigma_{\text{hyp}}$ at 1200°C are always greater than at 1300°C, which is expected as dislocation movement is slower at 1200°C than at 1300°C. No transition point in $\sigma_{\text{hyp}}$ is observed as the alloy does not undergo any phase transformations at the deformation conditions investigated.
Figure 5.23: Plots of high temperature proof strength against deformation temperature for Pt<sub>67</sub>Al<sub>13</sub>Cr<sub>4</sub>, Pt<sub>65</sub>Al<sub>18</sub>Ru<sub>4</sub>, and Pt<sub>56</sub>Al<sub>15</sub>Cr<sub>3</sub>Ru<sub>3</sub>.

Figure 5.24: Plots of high temperature proof strength against strain rate for Pt<sub>60</sub>Al<sub>10</sub>Ru<sub>4</sub> at 1400°C and Pt<sub>65</sub>Al<sub>15</sub>Cr<sub>3</sub> at 1400°C and 1300°C.
**Figure 5.25:** Plots of high temperature proof strength against strain rate for $P_{176Al_{44}Cr_{27}Ru_3}$ at 1300°C and 1200°C.
5.5 Post-stress annealing

In an attempt to reprecipitate the γ' phase from Pt80Al10Cr4 deformed at 1400°C (at 0.1/s and 1.1/s) and at 1300°C (at 0.1/s), it was decided to anneal the deformed specimens at 1200°C for a period of 4 hours. The annealed microstructures, which are shown in figure 5.26(a), figure 5.26(b) and figure 5.26(c), suggest that Pt80Al10Cr4 remains single-phased after the heat treatment. Note that the single-phased nature of these specimens were confirmed by x-ray mapping. In contrast, the specimen deformed at 1200°C, which gave rise to a partially two-phase microstructure after deformation (see figure 5.18(a)), showed a wide range of distinct changes in γ' morphology during an annealing treatment sequence at 1200°C at varying annealing times (see figure 5.27), which included γ' coarsening and eventual dissolution. Once again, all phases were analysed by EDS and x-ray mapping methods.

Figure 5.26: Light micrographs showing the grain sizes and shapes after annealing Pt80Al10Cr4 at 1200°C for 4 hours.
Figure 5.27 shows the changes in $\gamma'$ morphology in $\text{Pt}_{60}\text{Al}_{15}\text{Cr}_{25}$, which was deformed at 1200°C, during a post-annealing heat treatment at 1200°C for varying annealing times. After 4 hours (see figure 5.27(a)) $\gamma'$ coherency is lost and the precipitates coarsen significantly in certain regions (regions A) in order to minimise the surface energy whilst the morphology in the rest of the material (region B) remains relatively unaltered. After 8 hours (see figure 5.27(b)) the precipitates undergo substantial coarsening to form a conglomerate-like structure (see region A) and link up in other regions to form tiny string-like structures (see region B) in an attempt to further reduce surface energy, whilst undergoing dissolution in the $\gamma$ matrix at certain regions within the material (see region C). After 12 hours (see figure 5.27(c)) the volume fraction of $\gamma'$ is drastically reduced (see region A) by substantial precipitate dissolution and the material largely comprises of $\gamma''$ (see region B). After 16 hours (see figure 5.27(d)) most of the material is devoid of $\gamma'$ (see region A) whilst some $\gamma'$ join more extensively in other parts of the material to form larger string-like structures (see region B). After 20 hours (see figure 5.27(e)) the material is completely devoid of $\gamma'$ and comprises only of $\gamma''$ embedded in the $\gamma$ matrix. After 24 hours (see figure 5.27(f)) the $\gamma''$ dissolves completely and the material comprises only of $\gamma$ solid solution. X-ray mapping confirms that the microstructure obtained after annealing at 1200°C for 24 hours is completely single-phased and EDS analysis reveals that this single-phased structure, which is presumably platinum-enriched solid solution, is rich in platinum, aluminium and chromium.
Figure 5.27: Light micrographs showing the changes in the γ' morphology in Pt₆₆Al₂₆Cr₈ which was deformed at 1200°C at 0.1 s⁻¹ after annealing at 1200°C at different annealing times.
CHAPTER 6

DISCUSSION

6.1 Zener-Hollomon parameter

The Zener-Hollomon parameter has been determined for all the deformation conditions investigated for Pd$_{66}$Al$_{13}$Ru$_4$ as a function of true strain (see section 4.6) according to the method described by Zhang et al.$^{58}$ During the analysis it is assumed that the activation energy of Pd$_{66}$Al$_{13}$Ru$_4$ does not vary with the temperature of deformation. Although this assumption is consistent with the work of Robertson and McShane$^{71}$ on Ti-10V-2Fe-3Al (a BCC material) and that of Zhang et al.$^{58}$ on an aluminium alloy 6061/6Si Cp MMCs (an FCC material), it may not necessarily be true for Pd$_{66}$Al$_{13}$Ru$_4$. Humphreys and Hatherly$^{79}$ have suggested that the activation energies of some metallic materials increase with increasing deformation temperature. Unfortunately, the assumption that the activation energy of Pd$_{66}$Al$_{13}$Ru$_4$ does not change with temperature can only be validated by conducting additional compression tests at the mean nominal strain rates of 1.2/s, 0.5/s and 0.06/s at 1350°C and 1300°C, followed by determining the activation energies at these deformation temperatures according to the method described by Zhang et al.$^{58}$. Further compression testing of such a nature would provide additional flow data that could be used to determine the activation energies of Pd$_{66}$Al$_{13}$Ru$_4$ at 1350°C and 1300°C, which could then be used for comparison. The strain rate that is selected in the calculation of the Zener-Hollomon parameter during the current analysis is the mean nominal strain rate and not the instantaneous strain rate at the strain of interest. Although this is different from the works of Zhang et al.$^{58}$, where constant strain rates were used, it is nonetheless a more rigorous approach than those employed by Robertson and McShane$^{71}$ and Sheehy$^{80}$. Robertson and McShane$^{71}$ and Sheehy$^{80}$ simply used the initial and final strain rates in their respective calculations of the Zener-Hollomon parameter during non-constant strain rate uniaxial compression testing and in so doing, neglected any effect of the varying rate of strain accumulation experienced during the rest of the compression cycle. The correlation of the Zener-Hollomon parameter with the deformed microstructures of Pd$_{66}$Al$_{13}$Ru$_4$ is dealt with in detail in section 6.5.2.
6.2 Friction

Inspection of the load-displacement curves for Pt86Al10Ru4 (see figure 4.4 and figure 4.5) and Pt86Al10Cr4 (see figure 4.6) reveals that the load measured during compression testing continues to increase with increasing displacement, which suggests that the material work hardens throughout the course of plastic deformation. However, under the deformation conditions that correspond to the load-displacement curves in mention, it is observed that the specimens undergo significant height reductions without any evidence of cracking. The ability of the material to strain substantially in this manner and the evidence of metal softening in the deformed microstructures despite the apparent work hardening indicated by the load-displacement curves warrant further investigation into the possible effects of friction on the load-displacement data and the overall flow behaviour of the material. Friction at the loading face presents an additional force that has to be overcome to continually inflict deformation during compression, which is reflected in the load-displacement curves. In addition, the sliding component of friction causes the contact diameter of the specimen to increase as deformation proceeds. This implies that the surface area of the specimen that is contact with the deformation platens increases, which in turn causes the sticking component of friction to increase continuously during the course of deformation. This is manifested in a continuous increase in load that is measured with increasing displacement and the apparent work hardening response of the material as suggested by the load behaviour is an artefact of surface friction. Note that any significant temperature variations along the height of the compression specimen would cause the specimen to deform inhomogeneously. If the temperatures at the two end faces were to be equal and significantly lower than the temperature along the rest of the height, then barrelling could be due to a combination of such an inhomogeneous temperature distribution and surface friction. However, it is unlikely that temperature variations along the height of the compression specimen are responsible for the barrelling observed in the current study, as the skin effect of the induction heating process ensures that the temperature is uniform throughout the specimen along its height.

6.2.1 Use of a Finite Element Model (FEM) to solve the coefficient of friction

A Finite Element Model (FEM) is used to solve the coefficient of friction so as to provide the best possible correlation between the experimentally measured outer barreled diameter of the deformed specimen (\(d_{oa}\)) and the numerically simulated outer barreled diameter of the deformed structure (\(d_{on}\)). This method for solving the coefficient of friction by means of comparing the simulated and actual outer specimen diameters follows the procedure employed by Thiébaut et al.\(^9\).

In reality, unavoidable sliding friction at the loading face causes the actual contact diameter of the specimen to spread along the platen surface during uniaxial compression. Table 4.2 shows that the final simulated contact diameter (\(d_{on}\)) is always greater than the initial contact diameter, meaning that the FEM
correctly predicts an increase in the simulated contact diameter with increasing height reduction. This result is expected because at a given increment in specimen height reduction, the presence of a particular Coulombic coefficient of friction causes the specimen contact diameter to increase. This results in an increase in the surface area in contact with the platens, which in turn increases the Tresca coefficient of friction for a subsequent increment in height reduction. Note that the sliding component of friction does not increase with increasing contact surface area since Amonton's law of friction states that the area of the contact body has no influence on the magnitude of the Coulombic coefficient of friction. Therefore, the FEM solution for the Tresca coefficient of friction is an average value that is taken over the entire compression cycle.

Table 4.2 also shows that the simulated contact diameter (d_{sa}) is always less than the actual contact diameter (d_{ca}). The difference in the simulated and actual contact diameters may be inherent to the relationship that ABAQUS® 6.2 Standard employs in solving the contact diameter based on the input of an average value for the Tresca coefficient of friction. In other words, in reality, the amount by which the increase in the contact diameter affects the Tresca coefficient of friction with increasing height reduction may be different from what ABAQUS® 6.2 Standard determines during the FEM simulations. If this was the case, then the Tresca coefficient of friction, which is solved from the FEM, would be an overestimation of the actual Tresca coefficient of friction. The FEM developed by Sheen shows that the coefficient of friction experienced during the uniaxial compression testing of AISI 304 stainless steel in the Gleeble 1500 Physical Simulator is between 0.2 and 0.3. In view of this, the magnitude of the coefficient of friction determined in the current study, that is, between 0.4 and 0.5, is high. This result is not surprising as the end faces of the platinum compression specimens used in the present study were significantly less smooth (refer to section 3.3.2 for the preparation of the platinum compression specimens) than the polished end faces of the AISI 304 stainless steel compression specimens used by Sheen. Less smooth end faces means that the overall friction condition, and hence the coefficient of friction, is higher than for smoother end faces. The extent by which the FEM overestimates the coefficient of friction has not been investigated.

The effect of the input of a Tresca coefficient of friction of zero to the FEM on the final geometry of the deformed structure simulated is shown in figure 4.13. Clearly, the structure deforms in pure uniaxial compression and maintains a perfect cylindrical (or rectangular in two-dimensions) shape at the end of the compression cycle. The level of strain is equal in all the discrete elements within the meshed structure, meaning that the specimen has deformed homogeneously throughout compression. The absence of friction in the FEM is thus to inhibit specimen sticking at the loading face so that the structure extends outward laterally along the free surface in pure sliding friction. The final deformed structure is 6.73mm in diameter, which is the exact dimension that is obtained theoretically when assuming homogeneous deformation for a specimen height reduction of 3.9mm from an initial height of 6.4mm. The logarithmic
Discussion

(LG) true strain output of 0.51 as shown in figure 4.13(a) is also the exact value of true strain that is obtained when the mode of compressive deformation is assumed to be totally homogeneous. Therefore the localised strain output is equal to the nominal strain input in the absence of sticking friction. The PEEQ true strain output of 0.647 shown in figure 4.13(b) confirms that this form of strain output from the FEM is not suitable for comparison with the nominal true strain input. The output of LG true strain is contrary to the FEM outputs generated by Sheen and Buchanan, both of whom used PEEQ true strain during their correlations of localised strain with microstructural evolutions.

The inhomogeneous distribution in the grain morphologies and the flow patterns observed across the vertical etched sections of the barrelled specimens (see figure 4.11) suggests that friction at the loading face does have a significant influence on the flow behaviour and strain accumulation of the material when compressed to significant strains (i.e. $\varepsilon > 0.3$). The FEM shows that friction causes the compression specimen to barrel significantly during deformation such that the localised strain at the centre of the specimen is significantly greater than the nominal strain input (see table 4.2). This finding is consistent with the FEM studies conducted by Evans and Schaming and Sheen. Such information provides prospective researchers with a more quantitative reference for effective strain values when attempts are made to relate the microstructural evolution of the material with the processing variables during the use of different deformation instruments, where the relationship between the nominal strain input and the effective localised strain would almost always vary.

The development of localised strain at the centre of the simulated compression specimen is shown in the plots in figure 4.14(a) to figure 4.14(d) and reveals that the localised strain increases at a near-linear rate with the nominal strain input (or specimen height reduction). The close similarity in the equations of the trend lines that have been fitted to these plots strongly indicates that the development of the effective strain at the centre of the compression specimen is independent of the hot deformation variables, that is, deformation temperature ($T$), mean nominal strain rate ($\dot{\varepsilon}$) and the nominal true strain rate ($\dot{\varepsilon}$). In addition, the identical values for the outer and contact diameters and the localised strains obtained for Pt86Al14Ru4 and Pt86Al10Cr4 for a friction coefficient of 0.45 (see table 4.1) suggest that the development of the effective strain is independent of the composition of the material. The plot in figure 4.15, however, shows that the Tresca coefficient of friction does have an effect on the localised strain value. Therefore the FEM shows that it is the friction condition (and hence the quality of the surface finish of the compression specimen) that determines the extent of barrelling and hence the extent of strain and microstructural inhomogeneities of the material during compression testing using tantalum foil lubrication. This concurs with the findings presented by Sheen. The FEM analysis also reveals that neither the deformation conditions nor the material have any direct influence on the amount of lateral spread experienced by the specimen during compression.
6.2.2 Flow stress correction due to friction

The Gleeble® I, Gleeble® II, von Mises yield criterion and Siebel friction correction models yield corrected flow curves that are significantly different for a specific compression test. It is worth noting though that the two most widely documented friction models, the von Mises yield criterion as described by Chovet et al. [56], Zhang et al. [58] and Imbert and McQueen [56] and the Siebel friction model as described by Parteder and Bünten [59], coincide very closely in terms of the flow stress over the entire range of true strain for every compression test. Several researchers, such as Chovet et al. [56], have claimed to have used the von Mises yield criterion successfully in the adjustment of uniaxial flow stress data for a particular friction condition. The plots in figure 4.19, figure 4.20 and figure 4.21 reveal that the final outer diameters as predicted by the friction models at the end of deformation for every compression test are not equal to the actual diameter of the specimen that is physically measured in the laboratory (refer to table 4.2 for the values of the actual outer diameters). Similarly, the plots show that the initial specimen diameters as predicted by the friction models are significantly larger than the actual starting diameter of 4.1 mm. Therefore it appears that the friction models do not adequately account for the effects of surface friction during the compression tests owing to the poor predictive capabilities of the these models to solve the outer diameter of the specimen. The barrelling model described by Banerjee [42], Schey et al. [77] and Narayanasamy [44,49], unlike the Gleeble® I, Gleeble® II, the von Mises yield criterion and Siebel friction models, at least predicts the outer diameter at the end of every compression cycle and the initial diameter of the specimen accurately. It should not be forgotten though that the increase in the contact diameter with strain is set to obey the equation that describes the increase in the corresponding outer diameter with strain as predicted by homogeneous deformation. This assumption may not necessarily be true but unfortunately there is at present no obvious means of predicting the variation of the contact diameter during the course of deformation. The barrelling model is therefore used to provide adjusted flow stress curves for all subsequent calculations that require flow data.

6.3 Flow stress correction due to deformation heating

Figure 4.26 shows that an increase in the rise in temperature due to deformation heating (ΔT) with true strain occurs with increasing strain rate and decreasing deformation temperature. This finding is in agreement with the studies conducted by Brand et al. [19] during an investigation of the nickel-base superalloy IN718. Although, upon initial inspection, the observed trends in ΔT with strain appear reasonable in that ΔT increases with increasing strain rate and decreasing temperature, the difference in the isothermal and adiabatic stress values actually increase with increasing deformation temperature (see figure 4.29). This is contrary to the trend expected as highlighted by the works of Semiatin and Holbrook [52] on 304L stainless steel, that is, the difference in the isothermal and adiabatic stress values should decrease with increasing deformation temperature. The source of the inaccurate construction of the isothermal flow curves is
thought to be in the actual extrapolation of the individual isothermal flow stress values from the ln(σ) versus 1000/T curves (see figure 4.28).

It is possible that certain values of ΔT calculated are sufficiently inaccurate so as to give incorrect values of isothermal stress upon extrapolation. According to the thermal transfer model described by Oh et al. and Semiatin et al., it is clear that ΔT is dependent on four properties, namely, the adiabatic correction factor (η), the energy of deformation (ΔσΔε or EDE), the material density (ρ) and the heat capacity (C). EDE was determined by means of calculating the area under the flow curve by the method of Riemann summing as demonstrated in figure 4.25. Figure 4.25 illustrates that the error associated with Riemann summing in terms of its use in estimating the area under the flow curve is very small and it is therefore unlikely that the EDE contributes significantly to the apparently erroneous ΔT data. It is equally unlikely that C impacts significantly on the accuracy of the values of ΔT. Despite C being taken as the room temperature heat capacity for pure platinum, it is not expected that this quantity would vary much with increasing temperature. C increases with temperature until the Debye temperature is reached, after which it remains constant with any further increase in temperature. The Debye temperature is in the vicinity of room temperature for metallic materials and therefore it is unlikely that the room temperature value taken for C would vary significantly at the high temperatures of deformation investigated. In addition, although C is taken for pure platinum, it would not impact qualitatively on the rate of increase in ΔT with strain between the different deformation temperatures. The value of ρ, however, could influence the accuracy of ΔT, since the density of the material would decrease with increasing temperature due to the thermal expansion associated with increased atomic mobility at high temperatures. The value of η would also affect the accuracy of ΔT. Although η was taken as 1.00, 0.95, 0.90 and 0.85 for strain rates of 1.2/s, 0.5/s, 0.1/s and 0.06/s respectively according to Dadras and Thomas, Goetz and Semiatin have shown that strain affects the value of η in such a way that the relationship between strain rate and η described by Dadras and Thomas is very rarely valid. It is impossible to assess which values of ΔT are inaccurate and it is also not possible to estimate the error associated with such inaccuracies; however, it can be concluded that no useful deductions may be inferred about the affects of deformation heating of the true flow behaviour of Pt60Al10Ru4 within the confines of the limited experimental results.

6.4 Kocks-Mecking plots

The Kocks-Mecking plots have been conducted according to the procedures outlined by Davenport et al. The sharp changes in the work hardening rates, which arise from the slight fluctuations in the Zener-Hollomon parameter that occur during the non-constant strain rate compression testing, are filtered so as to reduce the associated scatter. This means that the actual values for the critical strains required for the onset of dynamic recrystallisation should be viewed with caution. The importance of the barrelling model is
illustrated in figure 4.41 to figure 4.44. Figure 4.41 shows no obvious relationship between the critical strain and the strain rate and figure 4.43 indicates that the critical strain decreases with increasing temperature. However, the increase in the critical strain with increasing strain rate and decreasing deformation temperature observed for Pt_66Al_10Ru_4 when using the flow data that has corrected for friction (see figure 4.44 and figure 4.43 respectively) is more reasonable in that the trend is consistent with that reported by Poliak and Jonas for an aluminium killed plain low carbon steel and for a type 305 stainless steel. It is not possible, within the framework of the experimental procedure employed, to conduct Kocks-Mecking plots according to the procedure developed by Poliak and Jonas for non-constant strain rate compression testing since flow data from constant strain rate testing is needed in this analysis. Nonetheless, despite the contention surrounding the exact values of the critical strains, it is possible to conclude that Pt_66Al_10Ru_4 and Pt_66Al_10Cr_4 do undergo dynamic recrystallisation during the course of the hot compression tests in the instances where the Kocks-Mecking plots display inflection points.

6.5 The deformed state

6.5.1 Mechanism of restoration

Figure 5.16 shows the deformed microstructures of Pt_66Al_10Ru_4 within the regions that are represented by the effective localised strains determined from the FEM analysis. The deformed microstructures suggest that the dominant mechanism of softening is dynamic recrystallisation, which is characterised by heavily deformed grains with highly irregular, wave-like grain boundaries. During hot compression testing, the grains within the material are deformed due to the applied load and as a result the dislocation density is raised within the grains. However, due to the applied temperature which is sufficiently high, these dislocations are able to recover and reconfigure themselves into lower energy arrangements. When the deformation conditions favour dynamic recrystallisation, then this process of reconfiguration usually involves the migration of dislocations at high angle boundaries within the deformed material, which are usually prior grain boundaries. As a result, the dislocation density is reduced in these vicinities of the material through the reduction of stored energy by recrystallisation.

During dynamic recrystallisation, the process of deformation and restoration is repetitive and cyclic throughout the course of the compression stroke. During this cyclic process, dislocations are continuously generated within the grain and are able to migrate and annihilate at the pre-existing grain boundaries. Since the dislocation density in the newly recrystallised grains continuously increases, the driving force for the reduction of surface energy through further growth of these recrystallised grains is reduced. Therefore the cycle of dislocation generation and grain boundary sliding during dislocation migration facilitates grain refinement and when the deformation event ceases, the frozen/quenched microstructure represents a "snapshot" of this process. This is seen in the portions that have been highlighted in the micrographs.
shown in figure 5.16(a), figure 5.16(b), figure 5.16(e) and figure 5.16(f) and to a lesser extent in figure 5.16(c), Figure 5.16(d) and Figure 5.16(g). The wave-like nature of the grain boundaries of both the original and newly recrystallised grains is a consequence of the process of freezing in the microstructure of the deformed material as dislocations migrate at high angle boundaries. From figure 5.16(a) to figure 5.16(g) the manner in which the grains tend to elongate in the quenched microstructure can be seen. This is a natural consequence of the work hardening process which leads to a texturing effect of the grains and elongation primarily in the direction normal to the principal loading axis.

6.5.2 Grain size effects and microhardness analysis

The size of the dynamically recrystallised grains is governed by the deformation conditions, that is, strain, strain rate and temperature. These conditions are not easily established during hot, variable strain rate uniaxial compression testing. As dealt with in previous discussion sections, friction causes specimen barrelling which in turn causes an inhomogeneous strain distribution within the compression specimen. In addition, the variation in strain rate with increasing height reduction of the specimen means that a mean nominal strain rate has to be defined. Also, straining at higher deformation rates causes an adiabatic rise in temperature within the specimen which can affect the normal flow pattern of the material during deformation. Finally, the problem of overshoot, which is specific to the testing technique employed in the current study, causes an accumulation of excess strain at higher deformation rates.

The value of determining the Zener-Hollomon parameter for Pt₆₀Al₁₀Ru₄ as a function of the deformation conditions is demonstrated in the inspection of the deformed microstructures in figure 5.17. The inverse relationship between the Zener-Hollomon parameter (Z) and the ranking of the recrystallised grain sizes (RX-GS) (which have now been provisionally identified as dynamically recrystallised grains) for the set of deformation conditions employed can be seen from table 5.1. This observed trend is in agreement with what is expected for dynamic recrystallisation, that is, an increase in strain rate and strain as well as a decrease in deformation temperature results in an increase in Z. An increase in Z implies an increase in flow stress and stored energy, which provides a greater driving force for dynamic recrystallisation, thereby leading to a finer dynamically recrystallised grain size.

The microhardness values also increase with increasing Z (see table 5.1), which is expected as higher Z would lead to a finer grain size. A finer grain structure has a higher total grain boundary volume than a coarser grain structure and thus presents larger resistance to dislocation motion which leads to higher microhardness. Microhardness is also a function of the amount of residual stress in the material that results from the residual strain imposed by the dynamic nature of the hot deformation process. The plot of microhardness against Z gives a logarithmic relationship (see figure 6.1) and the equation used to fit the
relationship could be used to predict the microhardness of Pt\textsubscript{60}Al\textsubscript{10}Ru\textsubscript{4} as a function of Z and hence as a function of different deformation conditions.

![Plot of average microhardness against Z displaying a logarithmic relationship.](image)

\(y = 26.823 \ln(x) - 19.8\)  
\(R^2 = 0.9878\)

**Figure 6.1:** Plot of average microhardness against Z displaying a logarithmic relationship.

6.5.3 Interpretation of true stress-true strain data

(a) Pt\textsubscript{60}Al\textsubscript{10}Ru\textsubscript{4}

The deformed microstructures in figure 5.16(f) and figure 5.17(f) demonstrate the importance of the barrelling model used for the correction of the flow curves due to frictional effects. The uncorrected flow curves for 1300°C (see figure 4.17(c)) indicate a continued increase in flow stress with true strain, which suggests that the dominant mechanism during deformation is work hardening. However, after adjusting the flow curve for friction according to the barrelling model, a peak in the flow stress can be seen (see figure 4.23). This peak suggests the occurrence of dynamic recrystallisation at 1300°C and that at strains greater than the peak strain, the effect of dynamic recrystallisation on the continuous reduction in flow stress outweighs the effect of work hardening in raising the flow stress of the material during deformation. This is achieved by means of the rate of the annihilation of dislocations and the nucleation of new grains exceeding that of the increase of the dislocation density within the material being deformed. Also, a steady state in flow stress is indicated by the uncorrected flow curves for 1400°C at 0.06/s (see figure 4.16(a)) and 1350°C at 0.1/s (see figure 4.16(b)), which suggest that the rate of flow stress reduction due to dynamic recrystallisation is balanced by the rate of the increase in flow stress due to work hardening. After adjusting the flow stress according to the barrelling model, the corrected flow curves show that a steady state in flow stress is not attained during the nominal strain input at 1400°C (at 0.06/s) (see figure 4.22) and 1350°C (at 0.1/s) (see figure 4.23).
The appearance of the flow curve for 1400°C at 0.5/s (see figure 4.16(c)) is peculiar in that after having attained a peak stress at about 0.18 strain, the flow stress decreases after which it increases again at about 0.35 strain. The unexpected increase in flow stress is probably due to some unfavourable friction condition that has a profound affect on the flow stress as a result of poor specimen surface finishing. Application of the barrelling model eradicates this anomalous increase in flow stress and the corrected flow curve at 1400°C (0.5/s) shows that a steady state in flow stress is reached within the nominal strain input.

The flow curve at 1250°C (at 0.1/s) does not show any signs of dynamic recrystallisation even after having been adjusted for frictional effects (see figure 4.23) despite observing microstructural evidence for dynamic recrystallisation in the quenched microstructure (see figure 5.16(g)). The reasons for this apparent discrepancy remains unclear, however, two possible theories are offered:

1. The flow stress adjustment according to the friction model employed may be inadequate under the specific friction and deformation conditions experienced during the compression of Pt$_6$Al$_{14}$Ru$_4$ at 1250°C. The barreled diameter ($d_b$), which is used to determine/adjust the flow stress due to friction according to the barrelling model (see equation 4.1), is dependent on the contact diameter ($d_c$). The variation of $d_c$ with strain is in turn set to obey the equation that is used to describe the variation of the outer diameter of the specimen with strain due to homogeneous deformation, which is always a linear relationship. This may not necessarily be the case; that is, the variation of $d_c$ with strain may not be linear under every set of deformation conditions. The implication is that the actual flow curve at 1250°C (at 0.1/s) could indeed display a peak stress if the true value of $d_c$ or $d_f$ could be known at each strain increment. Unfortunately, there is no way of establishing the true relationship between either $d_c$ or $d_f$ with strain experimentally or analytically.

2. The material might not recrystallise dynamically at 1250°C and the microstructural evidence for this softening mechanism (see Figure 5.16(g)) could be an artefact of metadynamic recrystallisation that occurs during the period between quenching from the deformation temperature to room temperature. The stored energy in the material provided by the deformation process provides a driving force for recrystallisation by a metadynamic mechanism through the reduction of the stored energy available at the end of the deformation cycle as the temperature drops to room temperature during quenching stage. If this was the case, then metadynamic recrystallisation would certainly have to be the dominant softening mechanism at deformation temperatures greater than 1250°C. The propensity for dynamic recrystallisation is greater at higher deformation temperatures where atomic mobility and hence dislocation migration and restoration is increased. During dynamic recrystallisation, grain boundaries align favourably so as to facilitate grain boundary sliding just prior to a subsequent nucleation process. A nucleus for subsequent recrystallisation is thus already in existence so that when quenching commences, the process of metadynamic recrystallisation is made easier at higher deformation temperatures. The flow data analysis does suggest the occurrence of dynamic recrystallisation at
deformation temperatures higher than 1250°C, so that if metadynamic recrystallisation takes place it would mask the microstructural evidence of dynamic recrystallisation in the quenched state.

A plot of the mean flow stress estimate (MFSE) against deformation temperature, which is shown in figure 6.2, shows that a relatively large reduction in the total flow stress is obtained upon deformation at temperatures greater than 1250°C. The MFSE is given by the energy required for deformation (EDE), that is, the area under the flow curve that had been corrected for friction, divided by the nominal strain input. The large reduction in MFSE could be attributed to the large decrease in flow stress associated with the annihilation of dislocations and nucleation of new grains that is characteristic of dynamic recrystallisation. The exercise of distinguishing grains which recrystallise dynamically from those which recrystallise metadynamically would involve a detailed investigation, either by TEM (Transmission Electron Microscopy) or EBSD (Electron Back-Scattered Diffraction) mapping methods, during which the texture of individual grains would be examined. This is beyond the scope of the present project.

![Figure 6.2: Plots of mean flow stress estimate (MFSE) against deformation temperature (T) showing the large reduction in flow stress at deformation temperatures greater than 1250°C.](image)

\(\text{MFSE (MPa)}\)

\(\text{T (°C)}\)

(b) \(\text{Pt}_{86}\text{Al}_{14}\text{Cr}_4\)

The load-displacement data that was gathered during compression testing is converted into true stress and true strain data respectively using the appropriate expressions so as to assess the hot deformation behaviour of \(\text{Pt}_{86}\text{Al}_{14}\text{Cr}_4\). The true stress curves in figure 5.13 reveal that the metal shows extensive high temperature ductility during hot compression testing at 1400°C at both 0.1/s and 1.1/s. The shape of the curves at 1400°C is typical of the flow curve for a metal which undergoes dynamic recrystallisation. The true (flow) stress increases rapidly during the initial stages of loading due to the effects of work hardening, after which
the rate of increase of true stress gradually decreases due to restorative events until a peak stress is attained. Thereafter, the true stress decreases owing to a large reduction in stored energy by dynamic recrystallisation until some steady state stress level is reached, where the rate of work hardening is balanced by that of restoration. Both the corrected and uncorrected true stress curves show that Pt$\text{66Al}_{10}\text{Cr}_4$ undergoes dynamic recrystallisation at 1400°C (at 0.1/s and 1.1/s).

The true stress curves at 1300°C (at 1.1/s), 1200°C (at 0.1/s) and 1100°C (at 0.1/s) (see figure 5.12) show that Pt$\text{66Al}_{10}\text{Cr}_4$ does not undergo significant softening under the conditions of the nominal strain inputs and strain rates investigated. The curves are characteristic of a metal which undergoes substantial work hardening during continuous straining. The true stress increases rapidly during the early stages of straining due to work hardening and then decreases gradually as a response to restoration, however, the true stress continues to increase throughout the course of deformation. This deformation behaviour is indicative of a material which, under a given set of deformation conditions, cannot soften sufficiently in order to reduce the rate of increase in true stress due to work hardening beyond the rate of decrease in true stress due to restoration. Note that the true stress data at 1100°C (at 0.1/s), 1200°C (at 0.1/s) and 1300°C (at 1.1/s) has been recorded until about 350MPa, which represents the approximate level of true stress that corresponds to the maximum load of 5kN recordable by the load cell employed during experimentation. Also, the nominal strain input of about 0.7 at 1400°C and 1.1/s is attributed to the overshoot accumulated whilst attempting a target nominal strain of 0.5 at this strain rate.

The inflection point at a true strain of about 0.18 in the true stress curve at 1200°C could be due to specimen buckling. During compression at 1200°C, the deformation temperature is not sufficiently high to allow efficient metal flow and the expansion of the specimen along the free surface is hindered. Therefore, instead of the specimen continuing to be reduced vertically, it caves in along the direction perpendicular to the loading axis. As a result, the LVDT detects a reduced rate in the increase in displacement as the compression specimen continues to buckle under the applied load and this information is logged and translated into the true stress data, owing to the kink observed true stress curve.

### 6.6 Post-stress annealing

Post-stress annealing heat treatments were conducted on Pt$\text{66Al}_{10}\text{Cr}_4$ that was deformed at 1400°C (at 0.1/s and 1.1/s), 1300°C (at 0.1/s) and 1200°C (at 0.1/s) in an attempt to reprecipitate $\gamma'$. The micrographs in figure 5.26(a) and figure 5.26(b) show a slightly larger average statically recrystallised grain size at 1.1/s at 1400°C than at 0.1/s at 1400°C. This observation is opposite to what is expected for dynamic recrystallisation, that is, a smaller recrystallised grain size at the higher strain rate. During straining at a higher deformation rate, more deformation energy is stored in the material, which increases the propensity for restoration and hence recrystallisation in a response to relieve the internal stress. The difference in the
average statically recrystallised grain size between 0.1/s and 1.1/s is relatively small despite the difference in strain rate being a full order of magnitude. This is attributed to the countering effect of strain on the size of statically recrystallised grains.

There is sufficient time allowed during static recrystallisation for significant grain growth and therefore an increase in the statically recrystallised grain size is obtained with an increase in strain. This is unlike dynamic recrystallisation, where insufficient time is available during the compression event for grain growth of any significance and thus a decrease in the dynamically recrystallised grain size takes place with an increase in strain. Therefore, it can seen that the increase in the statically recrystallised grain size with higher strain rate (1.1/s) is countered to a certain extent by the decrease in the statically recrystallised grain size with higher strain (0.7) associated with overshoot, during a post-annealing heat treatment performed on Pt_{60}Al_{10}Cr_{4} (which was deformed at 1400°C at 0.1/s and 1.1/s) at 1200°C for 4 hours.

The average statically recrystallised grain size is smallest for Pt_{60}Al_{10}Cr_{4} deformed at 1300°C and 0.1/s (see figure 5.26(c)). The recrystallised grains grow dynamically with more difficulty at a lower deformation temperature due to less thermal energy being available for atomic diffusion. Therefore, the dynamically recrystallised grain size is smaller and hence the total volume of grain boundaries is larger at a lower deformation temperature. A larger volume of grain boundaries means that more sites are available for the nucleation and subsequent growth of new statically recrystallised grains during the post-stress annealing heat treatment performed on Pt_{60}Al_{10}Cr_{4} deformed at 1300°C than at 1400°C at the same strain rate and nominal strain input.

6.7 Formability during forging

This section discusses the suitability of the three platinum-base alloys under investigation for a turbine disc forging operation. It is not the intention to attempt to develop a forging schedule for the material or to model the material behaviour during deformation since the experimental data gathered during the course of this study is insufficient for tasks of this magnitude. Therefore, instead of developing a processing route, the emphasis is deliberately placed on the potential of these alloys to meet the microstructural requirements needed for finished superalloy turbine disc products and the ability of the material to flow freely during a forging blow without suffering cracking.

6.7.1 Pt_{60}Al_{10}Ru_{4}

Pt_{60}Al_{10}Ru_{4} shows the greatest potential for ductile flow during a forging stroke of significant strains (ie. true strains above 0.3) over a wide range of deformation temperatures and strain rates. The metal shows no signs of cracking between 1250°C and 1400°C at 0.1/s and between 0.06/s and 1.2/s at 1400°C. This is
predominantly due to the softening effects of dynamic recrystallisation. Generally, the extent of grain size refinement at all the deformation conditions investigated is appreciable, which is largely attributed to the restorative effects of dynamic recrystallisation that occurs during the forging blow itself and possible effects of metadynamic recrystallisation that could be taking place during the period of cooling from the deformation temperature to room temperature.

Although dynamic recrystallisation facilitates extensive metal softening during the stroke, the volume fraction of finished material that has recrystallised has not been determined. A large volume fraction of recrystallised material is obviously needed to impart the properties associated with recrystallised grains to as large a portion of the material as possible. There is the possibility of achieving a large volume fraction of recrystallised material after single stroke forging at lower deformation temperatures and higher strain rates, where the propensity for dynamic recrystallisation and hence the volume fraction of dynamically recrystallised grains is greater. This is, however, constrained by the load capacity of the load cell and the flow rate through the pump of the Theta® Deformation Dilatometer. EBSD mapping would have to be conducted to positively determine the volume fractions of recrystallised material.

During most industrial forging operations, the preform forging stage comprises of several blows and interstroke periods during which the billet is reduced incrementally along its height. Therefore it should be possible to achieve high volume fractions of recrystallised material by means of selecting the appropriate forming parameters during all blows and holding periods so as to control the desired microstructural evolution. The volume fraction of the grain boundaries within the material is increased after the first blow, which means that the number of potential nucleation sites for recrystallisation is greater for each subsequent blow. The potential for obtaining high volume fractions of recrystallised material can thus be achieved by a multi-step forging schedule during which deformation is conducted at as low a temperature as possible and at high strain rates, as well as by suppressing grain growth during the interstroke periods.

During a forging operation, consideration should always be offered to the heating capabilities of the forging equipment. For example, although grain refinement is greatest at 1.2/s at 1400°C, it will be very difficult to practically preheat and soak an entire billet to temperatures that high. Therefore it may be necessary to explore the forging behaviour of the material in greater depth at lower deformation temperatures. However, the load capabilities of the tooling equipment would have to be taken into account as it may not necessarily meet the requirements for forging at lower temperatures, particularly at higher strain rates where the flow stresses are higher. In addition, care should be taken to conduct each forging blow at strains that exceed the critical strains required for the onset of dynamic recrystallisation in order to ensure sufficient softening to prevent cracking and to allow for substantial grain refinement.

Unfortunately, Pt₉₆Al₁₉Ru₄ shows no signs of γ' formation before and after hot deformation and can for this reason not be used for superalloy applications. Although research at Mintek has shown that γ' formation
can be achieved in Pt_{66}Al_{10}Ru_{4} after homogenisation at 1350°C for 96 hours followed by a slow furnace cool to room temperature\(^{36}\), it has also been shown that such precipitates are of the D\(_{001}\) form\(^{36}\) and Pt_{66}Al_{10}Ru_{4} can therefore not be classified as a superalloy. Nonetheless, the forging results presented on Pt_{66}Al_{10}Ru_{4} are valuable in that they provide some insight to the mechanisms of deformation and softening that operate within certain regimes of temperature, strain and strain rate. Open reports of such a nature have never before been made for any platinum-base material and hence the contribution in this context is significant.

6.7.2 Pt_{66}Al_{10}Cr_{4}

Pt_{66}Al_{10}Cr_{4} undergoes ductile flow during a single stroke at a deformation temperature of 1400°C at both 0.1/s and 1.1/s, without any evidence of cracking in the finished specimen. Once again, this is attributed to the extensive softening due to the restorative effects of dynamic recrystallisation, which is confirmed both by the analysis of the flow data and microstructural observations. The material displays similar flow behaviour at 1300°C (at 0.1/s) but unfortunately the flow behaviour at 1300°C (at 1.1/s) cannot be assessed due to the restriction of the load cell of the Theta® Deformation Dilatometer.

It should be noted that a multi-stroke forging schedule allows for other opportunities for significant billet height reduction. For example, it may be possible to inflict the majority of the billet height reduction during the early stages of the preform forging stage, say at 1300°C (at 0.1/s) where dynamic recrystallisation takes place. The holding periods between strokes can then be tailored so as to allow the metal to recrystallise metadynamically in such a way that the residual stresses developed during previous blows are relieved without allowing significant grain growth. The final blow can then be conducted at a temperature where a high volume fraction of recrystallised material can be obtained.

6.7.3 Pt_{66}Al_{10}Cr_{3}Ru_{3}

The high volume fraction of \(\gamma'\) embedded in the \(\gamma\) matrix of Pt_{66}Al_{10}Cr_{3}Ru_{3} after homogenisation is extremely desirable. Unfortunately it is not possible to forge this material under the deformation conditions investigated. This is largely attributed to the resistance of \(\gamma'\) to coarsening and/or dissolution during the holding periods prior to deformation even at temperatures as high as 1400°C. The \(\gamma'\) precipitates obstruct the movement of dislocations during restoration processes thereby causing dislocation pileups and high residual stresses in certain areas of the material. It may be possible to forge this material at lower strains and higher deformation temperatures, although the latter will require a specialised form of heating if an entire billet is to be preheated to temperatures above 1400°C during the preform forging stage. Alternatively, an entirely new approach to developing a forging schedule can be adopted, such as
starting the billet in the cast state rather than in the homogenised condition prior to the first forging stroke. The $\gamma'/\gamma$ microstructure would then have to be evolved during the final strokes of a multi-step preform forging operation.
CHAPTER 7

SUMMARY

1. The homogenisation treatment produces a high volume fraction of γ' arranged in a dendritic-like network in Pt_{60}Al_{14}Cr_{3}Ru_{3}; a low volume fraction of irregularly shaped γ' in Pt_{60}Al_{10}Cr_{4}; and a platinum-rich solid solution devoid of γ' in Pt_{60}Al_{10}Ru_{4}.

2. The high temperature proof strength of Pt_{60}Al_{14}Cr_{3}Ru_{3} is always greater than that of Pt_{60}Al_{10}Cr_{4} and Pt_{60}Al_{10}Ru_{4}, whilst the high temperature proof strength obtained for Pt_{60}Al_{10}Ru_{4} at and above 1250°C is higher than that for Pt_{60}Al_{10}Cr_{4}.

3. The change in the high temperature proof strength with deformation temperature for Pt_{60}Al_{14}Cr_{3}Ru_{3} does not show a transition point since the alloy does not undergo any phase transformations during the high temperature compression tests. The alloy resists γ' coarsening and/or dissolution between 1100°C and 1400°C during compression testing.

4. The change in the high temperature proof strength with deformation temperature for Pt_{60}Al_{10}Ru_{4} does not show a transition point since the alloy does not undergo any phase transformations during the high temperature compression tests. The alloy remains single-phased and devoid of γ' during compression testing.

5. The change in high temperature proof strength with deformation temperature for Pt_{60}Al_{10}Cr_{4} shows a transition point between 1200°C and 1300°C, which is in part due to the different phase transformations that take place during compression testing above and below the homogenisation temperature. The alloy undergoes γ' dissolution at 1300°C and 1400°C, whilst the increase in the volume fraction of γ' and the evolution of γ'' at 1200°C as well as the poor softening kinetics at
1100°C mean that the high temperature proof strength of the alloy is significantly higher at 1300°C and 1400°C than at 1200°C and 1100°C.

6. Pt$_{86}$Al$_{10}$Ru$_4$ was deformed to true strains of 0.4 and higher without cracking during single-stroke hot compression testing at deformation temperatures ranging from 1250°C to 1400°C at 0.1/s and strain rates ranging from 0.06/s to 1.2/s at 1400°C.

7. Pt$_{86}$Al$_{10}$Cr$_4$ was deformed to true strains of 0.4 and higher without cracking during single-stroke hot compression testing at deformation temperatures of 1400°C (at 0.1/s and 1.1/s) and 1300°C (at 0.1/s). Specimen buckling occurs at 1300°C (at 1.1/s), 1200°C (at 0.1/s) and 1100°C (at 0.1/s).

8 Specimen buckling and cracking within the material is commonly observed in Pt$_{86}$Al$_{10}$Cr$_3$Ru$_3$ after high temperature compression testing. No obvious signs of significant ductile flow are detected at deformation temperatures between 1100°C and 1400°C.

9. Metallographic analysis of the hot worked microstructures alone cannot disseminate whether the evidence of recrystallisation observed in the deformed states is due to a dynamic or a metadynamic mechanism.

10. The stress peaks observed in the flow curves and the inflection points in the Kocks-Mecking plots for the flow data that had been adjusted for frictional effects suggest that dynamic recrystallisation occurs in Pt$_{86}$Al$_{10}$Ru$_4$ at 1400°C (at 0.06/s, 0.1/s, 0.5/s and 1.2/s), 1350°C (at 0.1/s) and 1300°C (at 0.1/s). Similar evidence suggests that Pt$_{86}$Al$_{10}$Cr$_4$ undergoes dynamic recrystallisation at 1400°C (at 0.1/s and 1.1/s) and at 1300°C (at 0.1/s).

11. The extent of grain refinement and the value of the microhardness of Pt$_{86}$Al$_{10}$Ru$_4$ increases with increasing Zener-Hollomon parameter.
The high temperature mechanical properties and material behaviour of three platinum-base alloys have been investigated. The following conclusions are noteworthy:

1. The high temperature proof strength of Pt$_{86}$Al$_{10}$Cr$_4$ is not governed by the homogenised microstructure when deformation is carried out in the vicinity of the homogenisation temperature and at temperatures greater than the homogenisation temperature. Instead, it is the phase transformations that occur during soaking at the deformation temperature, prior to the onset of deformation, which largely determines the high temperature strength of Pt$_{86}$Al$_{10}$Cr$_4$.

2. Pt$_{86}$Al$_{10}$Ru$_4$ and Pt$_{86}$Al$_{14}$Cr$_3$Ru$_3$ do not undergo phase transformations during compression testing and therefore no transition points in the high temperature proof strength of these alloys are observed.

3. The extensive grain refinement by the recrystallisation processes at 1400°C in Pt$_{86}$Al$_{10}$Ru$_4$ and Pt$_{86}$Al$_{10}$Cr$_4$ is encouraging; however, further experimentation is required in developing significantly higher volume fractions of recrystallised material.

4. Pt$_{80}$Al$_{14}$Cr$_3$Ru$_3$ cannot be compressed to significant strains at 10$^{-2}$/s and specimen buckling and cracking commonly occurs in this alloy at 10$^{-2}$/s.

5. The problems associated with friction during uniaxial compression testing complicate the interpretation of the flow data such that it is impossible to extract quantitative results concerning the hot formability of the material from such analysis. Nonetheless, the flow data analysis, in conjunction with microstructural evidence, provides some insight to the mechanisms of restoration.
CHAPTER 9

RECOMMENDATIONS FOR FUTURE WORK

This study has established a basis for future work. Two areas of focus have been identified:

1. issues concerning the testing of experimental material
2. issues concerning instrumentation

6.1 Material testing

1. The starting microstructures should be optimised prior to embarking on a more extensive set of hot deformation tests. This can be achieved by further experimentation with the heat treatment parameters during the homogenisation process. The current study has already established that a significantly larger volume fraction of \( \gamma' \) can be obtained in Pt\(_{45}\)Al\(_{10}\)Cr\(_4\) during a post-stress annealing treatment. Similarly, the development of the Pt\(_3\)Al phase in the starting state of Pt\(_{45}\)Al\(_{10}\)Ru\(_4\) by means of slow cooling the material from the homogenisation temperature should be pursued. Testing of Pt\(_{45}\)Al\(_{10}\)Ru\(_4\) containing Pt\(_3\)Al, albeit of the D\(_{024}\) form, would facilitate an easier comparison of the hot deformation behaviour of this material to that of Pt\(_{45}\)Al\(_{10}\)Cr\(_4\) and Pt\(_{45}\)Al\(_{14}\)Cr\(_3\)Ru\(_3\), which also comprise of Pt\(_3\)Al embedded in (Pt) matrices in the homogenised condition.

2. The deformation conditions employed for each alloy should be the same so as to enable a more direct and comprehensive comparison of the flow properties and high temperature proof strength values between each alloy. On several occasions during the course of testing, it was possible to obtain a value for the high temperature proof strength of Pt\(_{45}\)Al\(_{10}\)Cr\(_4\) but the alloy did not show signs of extensive ductile flow. Therefore, if the availability of material permits, the deformation conditions used to obtain high temperature proof strength values and those used to assess the flow behaviour should be different. Ideally, high temperature proof strength values should be obtained over as wide a range of deformation temperature as possible, whilst the flow behaviour need only be assessed within a much smaller range of deformation temperatures where the material displays extensive ductile flow.

3. For each alloy, the change in the thermal diffusivity with temperature should be obtained experimentally in the laboratory. This material property is needed as an input parameter for a thermal/thermo-mechanical Finite Element Model simulating the temperature distribution within the
specimen during hot deformation. Such a model would predict the temperature rise at the centre of the compression specimen due to deformation heating, thereby allowing a more accurate reconstruction of the isothermal flow curves.

6.2 Instrumentation

1. Constant strain rate compression testing would eliminate the scatter in the work hardening rates obtained during non-constant strain rate testing by ensuring that a constant Zener-Hollomon parameter is maintained during the course of compression. A recommended facility for future testing is the Gleeble® 3500 Thermo-mechanical Simulator, which is capable of conducting constant strain rate compression tests. Unfortunately, such an apparatus is not presently available in South Africa. Alternatively, the Gleeble® 1500 Thermo-mechanical Simulator, which has constant velocity capabilities, can be modified to provide constant strain rate compression tests with the aid of the installation of a servo-hydraulic pump. This pump action can be programmed to achieve constant strain rate compression by means of exponentially reducing the ram velocity with increasing height reduction. Such modifications have been successfully reported by Zhang et al. and Poliak and Jonas. The Gleeble® 1500 is also a cheaper alternative to the Gleeble® 3500 version and there is currently a model in operation at the University of Pretoria. Alternatively, a similar modification could, in theory, be performed to provide a customised form of the Theta® Deformation Dilatometer.

2. The Gleeble® Thermo-mechanical Simulators have a built-in water-quenching facility, which is more effective in freezing-in the hot worked microstructure than the gas-quenching technique employed in this study. Water quenching could assist in solving the problems associated with the effects of possible metadynamic recrystallisation events that could be occurring during the relatively slow cooling periods that take place during gas quenching.

3. The Gleeble® Thermo-mechanical Simulators are also capable of attaining higher strain rates than the Theta® Deformation Dilatometer (about 10/s and 100/s on the standard versions of the 1500 and 3500 models respectively). The improved strain control, which is also obtainable on the Gleeble® instruments, would allow for better comparisons to be drawn between the microstructures observed at the higher and lower strain rates by eliminating the effects of the excess strain accumulated during the overshoot period at the higher strain rates.

4. The Gleeble® instruments usually require compression specimens that are 15mm in height and 10mm in diameter, which is significantly larger than the specimen size that is required for the Theta® Deformation Dilatometer. Therefore, when intending to test platinum samples, the specimen size should always be weighed against the extent of the control that is required for deformation and the rate of cooling that is needed.
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