Modelling of the Bonding Process in Powder Forging

by

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Declaration of Originality

I hereby certify that all the contents presented in this thesis are my own work under the supervision of Dr. Daniel Balint and Professor Jianguo Lin, and all the referenced and collaborating works have been given full acknowledgement. I also confirm that this thesis has not been submitted for any degrees or other purposes.

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Date:
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Abstract

Bonding between powders is a key factor that has a crucial influence on the mechanical performance of a component formed by powder forging. Consequently, modelling the bonding process is important for controlling the quality of the forged component and predicting the optimum process parameters (i.e. forming temperature, applied load and holding time). However, few numerical models have been developed to investigate this process. In the present study, a theoretical bonding model was established to predict the extent of bonding for a contact region with constant temperature and interfacial pressure, also to identify the bonding conditions for this region (i.e. a combination of temperature, pressure and time). The bonding time for a miniature component manufactured by direct powder forging (DPF) using FGH96 powders was determined at 1150 °C in terms of two applied pressures (103.5 and 198.9 MPa). The numerical predictions from the theoretical model were compared with the obtained experimental results and a good agreement was found, indicating that the model is applicable for predicting bonding between powder particles. A methodology was proposed to implement the analytical equations derived in the theoretical bonding model into finite element (FE) simulation via user subroutines, thus real-time bonding results can be predicted. Also, a micromechanical model was established for modelling the powder densification process in commercial FE software, Abaqus, which employs the hexagonal close-packed (HCP) and face-centred cubic (FCC) structures as a representative volume element (RVE) of the powder aggregate. Since the RVE model simulated the actual interaction between powders, realistic contact pressures were obtained during powder compaction. Finally, a multiscale model was developed to predict the bonding time (i.e. critical holding time) for a specified location within a forged component, which consists of a continuum FE model, the micromechanical (RVE) model, and the theoretical bonding model. The use of this multiscale model was demonstrated via a practical DPF process, which indicates promise in determining the critical value of an industrial process parameter, i.e. holding time, through which the mechanical strength of the forged components can be guaranteed.
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Nomenclature

\( F^c \) Contact force
\( F_n \) Normal component of the contact force
\( F_t \) Tangential component of the contact force
\( \omega_i \) Angular velocity
\( v_i \) Translation velocity
\( \mu_f \) Friction coefficient
\( k_n, k_t \) Spring constant
\( a \) Radius of the micro-void
\( b \) Radius of the surrounding region for a micro-void
\( a_0 \) Initial radius of the micro-void
\( b_0 \) Initial radius of the surrounding region for a micro-void
\( \dot{a}, \dot{a}_c, \dot{a}_l \) Rate of change of the micro-void radius
\( P_c \) Contact pressure
\( P_e \) External pressure applied on the surrounding region for a micro-void
\( P_i \) Internal pressure of a micro-void
\( r \) Radial distance
\( u, v, w \) Displacement
\( \sigma_r, \sigma_\theta, \sigma_\phi \) Normal stress in spherical polar coordinates
\( \varepsilon_r, \varepsilon_\theta, \varepsilon_\phi \) Normal strain in spherical polar coordinates
\( \dot{\varepsilon}_r, \dot{\varepsilon}_\theta, \dot{\varepsilon}_\phi \) Strain rate in spherical polar coordinates
\( \sigma, \sigma_p \) Applied stress
\( G \) Shear modulus
\( A' \) Material constant
\( S \) Sign of the stress
\( K, C, C_1, C_2 \) Constants
\( \gamma \) Surface free energy
\( b_v \) Burgers vector
\( A \) Power law creep constant
\( D_{V0} \) Volume diffusion pre-exponential coefficient
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Q_V$</td>
<td>Volume diffusion activation energy</td>
</tr>
<tr>
<td>$k$</td>
<td>Boltzmann’s constant</td>
</tr>
<tr>
<td>$R$</td>
<td>Gas constant</td>
</tr>
<tr>
<td>$T$</td>
<td>Temperature</td>
</tr>
<tr>
<td>$S_l$</td>
<td>Cross-section area of the micro-void</td>
</tr>
<tr>
<td>$\dot{S}_l$</td>
<td>Rate of change of the cross-section area</td>
</tr>
<tr>
<td>$\nabla \mu$</td>
<td>Chemical potential gradient</td>
</tr>
<tr>
<td>$J$</td>
<td>Total volume flux arrived at the neck surface</td>
</tr>
<tr>
<td>$j, j_B, j_V$</td>
<td>Diffusion flux of atoms per unit area per unit time</td>
</tr>
<tr>
<td>$\Omega$</td>
<td>Atomic volume</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Chemical potential</td>
</tr>
<tr>
<td>$r_n$</td>
<td>Bond neck radius</td>
</tr>
<tr>
<td>$P$</td>
<td>Applied pressure</td>
</tr>
<tr>
<td>$D$</td>
<td>Diffusion coefficient</td>
</tr>
<tr>
<td>$D_V$</td>
<td>Volume diffusion coefficient</td>
</tr>
<tr>
<td>$D_B$</td>
<td>Grain boundary diffusion coefficient</td>
</tr>
<tr>
<td>$A_B, A_V$</td>
<td>Limiting areas where flux travels</td>
</tr>
<tr>
<td>$\delta_B$</td>
<td>Thickness of boundary diffusion layer</td>
</tr>
<tr>
<td>$n$</td>
<td>Power law creep exponent</td>
</tr>
<tr>
<td>$L$</td>
<td>Length of the long strip void</td>
</tr>
<tr>
<td>$h, d$</td>
<td>Height and width of the lenticular void shape</td>
</tr>
<tr>
<td>$h', d'$</td>
<td>Height and width of the lenticular void shape after plastic deformation</td>
</tr>
<tr>
<td>$B$</td>
<td>Contact length of the void ridge</td>
</tr>
<tr>
<td>$A_l$</td>
<td>Area of the lenticular cross section</td>
</tr>
<tr>
<td>$\sigma_y$</td>
<td>Yield stress</td>
</tr>
<tr>
<td>$V_G$</td>
<td>Volume of a void in Guo’s model</td>
</tr>
<tr>
<td>$N$</td>
<td>Number of the representative elements</td>
</tr>
<tr>
<td>$m$</td>
<td>Weight of the powders required for a component</td>
</tr>
<tr>
<td>$V$</td>
<td>Volume of a forged component</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Theoretical density of the material</td>
</tr>
<tr>
<td>$\rho_r$</td>
<td>Relative density of a forged component</td>
</tr>
<tr>
<td>$\omega$</td>
<td>Bonding parameter</td>
</tr>
</tbody>
</table>
$a_c$ Current radius of the micro-void
$a_i$ Initial radius of the micro-void
$\Omega_c$ Extent of bonding for a contact region
$\Omega_{cr}$ Critical value for $\Omega_c$
$V_p$ Volume of the particles
$V_{RVE}$ Volume of the representative volume element
$r_p$ Radius of the particle
$\rho_p$ Particle density
$\rho_{RVE}$ RVE density
$\epsilon$ Normal strain
$\gamma$ Shear strain
$\hat{v}$ Velocity along axis $Z$
$\sigma_r$ Average stress
$F_{node}^{x_i}, F_{node}^{Z}$ Force along $x_i$ and $Z$ direction
$A_{Face}^{x_i}, A_{Face}^{Z}$ Area of the surface
$H, H_1, H_2$ Height of the model
$D_0$ Distance between two opposing boundary surfaces
$\sigma_a, \sigma_r$ Axial and radial stress
$\epsilon_a, \epsilon_r$ Axial and radial strain
$u_Z$ Displacement along axis $Z$
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Chapter 1  Introduction

1.1  Background and motivation

1.1.1  Direct powder forging

Powder metallurgy (PM) is an efficient method to produce net or near-net shape components. A typical PM process generally consists of three steps: powder blending, die compaction, and sintering. Optional secondary processing such as coining or heat treatment often follows to obtain special properties or enhanced precision [1]. Over the last few decades, a number of PM processes have been developed including powder forging [2], hot isostatic pressing (HIP) [3], metal injection moulding (MIM) [4], electric current assisted sintering (ECAS) [5], and additive manufacturing [6].

Powder forging is a natural extension of the conventional pressing and sintering PM process, in which a porous powder preform is forged to full density in a confined die and virtually no flash is generated [2,7,8]. Usually, the preform is heated, while the forging may also be performed using a preform in a warm or cold state [9].

Recently, a new forming process, direct powder forging (DPF), has been proposed. In this process, the vacuumed powders are heated up to a forming temperature (below the melting point of the powder material) and forged directly to the final shape using a high forming load within a short time [10,11]. Since a preform is not required, the DPF is more energy-saving and low-cost compared to the conventional PM processes. Also, readily available forging press machines can be used for this process. In terms of the following research, DPF is the main object of study.

1.1.2  Argon atomised FGH96 powder

Powder metallurgy nickel-based superalloys are usually used as high-temperature structural materials to fabricate the turbine disc which is one of the most important components in gas turbine engines [12–14]. The main powder material involved in the present work is a nickel-based superalloy, FGH96, supplied by Beijing Institute of Aeronautical Materials (BIAM).
The FGH96 powders are produced by argon atomization, and the average size is reported (by BIAM) to be 35 µm in diameter. Gas atomization is a common technique to manufacture spherical metal powders [15,16]. During the atomization process, an inert gas is introduced into a molten metal stream to form fine metal droplets which then cool down and are segregated from the air stream using gravity or cyclonic separation. The morphology of a typical argon atomised FGH96 powder employed in the subsequent analysis is shown in Figure 1.1, which was observed using a scanning electron microscope (SEM) at Imperial College London (ICL).

![Figure 1.1 A typical argon atomized FGH96 powder particle (observed using SEM).](image)

1.1.3 Bonding between powders

In terms of a powder metallurgy (PM) product, relative density is a standard parameter to evaluate its quality [2,17,18]. Also, bonding between powders is a key factor that has a crucial influence on the mechanical performance of a PM component. The influence can be explained by considering two single powder particles: imagining they are brought into contact, it will be easy to separate the two powders immediately after they come into contact, however, it becomes more difficult after a sufficient time is allowed for their contact, which is because bonding is time dependent. In a PM process, with regard to a certain contact region between powders, the time from establishing an initial contact to achieving a fully bonded state is called the bonding time, which is time for this region to obtain the required mechanical strength.
Since numerical modelling is advantageous over trial-and-error experiments in saving time and cost, various models have been proposed to investigate the PM process, and most of these models focus on the densification of powders during sintering, pressing or a combined process [19–28]. However, few models have put emphasis on the bonding between individual powders.

Instead of bonding between powders, solid-state diffusion bonding between prepared surfaces has been numerically studied for over 50 years, and many models have been proposed to simulate this process [29–36]. The aim of solid-state diffusion bonding, called diffusion bonding (DB) in the present work, is to bring the surfaces of two pieces into contact under pressure and elevated temperature such that interdiffusion can result in bond formation. In reality, because of inevitable roughness at an atomic scale on the prepared surface (arising from grinding or polishing marks that are produced during surface finishing), it is not possible to bring the two surfaces to within an interatomic distance by simple contact [37]. Thus, the DB process can be generally classified into two steps: (1) instantaneous plastic deformation of the surface asperities occurs as the pressure is applied, which generates a large volume of voids between the bonded area, and (2) the thermally activated mechanisms that lead to void shrinkage, i.e. increase of the bonded area. It has been widely accepted that the rate-determining step in DB is the elimination of the voids originating from the roughness of the two adjoining surfaces [29–36]. Also, almost all the existing DB models assume a long-ridge shape for the asperity which turns out to be reasonable in terms of the morphology of a prepared surface. Figure 1.2(a) shows the prepared surface of a nickel specimen (ground with 280# silicon carbide) [38].

When observing the local surface of argon atomized FGH96 powders, indents (including shallow channels and isolated pits) were found as seen in Figure 1.2(b), which are reminiscent of the surface asperities studied in the previous DB models as in Figure 1.2(a). Although the morphology of the powder surface is quite different from the prepared surface, a similar research methodology can be adopted, i.e. the bonding process between powders can be regarded as the shrinkage of micro-voids formed by the observed indents.

Based on the analysis above, bonding between powders cannot complete immediately after establishing contact, and a bonding time is required for the contact region to become fully bonded. Thus, the direct powder forging (DPF) process can be treated as two steps: (1) compacting the heated powders in vacuum using a high forming load within a short
time (i.e. compression time), and (2) retaining the applied load and temperature for a given time (i.e. holding time) to produce sufficient bonding between powders. Figure 1.3 shows the schematic of the DPF process. The pores formed after initial powder packing diminish during the compression mainly due to the plastic deformation of powders. Although the bonding between powders has already started (even completed) for some contact regions during the compaction, a holding time is still necessary to achieve a fully bonded state, especially for the regions that have just come into contact when the compaction finishes. During the holding time, the remaining micro-voids which are located on the interface between powders are eliminated, thus the adjoining powders are bonded together.

Figure 1.2 (a) SEM observation of the prepared surface of a nickel specimen (ground with 280# silicon carbide) [38]; (b) SEM observation of the local surface of a typical argon atomized FGH96 powder.

Figure 1.3 Schematic of the DPF process.
1.2 Aim and objectives

As mentioned in the last section, bonding between powders has a crucial impact on the mechanical performance of a powder metallurgy component. Sufficient mechanical strength can be achieved once all the powders are fully bonded together. Therefore, the aim of this research is to propose a method for modelling the bonding process in powder forging, through which the optimum process parameters for obtaining a component with required mechanical properties can be predicted. The specific objectives are as follows.

- Establish a theoretical (analytical) bonding model which is applicable to the contact between powders.
- Implement the theoretical (analytical) bonding model in an FE simulation, through which the real-time bonding results between powders can be derived.
- Develop a micromechanical model (at the powder particle level) in a finite element (FE) software, which is able to simulate the interaction between powders during the powder compaction process.
- Propose a multiscale model (including the analytical bonding model and the micromechanical model) to predict optimum process parameters for manufacturing a component via the direct powder forging technique (exemplified using the argon atomised FGH96 powders).

1.3 Structure of the thesis

The thesis is organised as follows.

Chapter 1 briefly introduces the direct powder forging process and the main powder material involved in the present work, i.e. argon atomised FGH96 powder. Also, the concept of bonding between powders is proposed. The aim and objectives of the research are presented in this chapter as well.

Chapter 2 reviews the existing solid-state diffusion bonding models, which are instructive in establishing a bonding model for powders; and gives an overview of the current models which are proposed for simulating the powder densification process.
Chapter 3 presents the specific procedure of establishing a theoretical bonding model for powders. FGH96 powder is used to demonstrate the capability of the established model including prediction of the extent of bonding for a contact region. Also, parametric studies are conducted on the two main inputs of the bonding model. Finally, the rationality of the model is verified by comparing to previous works on solid-state diffusion bonding.

Chapter 4 introduces an experimental method to investigate the bonding conditions during direct powder forging (DPF) for FGH96 powders, which comprises a DPF Gleeble test and a tensile test on a miniature component. At the end of this chapter, the experimental results are used to verify the applicability of the proposed bonding model in powder forging.

Chapter 5 provides a methodology to incorporate the theoretical bonding model in a finite element (FE) simulation, which is demonstrated via a hemisphere model to predict the bonding status of the contact surface.

Chapter 6 proposes a multiscale model to predict the bonding time for a specified location of a DPFed component. The model consists of a continuum FE model, a micromechanical model and the theoretical bonding model. The specific procedure of constructing the micromechanical model is detailed in this chapter. Finally, the use of the multiscale model is demonstrated through a practical DPF process.

Chapter 7 presents the conclusions and suggested future work.
Chapter 2   Literature Review

2.1 Review of existing solid-state diffusion bonding models

Solid-state diffusion bonding, termed as diffusion bonding (DB) in the present work, is a common welding technique by which two nominally flat surfaces are joined at an elevated temperature using an external pressure [39–41]. The DB process shares some similarities with the bonding process in powder forging: (1) the operating temperature is below the melting point of the material, and (2) an interfacial pressure is applied. These characteristics can probably result in a similar bonding mechanism for the two processes. Thus, as a good reference for establishing a bonding model for powders, it is worthwhile to conduct a review on the existing solid-state diffusion bonding models.

As mentioned in Chapter 1, surfaces asperities can always be observed on the prepared surfaces used for DB, which arise from the grinding or polishing marks produced during surface finishing [37]. Based on the common morphology of the asperities, most of the existing DB models assume a long-ridge shape for the asperity (for which the problem can be simplified by a plane strain state), while different cross-sectional shapes were assumed by the different models. Several typical initial contact geometries of two faying surfaces (before plastic deformation) are listed in Table 2.1.

<table>
<thead>
<tr>
<th>Initial contact geometry:</th>
<th>Corresponding model:</th>
</tr>
</thead>
<tbody>
<tr>
<td>rhombic</td>
<td>Derby and Wallach</td>
</tr>
<tr>
<td>Oval</td>
<td>Hill and Wallach</td>
</tr>
<tr>
<td>circular</td>
<td></td>
</tr>
<tr>
<td>lenticular</td>
<td></td>
</tr>
<tr>
<td>Corresponding model:</td>
<td>Pilling et al.</td>
</tr>
<tr>
<td></td>
<td>Guo and Ridley</td>
</tr>
</tbody>
</table>

Table 2.1 Typical initial contact geometries in different models [31,33,34,36].
Apart from the initial contact geometry, another feature that distinguishes different DB models is the bonding mechanisms involved in each model and the modelling approach adopted for each mechanism. In this section, some representative models are reviewed and up-to-date modelling techniques on DB are briefly introduced.

The first quantitative DB model was proposed by Hamilton [30], who summarised that DB is generally agreed to take place in four basic stages: (1) development of intimate physical contact, (2) formation of a metallic bond, (3) interdiffusion, and (4) recrystallization and/or grain growth across the interface. Also, he inferred that the first stage occurs through initial removal of local asperities under applied pressure, followed by the removal of micro-pores, or voids, by diffusion-aided mechanisms; this coincides with the phenomenological model proposed by King and Owczarski [42], and the viewpoint held by Kellerer and Milacek [43]. However, in Hamilton’s model, only creep deformation of asperities was considered to result in a flat and intimate contact face across the bond area.

Garmong et al. extended Hamilton’s model by adding the contribution of diffusion [29]. In their model, two different wavelengths (long- and short-wavelengths corresponding respectively to waviness and surface roughness of the joining surfaces) were superimposed to represent the surface asperities as shown in Figure 2.1. The DB process was modelled by two stages: (1) flattening of the long-wavelength asperity by creep, and (2) closure of the voids formed by the short-wavelength asperity through a combination of creep and diffusion. The diffusion mechanism in this model was described using the sintering equations which have been utilised by Coble to simulate the final stage of powder densification [44].

![Figure 2.1 Surface asperities in the model of Garmong et al. [29].](image-url)
The first fully comprehensive DB model was developed by Derby and Wallach [31,32] who considered that the mechanisms operating in DB were analogous to those in pressure sintering [45,46]. The bonding mechanisms proposed among their works were summarised by Hill and Wallach [36] as follows.

(1) Power law creep
(2) Plastic deformation
(3) Grain boundary diffusion from an interface source to a neck
(4) Volume diffusion from an interface source to a neck
(5) Surface diffusion from a surface source to a neck
(6) Volume diffusion from a surface source to a neck
(7) Evaporation from a surface source to condensation at a neck

These mechanisms cover all the possible routes of material transfer, and were divided into three groups: bulk deformation mechanisms, interface source mechanisms, and surface source mechanisms as illustrated in Figure 2.2 (assuming the void cross section shape is an ellipse). The driving forces for creep and plastic deformation are similar which is the applied pressure. For interface source diffusion, the transfer rate is governed by the chemical potential gradient along the bond interface. The mechanisms (5) to (7) are driven by the difference in surface curvature [36].

Figure 2.2 Illustration of different routes of material transfer: (a) bulk deformation mechanisms; (b) interface source mechanisms; and (c) surface source mechanisms.
In Derby and Wallach’s model, the methods for modelling each mechanism are mainly based on the previous works on sintering, which were modified for a two-dimensional geometry assumed for DB (as shown in Table 2.1). The DB process was postulated to occur in three stages: (1) initial instantaneous plastic deformation of asperities, (2) diffusion-dominant (time-dependent) stage based on voids with a small aspect ratio as in Figure 2.3(a), and (3) elimination of voids of cylindrical geometry as in Figure 2.3(b). It was indicated in their work that all the bonding mechanisms may operate either individually or in some combinations to affect each stage, hence the overall bonding rate for each stage is the sum of the individual rates of each operating mechanism. However, when applying Derby and Wallach’s model, a discontinuity was found for the predicted bonding rate, which is mainly due to the change in void geometry from the straight-sided (stage 2) to the circular (stage 3) as shown in Figure 2.3.

![Figure 2.3 Void shapes for different stages of the DB process in Derby and Wallach’s model: (a) stage 2; (b) stage 3 [31].](image)

Hill and Wallach improved Derby and Wallach’s model by proposing a different contact geometry (ellipse) with successive incremental changes in shape to circular [36]. Thus, the discontinuity of bonding rate was overcome, and due to the continuous evolution of void shape, stages 2 and 3 in Derby and Wallach’s model can be merged. Although Hill and Wallach applied the same modelling methodology as Derby and Wallach, the model for the power law creep mechanism was updated: a work for describing the growth of an elliptical hole in plane strain under power law creep [47] was modified for use in their model. Also, the predictions obtained were reported to be in good agreements with the experimental results for α-iron, γ-iron and copper [36].

Guo and Ridley followed the bonding mechanisms defined by Derby and Wallach, while assuming two concomitant modes for void closure [34]. The model for interface source
diffusion mechanisms in their work was modified by introducing a concept of effective diffusion distance [35]. Also, the models for other mechanisms were adapted according to the lenticular contact geometry assumed. A good agreement between numerical and experimental results was reported in Guo and Ridley’s work for Copper and Ti-6Al-4V alloy.

In terms of the modelling methodology proposed by Derby and Wallach, a high level of flexibility is apparent since the bonding mechanisms can be investigated individually and thus a corresponding model can be updated based on the practical situation, which has already been demonstrated in the works of Hill [36] and Guo [34]. Furthermore, the methodology is not specific to any particular material (e.g. works for both pure metal and alloy), which demonstrates its universality.

As an alternative to the sintering approach, a diffusive creep model was developed by Pilling et al. [33] for modelling DB. The work was mainly based on the theory of void growth in materials during creep, originally proposed by Chen and Argon [35], and good agreement for bonding Ti-6Al-4V was claimed. However, surface source diffusion was assumed to be sufficient to maintain the cylindrical void geometry in the Pilling model, which is not necessarily the case as the voids in micrographs often show [48]. Thus, the Pilling model appears to have been developed especially for fine-grained superplastic alloys (for which creep mechanisms might be expected to dominate) [36].

Takahashi and Inoue summarised an overview on the analytical void shrinkage models for diffusion bonding [49,50], also including models they proposed for interface diffusion and surface diffusion mechanisms [51,52]. The series and parallel operations in terms of interface and surface diffusions were discussed in their work, which depicted different modes of diffusional flow of atoms during DB [49].

The analytical models reviewed above have been used to simulate DB in terms of different materials and different processes where DB is involved [53–56]. Also, real surface roughness has been applied in the analytical model using a surface topography measuring system [57]. Although very few new theoretical models have been proposed in recent years, other modelling techniques have been applied to investigate the DB process which are introduced as follows.
Takahashi et al. adopted the finite element method (FEM) to conduct numerical analyses on the void shrinkage process controlled by power law creep \([58,59]\). In their work, the asperity with a triangular section (rhombic void) was modelled, and the effect of asperity angle (shown in Figure 2.4) was studied. It was indicated that surface roughness with large asperity angle has the advantage of breaking down the surface oxide films, which will impede the DB process.

![Figure 2.4 Schematic of the asperity angle \([58]\).](image)

Chen et al. used molecular dynamics (MD) modelling to analyse DB \([60,61]\), during which the movement of atoms in the interfacial region was observed. Through MD simulations of the DB process and subsequent tension deformation, the influences of interfacial pressure and temperature on the bonding process were studied. The simulation results suggested that when the interfacial pressure and temperature were lower than a certain value, diffusion was hardly observed. Also, it was found the thickness of the interfacial region (where the DB occurs) increased with pressure.

## 2.2 Overview of the modelling work for powder densification

As mentioned in the last chapter, little numerical research has been conducted on bonding between powders during a powder metallurgy (PM) process, while numerous models have been established to investigate powder densification during pressing, sintering or a combined process \([62–68]\). From a mechanical perspective, the powder compaction process (either at room or elevated temperature) can be generally divided into three stages: stage 0 refers to powder packing, while stages I and II deal with powder densification, which are characterized respectively by connected pores surrounding powders and isolated pores, making powder assembly behave like a porous material; the relative density of the powder assemblage during stage I is approximately in the range from 0.6 to 0.8-0.9 \([69]\). In this section, an overview was given on the modelling work for powder
densification. Since the interfacial pressure (i.e. contact pressure between powders) is a key factor for the bonding between powders (as indicated in Section 2.1), the models which can simulate the interaction between powders were highlighted in the following reviews.

Considering the scale where the models are established, there are mainly two types of distinct models for modelling the powder densification process [70]: macroscopic models and microscopic models. In macroscopic models, a constitutive law is developed to describe the densification and deformation behaviours of a porous continuum (representing a powder assemblage). This kind of model is always associated with the finite element method (FEM) to simulate dimensional changes and the density distribution of a PM component during the forming process. Microscopic models are established at the particle level, which aim to mimic the mass transfer mechanisms between particles or the deformation behaviours of discrete particles, thus a better understanding of how pores diminish can be obtained.

Some widely-used macroscopic models are as follows: Drucker-Prager Cap model [19,20], Gurson model [21,71], Shima-Oyane model [28,72] and Cam-Clay model [22,73]. These models were originally not developed for powder compaction – the Gurson model and Shima-Oyane model were developed for porous materials while the remaining two for granular materials such as rock and soil. In general, each of these models has its own yield criterion, hardening function and flow rule, through which the density evolution can be predicted. In terms of a specific material, the constitutive model should be carefully selected, since the accuracy of computed results is largely dependent on the capacity of the model employed.

A key advantage of the microscopic model is the ability to simulate individual powder interactions, which has not been achieved by the macroscopic models due to the assumption that a powder assemblage behaves as a continuous medium. Different types of microscopic models have been proposed during the last few decades. Apart from the analytical models [23,24,74], some relatively new modelling techniques have been applied to investigate the powder densification process which are detailed as follows.

The discrete element method (DEM) was originally proposed in the field of soil mechanics and granular flows [25,26,75], while recently it has been used to study powder
compaction [76–78] and sintering [79–81]. In the DEM, the object of study is represented by a large collection of rigid or deformable elements interacting with one another with contact forces [82]. It should be noted that the discrete elements can be of arbitrary shape [83]. The contact interaction between two spherical discrete elements are illustrated in Figure 2.5, in which the contact force $F^c$ can be decomposed into the normal and tangential components, $F_n$ and $F_t$ respectively. Although many mathematical models have been proposed to approximate the physical behaviour of true contacts between two elements, the most commonly used in a DEM simulation is a spring model which is schematically shown in Figure 2.6.

![Figure 2.5](image1.png)

Figure 2.5 Schematic of contact interaction between two discrete elements ($\omega_i$ and $v_i$ denote the angular velocity and translational velocity respectively, $i=1, 2$) [82].

![Figure 2.6](image2.png)

Figure 2.6 Schematic of the spring contact model in DEM ($\mu_f$ is the friction coefficient; $k_n$ and $k_t$ are spring constants) [82].

Figure 2.7 shows a typical loop of a discrete element algorithm, which mainly involves two steps. First, the contact forces are calculated for the contact pairs. Then, during the second step, the acceleration for each element is derived based on the law of motion; thus, after explicit integration, the displacement is obtained and the new location of the element can be determined.
In terms of a DEM for powder compaction, the interaction between powders was simulated as the plastic indentation of neighbouring particles and an evenly-distributed contact pressure was assumed for a contact surface [26]. Moreover, this model is valid only for powder assemblages with a relative density less than 0.82, since the assumption of independent contacts between powders does not hold at higher relative densities [84].

The multi-particle finite element model (MPFEM) was also developed for exploring the densification of powders. The main feature of MPFEM which differs from the conventional discrete element model is that every individual powder particle is discretized using a finite element mesh. Thus, a full description of the contact mechanics (including non-uniform local contact behaviour between powders) can be obtained.

In the work of Procopio and Zavaliangos [27], the MPFEM was used to extend the modelling of the powder densification process to a relative density of 0.95 (0.82 in DEM [84]) which covering both stages I and II compaction; while it is a two-dimensional (2D) model and only involves a limited number of powder particles (on the order of hundreds). Instead of periodic boundary conditions (PBC), their MPFEM introduced rigid surfaces to bound the particle assemblage, which brought about a ‘wall effect’: the particle packing pattern next to the rigid surfaces are different from that of the interior. This ‘wall effect’
results in the variation of macroscopic stress with the number of particles which was indicated as a drawback of this technique [27].

Three-dimensional (3D) MPFEM improves the accuracy of the particle simulation as compared to 2D MPFEM and DEM. It provides the necessary degrees of freedom, allowing for local non-uniform contact deformation which is not available in DEM. However the major drawback of 3D MPFEM is the incredibly long calculation times relative to DEM and 2D MPFEM [85,86].

In conclusion, DEM and MPFEM are prevalent numerical approaches to obtain macroscopic yield behaviour during the densification process by considering the contact interaction between powders at a microscopic scale. However, both methods possess shortcomings limiting their functionality in real applications: DEM assumes uniform local contact behaviour and is only valid for the powder compaction process with a relative density below 0.82 [84] (due to non-interacting contacts); while MPFEM is usually limited by the high demands on the available computational resources.
Chapter 3  Theoretical Bonding Model for Powder Forging

3.1  Model development overview

The present work aims to establish a mechanism-based model to quantitatively predict the extent of bonding for a certain contact area between powder particles during the forging process. Since the bonding process can be regarded as the shrinkage of micro-voids formed by the indents on the powder surface (as illustrated in Section 1.1.3), the extent of bonding for a certain contact area is evaluated using the percentage of micro-void size reduction, and the bonding rate is referred to as the micro-void shrinkage rate.

As presented in Section 2.1, most of the existing diffusion bonding models consider the shape of interfacial voids as a long strip while different cross-section shapes have been proposed such as circle, oval and rhombus. The assumption of such contact geometry has been reported to be effective for modelling the diffusion bonding between two prepared surfaces by grinding or polishing, etc. However, this assumption is probably not valid in terms of the surface morphology of powder particles. Figure 3.1(a) shows the scanning electron micrographs of two typical argon atomized nickel-based superalloy FGH96 powder particles. On the surface of the powders, indents can be observed including narrow and shallow channels and isolated pits. Also, it can be seen that some pits which are located at the junctions or along the channels are deeper than the channel or have larger size than the width of channels. The time required to heal the micro-voids formed by these pits would be longer than those formed by the channels, thus it is considered that the pits have a dominant influence on the bonding between powders. In the present model, only the micro-voids formed by the pits are directly included, while the influence of the channels is considered when determining the initial size of the micro-voids. Figure 3.1(b) shows the pits within a specified area on the powder surfaces, which are indicated using small circles (in red and blue). The distribution of the micro-voids between two contact surfaces are demonstrated by overlapping the two specified areas (green dashed circles). Therefore, in the present model, it seems reasonable to assume that discrete micro-voids
are located on the interface between powder particles, and this assumption would be valid for other powder materials which have a similar surface morphology as FGH96.

Figure 3.1 Analysis of the Ni-based superalloy FGH96 powder surface: (a) SEM observation results; (b) pits observed in a specified area.
The methodology of establishing the bonding model for powder forging is mainly based on Derby and Wallach’s work on modelling diffusion bonding between two prepared surfaces [31,32] due to its flexibility in quantifying the contributions of different operating mechanisms, which include plastic deformation, power law creep, interface source diffusion and surface source diffusion. Also, since each mechanism is investigated individually in their work, it appears to be more convenient to modify their corresponding sub-models, allowing the integrated bonding model to be optimised according to the practical situation. In the present work, assumptions were made based on the morphology of the powder surface, and the sub-model for each bonding mechanism was established according to the circumstance of powders (rather than prepared surfaces). The specific procedure to establish the present model is detailed in the subsequent sections.

3.2 Model assumptions

The present bonding model for powder forging was established based on the following assumptions.

a) The whole powder forging process is performed in vacuum, including filling and forging powders, etc. Therefore, the influence of an oxidation layer on the powder surface is not considered during bonding or the powder forging process.

b) The two contact surfaces bond immediately once the micro-voids between them are eliminated.

c) The pressure and temperature applied on the contact surface are constant.

d) Only solid-state diffusion bonding is considered so that no liquid or vapour phase is treated in this model.

e) The initial shape of the micro-voids is assumed to be spherical, which makes the problem more tractable. In Takahashi and Inoue’s work, it has been reported that the void morphology (except for the curvature radius at the void tip, i.e. at the point of contact between the void surface and the bonded interface) has little impact on the void shrinkage rate. Thus, it is speculated that the shape effects in the present model can be neglected.
Since the present model considers spherical micro-voids, the curvature is uniform along the void surface. Thus, surface source diffusion mechanism [31,36], of which the driving force is the difference in surface curvature, is not invoked in the following analysis.

f) Equal-size micro-voids are assumed to be uniformly distributed in a certain contact area, which is small enough to be considered as flat. The arrangement of the micro-voids and their surrounding regions are demonstrated in Figure 3.2. The determination of the initial micro-void size is demonstrated in Section 3.5.1.

Figure 3.2 Schematic of micro-voids in contact area: (a) Adjoining compressed powder particles; (b) Micro-voids formed in between contacting surfaces; (c) Arrangement of the micro-voids (\(a\) is the radius of the micro-voids and \(b\) is the radius of the imaginary surrounding region).

### 3.3 Underlying mechanisms

The bonding process is considered as the transfer of material to the micro-voids, i.e. shrinkage of the micro-voids. In Derby and Wallach’s model, a long strip void with rhombic section is assumed, and the diffusion bonding process has been treated to be analogous to that of pressure sintering [45,46]. Accordingly, the three main types of material transfer mechanisms proposed are surface source mechanisms, bond-line source mechanisms and bulk deformation mechanisms [31,32]. Based on their work, also considering that the micro-voids were assumed to be spherical in the present model, the following bonding mechanisms were postulated: (1) power law creep and (2) interface
source diffusion [87]. Both mechanisms describe a common phenomenon: transfer of materials to micro-voids, but differ in their routes and driving forces. Figure 3.3 schematically illustrates the two mechanisms.

Power law creep (also known as dislocation creep), from a macroscopic viewpoint, is an operation in which the micro-void is crushed by plastic flow of the surrounding matrix (bulk deformation), leading to a reduction in the size of the micro-void [3]. From a microscopic view, shrinkage of micro-voids arises from glide and climb of dislocations in its adjacent matrix under an applied load (vacancy diffusion also occurs during this process to facilitate dislocation climb as shown in Figure 3.3(a)). The driving force for power law creep thus originates from the external pressure applied.

Interface source diffusion refers to the transfer of materials (the essence of which is transfer of atoms) from the bonded interfaces to the micro-voids. Interface source diffusion follows one of the two routes as indicated in Figure 3.3(b), addressed respectively as grain boundary diffusion (along the interface) and volume diffusion mechanisms. (Volume diffusion, also called bulk or lattice diffusion, refers to atomic diffusion within a crystalline lattice.) Despite the difference in their routes for material transfer, the two mechanisms share the same driving force which is the gradient of chemical potential along the bond line.

![Figure 3.3 Illustration of routes of material transfer: (a) Power Law Creep (PLC) mechanism; (b) Interface Source Diffusion (ISD) mechanism.](image)

(a) Vacancy Dislocation

(b)
3.4 Mathematical Formulation

3.4.1 Power law creep

The shrinkage of the void can be quantitatively represented by the decrease of its radius. The end goal of establishing this power law creep (PLC) model is to derive an expression describing the evolution of the radius, $\dot{a}_C$. Since spherical void shrinkage in pressure sintering by power law creep has already been studied in Wilkinson and Ashby’s paper [46], this procedure was applied directly in analysing the power law creep mechanism, while an updated constitutive equation adopted by Derby and Wallach [32] was utilised in the present model.

In order to formulate the equations for the power law creep mechanism, a unit structure (indicated in Figure 3.2(c)) was considered. This unit structure was treated as a thick-walled spherical shell, which is shown schematically in Figure 3.4. A spherical coordinate system was introduced to facilitate the derivation of the equations: taking the origin of the spherical coordinate set to be at the centre of one micro-void, of initial radius $a_0$; defining the surrounding region of the micro-void by constructing an imaginary spherical surface of initial radius $b_0$. The external pressure applied on the imaginary surface (i.e. at $r = b_0$) was assumed to be equal to the interfacial contact pressure $P_c$ between powders. (This is a reasonable assumption when the ratio between the size of the imaginary surrounding region and the radius of the powder particle is smaller than 0.1.) Based on the assumption that the forging process is performed in vacuum, the internal pressure $P_i$ was set to be zero.

![Spherical polar coordinates](image)

Figure 3.4 Schematic of a micro-void and its surrounding region in the PLC model.
For the case of complete spherical symmetry,

a) The variations in stresses, strains and displacements are functions of only the coordinate \( r \).

b) The only non-vanishing stresses are \( \sigma_r \) and \( \sigma_\theta = \sigma_\phi \), while all shear stresses are zero.

c) The strains in terms of the only non-vanishing displacement \( u \) are given by \( \varepsilon_r = du/dr \) and \( \varepsilon_\theta = \varepsilon_\phi = u/r \), while all shear strains are zero.

According to the analysis above, strain rate \( \dot{\varepsilon}_\theta \) at any specified radius \( r \) is given by \( \dot{\varepsilon}_\theta = \dot{r}/r \). Since the material itself flows at constant volume, it can be obtained that

\[
\dot{\varepsilon}_r = -2\dot{\varepsilon}_\theta. \tag{3.1}
\]

As a result,

\[
\frac{\dot{r}}{r} = \dot{\varepsilon}_\theta(r) = -\frac{\dot{\varepsilon}_r}{2}(r) \tag{3.2a}
\]

so that

\[
\dot{a}_c/a = -(1/2)\dot{\varepsilon}_r(a) \quad \text{and} \quad \dot{b}_c/b = -(1/2)\dot{\varepsilon}_r(b). \tag{3.2b}
\]

It should be noted that \( a \) and \( b \) denote the radius of the micro-void and its surrounding region respectively; while \( \dot{a}_c \) and \( \dot{b}_c \) represent the rate of change of them derived for the power law creep mechanism, and the subscript is used to distinguish them from those derived for the interface source diffusion (ISD) mechanism, i.e. \( \dot{a}_I \).

In order to deduce an expression for \( \dot{a}_c \), following analysis was conducted.

a) Equilibrium: [88]

The bulk deformation by creep is regarded as a quasi-equilibrium process, which satisfies the equilibrium equation below at any instant.
\[
\frac{d\sigma_r}{dr} + \frac{2}{r}(\sigma_r - \sigma_\theta) = 0 \quad (3.3)
\]

b) The boundary conditions:

Constant inner and external pressures are applied as shown in Figure 3.4.

\[
\sigma_r(a) = -P_i \quad \text{and} \quad \sigma_r(b) = -P_e \quad (3.4)
\]

c) Compatibility: [88]

\[
\frac{d}{dr}(r\dot{\epsilon}_\theta) = \dot{\epsilon}_r \quad (3.5)
\]

d) The constitutive law:

Experiments in uniaxial tension show that, at a constant applied stress, \(\sigma\), a steady-state strain rate, \(\dot{\epsilon}\), occurs. The strain rate is related to the stress by a power law expression of the form [32]

\[
\dot{\epsilon} = A' \left(\frac{\sigma}{G}\right)^n \quad (3.6)
\]

where \(\sigma\) is the tensile stress, \(A'\) and \(n\) are material constants, and \(G\) is the shear modulus. As is shown in the equation above, the sign of the stress may change, leading to problems when \(n\) is even, or is not an integer. It is convenient to rewrite this constitutive law in the form

\[
\dot{\epsilon} = SA' \left(\frac{\sigma}{G}\right)^n \quad (3.7)
\]

where \(S\) is the sign of the stress.
In order to describe steady-state creep material flow under a multi-axial stress state, through introducing von Mises stress, Equation (3.7) was generalised in the normal way as below [46,89].

\[
\dot{\varepsilon}_r = SA' \left( \frac{\sigma_r - \sigma_\theta}{G} \right)^n
\]  

(3.8)

Since \((\sigma_r - \sigma_\theta)\) is positive here, \(S = 1\).

According to the conditions listed above, it is feasible to seek the solution to the proposed problem (expression for \(\dot{a}_C\)). First, combining Equation (3.1) and (3.5) and integrating, it could be found that

\[
\dot{\varepsilon}_\theta = \frac{K}{r^3} = -\frac{\dot{\varepsilon}_r}{2}
\]  

(3.9)

where \(K\) is a constant to be evaluated.

Substituting this result into the constitutive equation (3.8) and applying it to the equilibrium equation (3.3) gives

\[
d\sigma_r = -\frac{2G}{r} \left( \frac{-2K}{SA'r^3} \right)^\frac{1}{n} dr.
\]  

(3.10)

Integrating this between the limits indicated by the boundary conditions, Equation (3.4) and solving for \(K\) gives

\[
K = -\frac{SA'}{2} \left( \frac{ab}{b^n - a^n} \right)^n \left[ \frac{3}{2nG} (P_e - P_i) \right]^n
\]  

(3.11)

In terms of the effect of surface tension, it is usually ignored if the pressure difference \((P_e - P_i)\) is large compared with \(2\gamma/a\), where \(\gamma\) is the surface free energy of the material. When this difference is not large (the radius \(a\) is sufficiently small), surface tension needs
to be considered and the term in Equation (3.11), \((P_e - P_i)\), should be redefined as \((P_e - P_i + 2\gamma/a)\) [46].

Now combing Equation (3.2b) and (3.9), the rate of change of the micro-void radius \(\dot{a}_c\) could be calculated as follows.

\[
\dot{a}_c = a \cdot \left[ -\frac{1}{2} \dot{\varepsilon}_c(a) \right] = a \cdot \frac{K}{a^3} = \frac{K}{a^2}
\]

\[
\dot{a}_c = \frac{K}{a^2} = -\frac{S A'}{2} \frac{a b^3}{\left(b^\frac{3}{n} - a^\frac{3}{n}\right)^n} \left[ \frac{3}{2nG} (P_e - P_i) \right]^n
\]

Similarly,

\[
\dot{b}_c = -\frac{S A'}{2} \frac{a^3 b}{\left(b^\frac{3}{n} - a^\frac{3}{n}\right)^n} \left[ \frac{3}{2nG} (P_e - P_i) \right]^n
\]

The constant \(A'\) has been given in Derby and Wallach’s paper [32] by

\[
A' = \frac{AG b_v}{kT} D_{v0} \exp \left( \frac{-Q_v}{RT} \right)
\]

where \(A\) is the power law creep constant, \(b_v\) is the Burgers vector, \(D_{v0}\) is the volume diffusion coefficient, \(Q_v\) is the volume diffusion activation energy, \(k\) is Boltzmann’s constant, \(R\) is gas constant and \(T\) is temperature.

Equation (3.12) represents the evolution of the micro-void radius under the power law creep mechanism, which bears a different format with that in Derby and Wallach’s model. This is mainly due to the use of a different prototype in the present model: thick-walled sphere rather than cylinder.
3.4.2 Interface source diffusion

The end goal of establishing this interface source diffusion (ISD) model is to derive an expression describing the evolution of micro-void radius as a result of ISD, $\dot{a}_I$. Derby and Wallach have modified the approach of Johnson (used for sintering) [90] to model the diffusion bonding for a two-dimensional geometry under the interface source diffusion mechanism [31]. However, 3-D spherical micro-void was postulated in the present model (see Figure 3.5). Thus, the derivation of $\dot{a}_I$ was modified based on the current situation: due to the axial symmetrical void shape, the problem could be simplified to a 2-D case, i.e. only considering the circular cross-section. Since the cross-section is also axially symmetric, one quarter of it was considered in the following analysis, of which the area, $S_I$, is

$$S_I = \frac{1}{4} \pi a^2.$$  \hfill (3.14)

where $a$ is the micro-void radius.

Differentiating this area with respect to time,

$$\dot{S}_I = \frac{1}{2} \pi a \cdot \dot{a}_I$$  \hfill (3.15a)

thus

$$\dot{a}_I = \frac{2\dot{S}_I}{\pi a}.$$  \hfill (3.15b)

Hence once the rate of change of the cross-sectional area (denoted by $\dot{S}_I$) is determined, $\dot{a}_I$ can be determined. The following assumptions were made for the derivation of $\dot{S}_I$:

a) Diffusion occurs in a quasi-steady state, i.e. the rate of material transport is large compared to the rate of change of geometry [31].
b) Material transported by dislocation motion is negligible. This means that the divergence of the diffusion flux must be a constant everywhere along the interface at any instant [90].

c) Based on the arrangement of the micro-voids, the chemical potential gradient $\nabla \mu$ at the external rim of the surrounding region is zero (e.g. at $x = 0$ in Figure 3.5).

d) The chemical potential gradients for grain boundary diffusion and volume diffusion along the contact area are identical.

e) The interface can be considered as a normal high-angle grain boundary; thus, the chemical potential of the interface is proportional to the normal stress together with the effect of curvature beneath the neck surface [31].

![Figure 3.5 Schematic of micro-void and its surrounding region in the ISD model.](image)

Since this ISD model describes the materials transferring from the bonded interface to the micro-void, the reduction of the micro-void volume should be equal to the total volume flux arrived at the neck surface, $J$:

$$ S_I = -J $$

(3.16)

Since the simplified ISD model is under the same circumstances as the one of Derby and Wallach [31], their approach to derive the total volume flux was utilised directly as follows:
Based on Fick’s first law, the diffusion flux of atoms per unit area per unit time $j$ is defined as

$$ j = - \frac{D}{\Omega kT} \nabla \mu $$  \hspace{1cm} (3.17)

where $D$ is the diffusion coefficient, $\Omega$ is the atomic volume, $k$ is the Boltzmann’s constant, $T$ is the bonding temperature and $\mu$ is the chemical potential.

According to assumption (b) and Equation (3.17),

$$ \nabla j = - \frac{D}{\Omega kT} \nabla^2 \mu = constant $$  \hspace{1cm} (3.18a)

thus

$$ \nabla^2 \mu = C $$  \hspace{1cm} (3.18b)

where $C$ is a constant.

Equation (3.18b) could be solved using the following boundary conditions.

a) Based upon assumption (c),

$$ \nabla \mu = 0 \text{ at } x = 0 $$  \hspace{1cm} (3.19)

where $x$ is the distance measured from the external rim of the surrounding region, as indicated in Figure 3.5.

b) From the Gibbs-Thompson equation the chemical potential just beneath the neck (for the 2D case) [91] is

$$ \mu = \frac{\gamma \Omega}{r_n} $$  \hspace{1cm} (3.20)
where $\gamma$ is surface energy (surface tension) of the micro-void, $\Omega$ is atomic volume and $r_n$ is the bond neck radius. In the present model,

$$\mu = \frac{\gamma \Omega}{a} \text{ at } x = (b - a). \quad (3.21)$$

c) According to assumption (e), the total force acting normal to the interface is described by the chemical potential along it and so the two must balance, i.e.

$$\int_0^{(b-a)} \mu dx = (Pb - \gamma)\Omega \quad (3.22)$$

where $P$ is the bond pressure applied normal to the interface, which is treated to be the contact pressure between two powder particles in the present model.

The specific procedure of determining $\nabla \mu$ was detailed as follows.

Integrating Equation (3.18b),

$$\nabla \mu = Cx + C_1 \quad (3.23)$$

where $C_1$ is an integration constant.

Substituting boundary condition (a) into Equation (3.23), $C_1 = 0$ thus

$$\nabla \mu = Cx \quad (3.24)$$

Integrating Equation (3.24),

$$\mu = \frac{C}{2} x^2 + C_2 \quad (3.25)$$
where $C_2$ is an integration constant.

Substituting boundary condition (b) into Equation (3.25),

$$C_2 = \frac{\gamma \Omega}{a} - \frac{C}{2} (b - a)^2$$

(3.26)

Substituting Equation (3.25) into Equation (3.22),

$$\frac{C}{6} (b - a)^3 + C_2 (b - a) = (Pb - \gamma) \Omega$$

(3.27)

Combining Equation (3.26) and (3.27), the constant $C$ could be calculated.

$$C = \frac{-3\Omega}{(b-a)^2} \left( \frac{Pb - \gamma}{b-a} - \frac{\gamma}{a} \right)$$

(3.28)

Therefore, the gradient of chemical potential $\nabla \mu$ could be obtained and, based upon assumption (d), the flux of atoms by volume and grain boundary diffusion at $x = (b - a)$ were derived by combining Equation (3.17), (3.24) and (3.28) as the following two expressions respectively.

$$j_V = \frac{3D_V}{kT(b-a)} \left( \frac{Pb - \gamma}{b-a} - \frac{\gamma}{a} \right), \quad j_B = \frac{3D_B}{kT(b-a)} \left( \frac{Pb - \gamma}{b-a} - \frac{\gamma}{a} \right)$$

(3.29)

where $D_V$ and $D_B$ are diffusion coefficients for volume and grain boundary diffusion respectively.

Thus, the total volume flux arriving at the neck surface is found to be

$$J = \Omega (j_B A_B + j_V A_V)$$

(3.30)

where $A_B$ and $A_V$ are the limiting areas through which flux travels by grain boundary diffusion and volume diffusion respectively. For grain boundary diffusion, volume only
transfers along a small thickness of $\delta_B/2$ (where $\delta_B$ is the thickness of boundary diffusion layer), and for volume diffusion, it is assumed that the limiting area through which the flux occurs is equal to that of the neck [31]. Thus, Equation (3.30) becomes

\[ J = \frac{3\Omega}{kT(b - a)} \left( \frac{Pb - \gamma}{b - a} - \frac{\gamma}{a} \right) \left( \frac{\delta_B}{2} D_B + aD_V \right). \]  

(3.31)

Substituting Equation (3.31) into Equation (3.16) and (3.15b), the rate of change of the micro-void radius $\dot{a}_I$ was given by

\[ \dot{a}_I = \frac{-6\Omega}{\pi akT(b - a)} \left( \frac{Pb - \gamma}{b - a} - \frac{\gamma}{a} \right) \left( \frac{\delta_B}{2} D_B + aD_V \right) \]  

(3.32)

Equation (3.32) represents the evolution of the micro-void radius under the interface source diffusion mechanism. The equation bears a similar format as that in Derby and Wallach’s model since the volume flux of the cross-section for the two models are the same. However, the present model considers a spherical void shape (rather than cylindrical), which leads to a different derivation and format of the rate equation as shown in Equation (3.32).

### 3.4.3 Combined effects

Combined effects of power law creep and interface source diffusion on the shrinkage of micro-voids are described by Equation (3.33), which calculates the total rate of change in micro-void radius, $\dot{\bar{a}}$, as the arithmetic sum of $\dot{a}_C$ (rate of change in micro-void radius under powder law creep mechanism) and $\dot{a}_I$ (rate of change in micro-void radius under interface source diffusion mechanism).

\[ \dot{\bar{a}} = \dot{a}_C + \dot{a}_I. \]  

(3.33)

Equation (3.33) is the final form of the mathematical model governing the bonding process in powder forging.
3.5 Implementation of the theoretical bonding model for nickel-based superalloy FGH96 powders

To implement the theoretical bonding model under a given temperature and pressure, two main inputs are required for a specific material: (1) initial size of the micro-voids and (2) creep and diffusion data. In this section, a specific procedure of operating the present model to predict bonding is illustrated for nickel-based superalloy FGH96 powders. Also, parametric studies in terms of the two inputs were performed.

3.5.1 SEM observation on the surface of FGH96 powders

As an important input of the present model, the initial size of the micro-void is mainly determined by the surface morphology of the powders, which is analogous to the concept of surface roughness in previous diffusion bonding models. Argon atomised FGH96 powders, supplied by Beijing Institute of Aeronautical Materials (BIAM), were observed using the scanning electron microscope (SEM). A double-sided carbon tape was attached to an aluminium substrate, then the substrate was dipped in powders and fitted in the SEM as shown in Figure 3.6(a). The observation result is presented in Figure 3.6(b) and eight typical powder particles with different diameters were picked and closer observations were taken on their local surfaces (see Figure 3.7).

Figure 3.6 (a) SEM test set-up; (b) Scanning electron micrograph of FGH96 powders.
Figure 3.7 Scanning electron micrographs of FGH96 powder particles: (a) – (h) present different particles.
In order to determine the initial micro-void size of FGH96 powders, an analysis was performed on the local surface of the powder particles. The local surface shown in Figure 3.7(b) was used to demonstrate the specific analysing procedure (see Figure 3.8). The observed pits are indicated using closed curves (in red), which represent the micro-voids formed by these pits. It was ensured that every pit is fully enclosed by the corresponding closed shape, i.e. the size of the closed shape is larger than the pit. The excess is regarded as a compensation for the adjacent channels observed, which are not directly included in the model.

![Image](image.png)

Figure 3.8 Analysis of pits observed on the local surface of FGH96 powder.

The areas of all the closed shapes, the distances between them and the diameters of the powder particles were measured using the software ImageJ. Since the micro-voids were assumed to be spherical in the present model, an equivalent circle (with the same area) was presumed for each closed shape, thus the radius of the equivalent circle (i.e. micro-void radius) could be obtained. Since the influence of the largest pit on the bonding time is predominant at given pressures and temperatures, the maximum micro-void radius was employed to investigate the problem. Figure 3.9 shows the variation of the maximum micro-void radius with respect to the diameter of the powder particle. It can be found that the micro-void radius increases with the diameter of the powder particle, and a linear relationship could be roughly captured (dotted line).
As mentioned in Section 1.1.2, the average size of the FGH96 powders is 35 µm in diameter (indicated using the red dashed line in Figure 3.9). Thus, from the linear relationship obtained, the radius of maximum micro-void, i.e. the initial size of the micro-void used for FGH96 powder in the present bonding model, could be roughly identified, which is 0.55 µm.

![Figure 3.9 Relationship between the diameter of FGH96 powder and the radius of corresponding maximum micro-void.](image)

**3.5.2 Implementation of the model**

Another input of the bonding model is the creep and diffusion data. Since the information for FGH96 is not available for the moment, the material data for another nickel alloy, MAR-M200 (see Table 3.1), was utilised as a substitution, which is found to be the most similar one available in the literature [92]. The chemical composition of these two materials are listed in Table 3.2. The influences of using material data for different nickel alloys in the present bonding model were investigated through a parametric study, which is detailed in Section 3.5.3. The modelling work was undertaken using an iterative computing program in MATLAB (see Appendix 1), which produced the following outputs.
Table 3.1 Material parameters for MAR-M200 [92].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic volume</td>
<td>( \Omega )</td>
<td>( 1.1 \times 10^{-29} m^3 )</td>
</tr>
<tr>
<td>Burgers vector</td>
<td>( b_v )</td>
<td>( 2.5 \times 10^{-10} m )</td>
</tr>
<tr>
<td>Melting temperature</td>
<td>( T_M )</td>
<td>( 1600 K )</td>
</tr>
<tr>
<td>Shear modulus at 300K</td>
<td>( \mu_0 )</td>
<td>( 8.0 \times 10^4 MN/m^2 )</td>
</tr>
<tr>
<td>Temperature dependence of shear modulus</td>
<td>( \tau_T )</td>
<td>(-0.5)</td>
</tr>
<tr>
<td>Power-law creep exponent</td>
<td>( n )</td>
<td>7.7</td>
</tr>
<tr>
<td>Power-law creep pre-exponential coefficient</td>
<td>( A'' )</td>
<td>( 5.3 \times 10^{34} s^{-1} )</td>
</tr>
<tr>
<td>Power-law creep activation energy</td>
<td>( Q_{cr} )</td>
<td>( 556 kJ/mole )</td>
</tr>
<tr>
<td>Volume diffusion pre-exponential coefficient</td>
<td>( D_{0v} )</td>
<td>( 1.6 \times 10^{-4} m^2/s )</td>
</tr>
<tr>
<td>Volume diffusion activation energy</td>
<td>( Q_v )</td>
<td>( 285 kJ/mol )</td>
</tr>
<tr>
<td>Boundary diffusion pre-exponential coefficient</td>
<td>( D_{0b} )</td>
<td>( 2.8 \times 10^{-5} m^2/s )</td>
</tr>
<tr>
<td>Boundary layer thickness</td>
<td>( \delta_B )</td>
<td>( 10^{-10} m )</td>
</tr>
<tr>
<td>Boundary activation energy</td>
<td>( Q_B )</td>
<td>( 115 kJ/mol )</td>
</tr>
<tr>
<td>Surface energy</td>
<td>( \gamma )</td>
<td>( 1.71 J/m^2 )</td>
</tr>
</tbody>
</table>

Table 3.2 Chemical composition of (1) FGH96 [11] and (2) MAR-M200 [92] (wt%).

<table>
<thead>
<tr>
<th>No.</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Ti</th>
<th>Al</th>
<th>Nb</th>
<th>Zr</th>
<th>C</th>
<th>B</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>16</td>
<td>13</td>
<td>4</td>
<td>4</td>
<td>3.7</td>
<td>2.2</td>
<td>0.8</td>
<td>0.036</td>
<td>0.03</td>
<td>0.011</td>
<td>Bal</td>
</tr>
<tr>
<td>(2)</td>
<td>9</td>
<td>10</td>
<td>-</td>
<td>12.5</td>
<td>2</td>
<td>5</td>
<td>1</td>
<td>0.05</td>
<td>0.15</td>
<td>0.015</td>
<td>Bal</td>
</tr>
</tbody>
</table>

1) **Prediction of the extent of bonding**

The fundamental function of the present bonding model is to predict the extent of bonding for a certain contact area (where all micro-voids involved begin to shrink at the same time) at a given pressure and temperature, which is represented by the fractional volume bonded of a micro-void (i.e. the percentage of reduction of the micro-void volume). The micro-void volume decreases with time and eventually reaches ‘zero’ (a sufficiently small scale
which will not affect the mechanical property of the material), at which a contact area between neighbouring powders are considered to be fully bonded. The time for achieving a fully bonded state is termed the bonding time (as defined in Section 1.1.3). The model works for different magnitudes of contact pressure and temperature. Figure 3.10 serves to demonstrate the capability of the model at 1150 °C and 200 MPa.

![Graph showing bonding time vs. contact pressure](image)

**Figure 3.10** Prediction of the extent of bonding for a contact area (powder material: FGH96, temperature 1150 °C, contact pressure 200 MPa).

2) **Effects of contact pressure on bonding time**

The effects of varying contact pressures on bonding time can be evaluated from the present model. Figure 3.11 shows the variation of bonding time with contact pressure at three different temperatures. All the three curves indicate that an increase in contact pressure leads to a reduction in bonding time, which is reasonable from a practical viewpoint. The results obtained can be regarded as a criterion to judge whether a contact surface, at a given temperature, has been fully bonded under certain contact pressure and load-holding time, which shows the potential of the present model in predicting the bonding status for a particular area of a powder-forged component.
Figure 3.11 Variation of bonding time with contact pressure at different temperatures (1050, 1100, 1150 °C).

3) Effects of temperature on bonding time

The effect of temperature on bonding time is shown in Figure 3.12, where three different pressures are applied to plot the relationship between bonding time and temperature. Consistent with Figure 3.11, the bonding time decreases with contact pressure for the full range of temperatures from 1000 to 1200 °C. When contact pressure is fixed, it is noted that the bonding time is less for higher temperatures. Therefore, it can be concluded that an increase in temperature results in a reduction in bonding time. Although the processing temperature, in practice, is mainly dependent on the melting temperature, the curves obtained below can be used in determining the optimum temperature for a certain pressure within a relatively small range of temperatures.
4) **Power law creep vs. Interface source diffusion**

Power law creep and interface source diffusion mechanisms cooperate during the bonding process as reflected from Equation (33). The contribution of each mechanism is evaluated under a temperature of 1150 °C and different contact pressures (shown in Figure 3.13). The green and blue lines indicate the bonding process governed only by power law creep and interface source diffusion respectively; while the red line refers to the total effect. Through comparison, it implies that interface source diffusion dominates bonding at relatively low contact pressure and its contribution is comparable with power law creep at high contact pressures. The dramatic enhancement of power law creep at high contact pressure is due to the fact that power law creep strongly depends on pressure, which is demonstrated by Equation (3.12).
Figure 3.13 Comparison of the contribution of two operating mechanisms under different contact pressures at 1150 °C.
3.5.3 Parametric study

In order to test the robustness of the results of the bonding model, and to increase the understanding of the impact of inputs on the outputs, sensitivity studies in terms of the two inputs of present model were performed as follows.

1) Creep and diffusion material parameters

Since the material data of MAR-M200 was utilised as a substitution for FGH96, it is of great significance to identify the influence of changing the values of material parameters on the bonding results. Each of the material parameters listed in Table 3.2 was investigated individually. The model was originally operated using the data of MAR-M200 then the value of a designated parameter was replaced with the data of other four typical nickel alloys: (1) Ni-10 at. % Cr, (2) Ni-20 at. % Cr, (3) Ni-13.5 wt. % Cr-1 vol. % ThO$_2$ and (4) Ni-22.6 wt. % Cr-1 vol. % ThO$_2$ (see Appendix 2) [92], and the bonding times obtained under each value were compared. Thus, the influence of this parameter on the modelling results could be recognised. Through comparison, it was found that all the parameters, except for those related to power-law creep (i.e. power-law creep exponent and constant), brought little differences on the bonding time at a certain temperature and pressure, which indicates that the power-law creep exponent and constant might well be the dominant parameters that distinguish different nickel alloys in terms of bonding between powders.

Figure 3.14 shows the bonding results in terms of different values for power-law creep exponent and constant (while all other material parameters were kept the same for the five materials). It can be found that the influence of using different values for these two parameters is negligible when the contact pressure is relatively low (e.g. 1000 MPa); when the contact pressure increases, an evident difference for the bonding time is noticed. This is consistent with the earlier finding that power-law creep mechanism has a strong impact on the bonding process at a higher contact pressure as shown in Figure 3.13.

Based on the study, it could be speculated that the use of data for MAR-M200 instead of FGH96 would make little difference at relatively low pressures (e.g. 1000 MPa). However, for higher pressures (e.g. 2000 MPa), the percentage difference could be around 67%. Thus, more experimental calibrations in terms of the material parameters would be required if MAR-M200 data was to use for the present model at high pressures.
Figure 3.14 Bonding results in terms of different values for power-law creep related parameters at 1150 °C and different pressures: (1) Ni-10 at. % Cr, (2) Ni-20 at. % Cr, (3) Ni-13.5 wt. % Cr-1 vol. % ThO₂, (4) Ni-22.6 wt. % Cr-1 vol. % ThO₂, (5) MAR-M200.

2) *Initial size of micro-voids*

As another important input of the model, it is also worthwhile to explore how the initial size of the micro-voids would influence the bonding results. Figure 3.15 shows the relationship between bonding time and initial micro-void radius at 1150 °C and three different contact pressures. It can be observed that the three lines share a common trend: with the increase of micro-void radius, the model tends to become more sensitive, i.e. bonding time rises more rapidly, which indicated that errors for relatively small micro-void (e.g. 0.1 or 0.2 µm in radius) would have little impact on the required bonding time; however, for larger micro-void, this influence would increase. It is also noticed that this trend becomes more obvious in terms of lower contact pressure (e.g. 100 MPa), which implies the determination of the initial micro-void radius would be so critical that any small variations (e.g. 0.1 µm) would lead to a great change of the bonding results. Thus, an experimental calibration with regard to this parameter would be necessary, especially when the model is to be applied at lower pressures (e.g. 100 MPa).
3.6 Verification of the theoretical bonding model

According to the literature review, most of the diffusion bonding models are used to investigate the bonding between two prepared surfaces, which considers the void shrinkage in a plane strain state while proposing different assumptions on the morphology of surface roughness, i.e. shape of the void cross section. Thus, the problem has always been solved in a two-dimensional (2D) state. However, the present work is developed for bonding between powder particles. It is established in a three-dimensional (3D) state and a spherical void is assumed on the contact surface. To validate the rationality of the present model, an approach was proposed to compare the bonding results between the present model and the previous diffusion bonding models. Guo and Ridley’s model, which has claimed an improved agreement with the experimental data for copper compared to Derby and Wallach’s model [34], was adopted to compare with the present model.

First, an equivalent initial condition was set for the two cases. Area fraction has always been adopted to evaluate the extent of bonding, however, this quantity should change to volume fraction in the 3D state. Thus, an equal initial void volume fraction was
established, i.e. the total volumes of the voids within a representative space (indicated in Figure 3.16) were equal. A lenticular void shape is assumed in Guo and Ridley’s work [34] and the volume of an individual void can be calculated once the parameters shown in Figure 3.17(a) are given. In the present model (see Figure 3.16(b)), the size and number of the spherical voids within the representative space were to be determined to obtain an equal volume fraction.

![Figure 3.16 Schematics of the initial void morphology.](image)

![Figure 3.17 Schematics of individual void.](image)

The voids in the present model follow the close-packing arrangement as shown in Figure 3.18 (vertical view). Each of the small circles (in red) refers to a void and its surrounding region. A representative element was identified (blue rectangle with centered line), of which the width is the representative length L as indicated in Figure 3.17(a). Each element contains six spherical voids and its dimension is $6b \times 2\sqrt{3}b$ where $b$ is the radius of the adjacent region of a void as shown in Figure 3.17(b). The number and size of the voids in the present model could thus be determined based on the following two conditions:
a) The total volume of the micro-voids should be equal to that of the lenticular void in a representative space as indicated in Figure 3.16.

b) To ensure multiple representative elements can be placed in one representative space, the total horizontal length of multiple representative elements in the present model should be equal to the width of the representative space, i.e. the initial width of the lenticular void.

![Figure 3.18](image)

Figure 3.18 The arrangement of voids and their surrounding region in the present model.

In Guo’s model, the two contact surfaces have been assumed to be in a ridge-to-ridge and valley-to-valley manner, and an instantaneous plastic deformation occurs once a pressure is applied as shown in Figure 3.19. The contact length of the void ridge, i.e. $B$, could be estimated as [34]

$$
B = \frac{\sqrt{3}(d/2)}{2 + \pi - 2 \tan^{-1}(2h/d)} \left| \frac{\sigma_p}{\sigma_y} \right|
$$

(3.34)

where $h$ and $d$ are parameters shown in Figure 3.17(a). $\sigma_p$ is the applied stress and $\sigma_y$ is the yield stress.

Thus, after deformation, the void width and height are, respectively,

$$
d' = d - 2B
$$

(3.35)
and

\[
h' = \frac{h[h^2 + (d/2 - B)^2 + Bd/2 - (2Bh^2/d)]}{(d/2)^2 + h^2}
\]  
(3.36)

Figure 3.19 Void shape after instantaneous plastic deformation in Guo’s model.

The area of the lenticular cross section can be calculated as

\[
A_l = 2\left(\sin^{-1}\frac{4d'h'}{d'} + 4h'^2\right)\left(\frac{d'^2}{8h'} + \frac{h'}{2}\right)^2 - d\left(\frac{d'^2}{8h'} - \frac{h'}{2}\right)
\]  
(3.37)

and the volume of a void in Guo’s model is

\[
V_G = A_l \times L
\]  
(3.38)

where \(L = 2\sqrt{3}b\) is the representative length.

According to the two conditions mentioned above, the following equations should be satisfied.

\[
N \times 6 \times \frac{4}{3} \pi a^3 = V_G
\]  
(3.39)

\[
N \times 6b = d
\]  
(3.40)
where $N$ is an integer referring to the number of representative elements in the present model, and $\alpha$ is the radius of the spherical void.

Assuming $b = ka$ ($k$ is a constant),

$$N = \frac{\pi d^2}{9\sqrt{3}k^3A_l}$$  \hspace{1cm} (3.41)

Rounding $N$ upwards to the nearest integer, from which the radius of void could be derived as

$$\alpha = \frac{d}{6kN}$$ \hspace{1cm} (3.42)

Once $\alpha$ is determined, with the required material parameters, the evolution of fractional volume bonded within a representative space could be obtained for the present model. Figure 3.20 shows the comparison of the bonding results, i.e. predictions of fractional volume bonded, obtained using the present model and previous diffusion bonding models, also involving the experimental data provided by Derby and Wallach [48]. The material utilised is copper, and relevant parameters are listed in Table 3.3.
Figure 3.20 Comparison of bonding results of present model with previous models and experimental results of Derby and Wallach; Temperature: 550 °C, Pressure: 35 MPa, Surface Roughness Height: 3 µm, Wavelength: 40 µm.

Table 3.3 Material Parameters for Copper [6].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic volume</td>
<td>( \Omega )</td>
<td>1.18\times 10^{-29} m^3</td>
</tr>
<tr>
<td>Burgers vector</td>
<td>( b_v )</td>
<td>2.56\times 10^{-10} m</td>
</tr>
<tr>
<td>Melting temperature</td>
<td>( T_M )</td>
<td>1356 K</td>
</tr>
<tr>
<td>Shear modulus at 300K</td>
<td>( \mu_0 )</td>
<td>4.29\times 10^4 MN/m^2</td>
</tr>
<tr>
<td>Temperature dependence of shear modulus</td>
<td>( \tau_T )</td>
<td>-0.54</td>
</tr>
<tr>
<td>Normalized yield stress</td>
<td>( \sigma_y/\mu )</td>
<td>5\times 10^{-3}</td>
</tr>
<tr>
<td>Power-law creep exponent</td>
<td>( n )</td>
<td>4.8</td>
</tr>
<tr>
<td>Power-law creep constant</td>
<td>( A )</td>
<td>7.42\times 10^5 s^{-1}</td>
</tr>
<tr>
<td>Volume diffusion pre-exponential coefficient</td>
<td>( D_{0v} )</td>
<td>6.2\times 10^{-5} m^2/s</td>
</tr>
<tr>
<td>Volume diffusion activation energy</td>
<td>( Q_v )</td>
<td>207 kJ/mol</td>
</tr>
<tr>
<td>Boundary diffusion pre-exponential coefficient</td>
<td>( D_{0b} )</td>
<td>5.12\times 10^{-5} m^2/s</td>
</tr>
<tr>
<td>Boundary layer thickness</td>
<td>( \delta_B )</td>
<td>10^{-10} m</td>
</tr>
<tr>
<td>Boundary activation energy</td>
<td>( Q_B )</td>
<td>105 kJ/mol</td>
</tr>
<tr>
<td>Surface energy</td>
<td>( \gamma )</td>
<td>1.75 J/m^2</td>
</tr>
</tbody>
</table>

It can be observed that the present model (solid line) shares a similar trend with the two previous models, i.e. Derby and Wallach (dashed line) and Guo and Ridley (dotted line). Also, the prediction of present model is in a reasonable range of the experimental results, through which the rationality of present model could be validated. However, among the three predictions, a gap during the initial stage can still be found. This is due to the initial void shape assumed - the curvature radius of the neck has an influence on the bonding rate. In the previous models, lenticular or similar void shape has a larger curvature radius (compared to spherical) at the void neck, which leads to a faster bonding rate in the initial stage as shown in Figure 3.20. Due to the essence that spherical shape leads to a faster volume reduction rate than long strip shape (e.g. cylinder), the fractional volume bonded in the present model approaches the other two models in the later stage. Based on the
above analysis, a long strip void shape with lenticular or elliptic cross-section would be more realistic for the initial stage. However, the present model was built to model the bonding between powder particles, and the spherical void shape was proposed for the surface condition that is analogous to FGH96 powder. A further experimental verification of the applicability of present model in powder forging will be introduced in the next chapter.

3.7 Summary

In this chapter, a theoretical (analytical) model for bonding between powders was established based on the methodology by Derby and Wallach which was originally developed for diffusion bonding (DB) between two prepared surfaces. In the present work, the bonding process was regarded as the shrinkage of micro-voids formed by the indents observed on the powder surface. Considering the surface morphology of argon atomised FGH96 powders, discrete spherical micro-voids were assumed to be located on the contact interface, thus the theoretical bonding model was formulated in a three-dimensional state, which is the main difference from the existing DB models (built in a plane strain, i.e. two-dimensional, state). It was suggested that the established bonding model is applicable for powders with a similar surface morphology as FGH96 where the assumptions for the micro-voids hold; while for those which are different, the analytical model can be modified according to a different micro-void shape and arrangement using the same modelling methodology.

Two main inputs are required to implement the proposed bonding model on a specific powder material, which are (1) initial size of the micro-void and (2) creep and diffusion material data. The FGH96 powders were applied to demonstrate the use of the present bonding model. Based on the numerical results, it was indicated that higher contact pressure and temperature both result in a shorter bonding time; and the contribution of the power law creep mechanism on bonding increases with the contact pressure. Also, parametric studies were conducted on the two main inputs, which suggested that the use of alternative material data for FGH96 has little impact on the bonding results under relatively low contact pressures; and the bonding results become more sensitive to the larger size of the micro-void. Finally, the present model was compared with an existing diffusion bonding model by Guo and Ridley and a good agreement was found in terms of
the modelling results. However, the applicability of the proposed bonding model in powder forging will be demonstrated through experiments, which is detailed in the next chapter.
Chapter 4  Investigation of Bonding Conditions During Direct Powder Forging for a Nickel-Based Superalloy

4.1 Introduction

Direct powder forging (DPF), as mentioned in Chapter 1, is a process during which powders are heated up to a forming temperature in vacuum and forged directly to the final shape, by using a high forming load for a very short time [11]. Figure 4.1 shows the schematic of a typical DPF process. The temperature-holding time is set to ensure the powders reach the target temperature uniformly. The compression time refers to the process when the powder assemblage is compressed to full density. At a given temperature and load, a critical holding time is termed the bonding time, which is necessary to guarantee the powders are bonded together after compression.

Figure 4.1 Schematic of a typical DPF process.
Since temperature, applied load and holding time are three main powder forging process variables which have a dominant influence on the bonding between powder particles, it is important to explore the relationship among these parameters; thus, critical conditions for bonding could be identified.

In this chapter, DPF tests were carried out on argon atomised nickel-based superalloy FGH96 powders using a dynamic testing machine, Gleeble 3800, under different forming conditions. For each condition, components were produced with different holding times. These components were then manufactured to a dumbbell shape, and investigated through a miniature tensile test. As a straight-forward parameter for evaluating bonding, tensile strengths of these components were compared and a critical holding time, i.e. bonding time, was identified in terms of a given temperature and load. At the end of this chapter, the experimental results were compared with the numerical predictions, through which the applicability of the proposed bonding model was validated.

4.2 Direct powder forging Gleeble test

4.2.1 Experimental apparatus

1) Gleeble 3800

A Gleeble is a dynamic testing machine that can simulate a wide variety of thermal-mechanical metallurgical situations. Gleeble 3800 is one of the available models, of which the main operating units are shown in Figure 4.2 including the digital control console, load unit, vacuum tank and mobile conversion unit. Other units such as vacuum system, hydraulic pump and water chiller are not presented in the figure. The Gleeble 3800 system is capable of exerting as much as 20 tons of static force in compression and as fast as 2000 mm/s in stroke displacement rate. It adopts a direct resistance heating system with closed-loop control. The maximum heating rate for a 10 mm diameter × 12 mm high compression specimen can be 50 °C/s; and a steady state temperature can be hold for all specimen sizes [93]. Thus, this system is well-suited for a powder forging test.

All the Gleeble tests are completed in the vacuum tank where a pressure of 0.3 mmHg can be reached through a rough vacuum pumping system [93]. The set-up for a uniaxial compression test within the tank is shown in Figure 4.3. A couple of Tungsten carbide
anvil assemblies (including ISO-T anvils and anvil bases) are fit into the jaws, and heavy duty U-jacks are utilized to guarantee an intimate contact between the anvil base and jaw as indicated in the figure, thus the current and load can be passed to the specimen smoothly. The right jaw is fixed while the left one is connected to the Gleeble load unit which allows it to move horizontally. The stroke of the left jaw is recorded automatically during a test, and the maximum stroke of Gleeble 3800 is 125 mm [93]. The specimen is placed in between the two anvils and always aligned with the central axis. The temperature can be measured through connecting thermocouple wires to the push posts as shown in Figure 4.4.

Figure 4.2 Main units of Gleeble 3800.

Figure 4.3 Set-up for a uniaxial compression test in Gleeble 3800.
2) Graphite tool

In the present work, a graphite tool set was used to conduct the powder forging test (see Figure 4.5). This tool set consists of two punches and a die, and it has been proved to be effective in forming micro-scale components with high relative density [94]. In terms of a miniature component, it is easier to maintain a uniform temperature during the forging process compared to a large-scale one. The tool material was selected as graphite due to its excellent conductivity and good workability at high temperature (up to 2500 °C). Also, given the lubricating property of graphite, it could prevent the forged component being stuck during the ejection process [95]. However, low mechanical strength is a major issue for graphite material, and a high load cannot be applied considering its mechanical strength [96].
As shown in Figure 4.5, a blind hole is set in the middle section of the graphite die to measure the temperature during the forging test. Thermocouple wires were fed through a two-hole ceramic insulating tube separately and twisted together at the end as shown in Figure 4.6(a). The tube was then put in the blind hole of the graphite die, while the other ends of the thermocouple wires were connected to the push posts correspondingly. The experimental set-up for a powder forging test using the graphite tool set is illustrated in Figure 4.6(b).

Figure 4.6 (a) Ceramic insulating tube with thermocouple wires; (b) Experimental set-up for the powder forging test.

In order to verify that the temperature measured from the blind hole (using thermocouple TC2) can accurately reflect the temperature of powders during the forging process, a proving test was designed, in which the graphite tool was modified: a through hole was drilled next to the blind hole within the same cross-section as shown in Figure 4.7. During the test, another thermocouple (TC1) was placed in the through hole aiming to measure the temperature of powders directly. Thus, the two temperatures can be compared.

Figure 4.7 Section view of the modified graphite tool and thermocouples.
The FGH96 powders were heated to 620 °C using two heating rates, 10 °C/s and 20 °C/s, then held for 60 seconds. The test results are presented in Figure 4.8 and 4.9. It should be noted that there is fluctuation during the late stage of the test, which is considered to be the results of reprocessing the tool (the through hole was manufactured using a hand drill). However, as the objective of this test is to compare the temperatures measured from TC1 and TC2, the reason for the fluctuation was not further discussed. In terms of the heating rate of 10 °C/s, the results measured from TC2 and TC1 were very close, while obvious differences were observed for 20 °C/s. This indicates that the temperature measured from the blind hole is reliable for a low heating rate (e.g. 10 °C/s or below).

Figure 4.8 Results of the proving test for the heating rate of 10 °C/s.

Figure 4.9 Results of the proving test for the heating rate of 20 °C/s.
4.2.2 Experimental procedures

Nickel-based superalloy FGH96 powders were used as raw materials, of which the theoretical density ($\rho$) is 8.33 g/cm$^3$ [97]. In terms of an assembled graphite tool set, the minimum distance between two punches is 4 mm. Thus, in the present work, the length of the component was designed as 5 mm to ensure the applied load can be totally delivered to the powders during compression. Since the inner diameter of the die is 4 mm, the volume of the forged component (V) can be calculated thus the weight of powders required for a cylindrical component (m) can be derived as Equation (4.1).

$$m = \rho \cdot V \quad (4.1)$$

The powders were weighed using an electronic scale according to the calculated value, and filled into the die. The filled tool set was then compressed using the Gleeble 3800 as shown in Figure 4.10. The experiments were conducted using a force-control method.

Conforming to the industrial experience (from BIAM), 1150°C was selected as the forging temperature. Three different loads were employed, i.e. 1.3, 2 and 2.5 kN, considering the mechanical strength of the graphite tool, and the corresponding pressures applied on the powders were calculated based on the diameter of the punch (4 mm) as 103.5, 159.2 and 198.9 MPa respectively. The specific heating and loading process are designed as shown in Figure 4.11. In terms of a specified load, powders were forged with different loading times. The specific process parameters are listed in Table 4.1. The heating time for all the tests were identical (155 s), which aims to minimize the difference between every single test during the heating process, thus the only changing variable for the forging tests under a designed load is the loading time.

![Figure 4.10 Direct powder forging Gleeble test.](image)
Table 4.1 Process parameters for the DPF Gleeble tests.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Load (kN)</th>
<th>Loading Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1150</td>
<td>1.3 (103.5MPa)</td>
<td>51, 71, 91, 106, 121, 141, 161, 191, 251</td>
</tr>
<tr>
<td></td>
<td>2 (159.2MPa)</td>
<td>161, 191</td>
</tr>
<tr>
<td></td>
<td>2.5 (198.9MPa)</td>
<td>11, 21, 31, 41, 56, 71, 81, 91, 106, 121</td>
</tr>
</tbody>
</table>

4.2.3 Results and discussion

During the loading process, the heated powder assemblage was compressed to a final position and then held for a certain period as shown in Figure 4.12 (ignoring thermal expansion effects and the deformation of graphite tool). Accordingly, the compression time in terms of a given temperature and load can be deduced based on the stroke curve obtained during the forging test.

The stroke displacement history of all the tests under 2.5, 2 and 1.3 kN are shown in Figure 4.13, 4.14 and 4.15 respectively. As can be seen from the stroke curves, all the
components were compressed to a final position after which the stroke almost remained constant (ignoring the influence of the deformation of graphite tool). Thus, the compression time for each condition could be approximately identified (as indicated using the red dashed line), after which all the forged components were supposed to have a full density assuming the applied load was sufficiently high.

Figure 4.12 Schematic of compression and holding process.

Figure 4.13 Evolution of stroke for all the tests under 2.5 kN (198.9 MPa).
The density of each forged component was measured using the Archimedes method [47]. (The temperature of the water used for the measurement was 24 °C, and the density was 0.9973 g/cm³.) The measurement results of relative density ($\rho_r$) are presented in Table 4.2.
As shown in the table above, the relative densities of all the forged components were above 97% of the theoretical density (8.33 g/cm³) and some of them were 100%, which indicates that the temperature and loads applied in the present work is sufficient for yielding components with full density. It was also found that, for some components with shorter loading time (e.g. 2.5-1, 11 s), the relative density could reach 1 while some of those with longer loading time (e.g. 2.5-9, 106 s) only achieved 0.97. This phenomenon implies that the loading time is not the main reason for the difference of relative densities between forged components in the present work. However, following issues may impede the attainment of a full density: (1) When filling the powders, the condition was not absolutely airtight, which might lead to an impurity of the powders. (2) Although the quantity of powders for every test was designed to be identical, the geometric error and dimension error of the graphite tool during manufacturing could cause the difference in initial length of powder assemblage, which could have an impact on the densification process. (3) The filled tool set was shaken by hand before forging to reduce the porosity of initial powder packing, while the initial particle arrangement also has an influence on the powder compaction and sintering process [80,98,99].

Table 4.2 Measurement results of relative density.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Loading time (s)</th>
<th>( \rho_r )</th>
<th>Test No.</th>
<th>Loading time (s)</th>
<th>( \rho_r )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5-1</td>
<td>11</td>
<td>1.00</td>
<td>2.5-2</td>
<td>21</td>
<td>0.99</td>
</tr>
<tr>
<td>2.5-3</td>
<td>31</td>
<td>0.98</td>
<td>2.5-4</td>
<td>41</td>
<td>0.99</td>
</tr>
<tr>
<td>2.5-5</td>
<td>56</td>
<td>0.97</td>
<td>2.5-6</td>
<td>71</td>
<td>1.00</td>
</tr>
<tr>
<td>2.5-7</td>
<td>81</td>
<td>0.99</td>
<td>2.5-8</td>
<td>91</td>
<td>1.00</td>
</tr>
<tr>
<td>2.5-9</td>
<td>106</td>
<td>0.97</td>
<td>2.5-10</td>
<td>121</td>
<td>1.00</td>
</tr>
<tr>
<td>2-1</td>
<td>161</td>
<td>1.00</td>
<td>2.5-10</td>
<td>121</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As shown in the table above, the relative densities of all the forged components were above 97% of the theoretical density (8.33 g/cm³) and some of them were 100%, which indicates that the temperature and loads applied in the present work is sufficient for yielding components with full density. It was also found that, for some components with shorter loading time (e.g. 2.5-1, 11 s), the relative density could reach 1 while some of those with longer loading time (e.g. 2.5-9, 106 s) only achieved 0.97. This phenomenon implies that the loading time is not the main reason for the difference of relative densities between forged components in the present work. However, following issues may impede the attainment of a full density: (1) When filling the powders, the condition was not absolutely airtight, which might lead to an impurity of the powders. (2) Although the quantity of powders for every test was designed to be identical, the geometric error and dimension error of the graphite tool during manufacturing could cause the difference in initial length of powder assemblage, which could have an impact on the densification process. (3) The filled tool set was shaken by hand before forging to reduce the porosity of initial powder packing, while the initial particle arrangement also has an influence on the powder compaction and sintering process [80,98,99].
Based on the stroke curves and the measured relative densities, the compression times, during which a full-density component can be obtained, were identified in terms of different loads at 1150 °C. Figure 4.16 shows the relationship between applied pressure and compression time. However, this result is only valid for the situation of hot repressing, in which material flow during densification is mainly in the direction of compression [2].

Once the compression time under each condition is obtained, the holding time for every single test can be calculated using Equation (4.2).

\[
\text{Holding time} = \text{Loading time} - \text{Compression time} \quad \text{(4.2)}
\]

Thus, the influence of holding time on material strength, i.e. bonding between powders, can be investigated through the miniature tensile test which will be detailed in the next section.

![Figure 4.16 Relationship between applied pressure and compression time for FGH96 powder in the situation of hot-repressing.](image)

4.3 Tensile test on DPFed miniature components

As a straight-forward method to evaluate bonding between powders, tensile tests were conducted on the two groups of forged miniature components (one group is forged under
the condition of 2.5 kN, 1150 °C and the other is 1.3 kN, 1150 °C as listed in Table 4.1) to obtain their ultimate tensile strengths. Thus, a critical holding time for each group could be derived through comparison (the experimental results were compared only within the group). The specific experimental design was detailed as follows.

4.3.1 Specimen geometry and grip design

The cylindrical component, which was direct-forged using the graphite tool, is shown in Figure 4.17(a) (4mm in diameter and 5 mm in length). Considering the length of the component, it is impossible to machine it to a standard tensile specimen [100] based on the accessible processing conditions (the stiffness of the material and the way of clamping during machining are the main issues). Also, since the tensile testing in the present work is not for obtaining accurate material parameters nor comparing with standard values, a non-standard tensile specimen was designed as shown in Figure 4.17(b). A butt end is a common style for the shoulder of a tensile specimen, which is easy to manufacture. The diameter of the gauge section was designed as 1.5 mm considering the stiffness of the component (a thinner diameter will lead to a deflection of the gauge section). The tensile specimen, shown in Figure 4.17(c), was manufactured using a mini lathe.

Figure 4.17 (a) Direct-forged cylindrical component; (b) Design of the tensile specimen; (c) Machined tensile specimen.
A threaded grip set (see Figure 4.18(a)) was also designed to be fitted in an Instron tensile testing machine. The material was selected as an ultrahigh-strength steel, AerMet100 [101]. The notch on one end of the grip was designed to ensure a good alignment when the test specimen is fitted in the grip as shown in Figure 4.18(b).

![Figure 4.18 (a) The designed grip set; (b) Assembly of the tensile specimen and grips.](image)

### 4.3.2 Experimental procedures

As illustrated in Figure 4.19, the tensile test was conducted using an Instron tensile testing machine. The grips were screwed into the arms of the equipment, and the two notches of the grips were adjusted to ensure the test specimen could be fitted in properly. The specimen was then pushed to the end of the notch (to ensure a good alignment) before the start of the test. The information of all the tensile specimens are listed in Table 4.3.

The diameter of the gauge section was measured for every specimen thus the engineering stress could be derived during the test. Because of the small size of the specimens, it is not possible to measure the strain directly. Considering that the stiffness of the grip is high enough, its deformation can be ignored during the stretching process. Thus, the crosshead displacement was used to calculate the engineering strain (the gage length was considered as the distance between the two shoulders).
Figure 4.19 Set-up for the tensile test.

Table 4.3 Information of all tensile specimens.

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Holding time (s)</th>
<th>Specimen No.</th>
<th>Holding time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5-1</td>
<td>1</td>
<td>1.3-1</td>
<td>1</td>
</tr>
<tr>
<td>2.5-2</td>
<td>11</td>
<td>1.3-2</td>
<td>21</td>
</tr>
<tr>
<td>2.5-3</td>
<td>21</td>
<td>1.3-3</td>
<td>41</td>
</tr>
<tr>
<td>2.5-4</td>
<td>31</td>
<td>1.3-4</td>
<td>56</td>
</tr>
<tr>
<td>2.5-5</td>
<td>46</td>
<td>1.3-5</td>
<td>71</td>
</tr>
<tr>
<td>2.5-6</td>
<td>61</td>
<td>1.3-6</td>
<td>111</td>
</tr>
<tr>
<td>2.5-7</td>
<td>71</td>
<td>1.3-7</td>
<td>141</td>
</tr>
<tr>
<td>2.5-8</td>
<td>81</td>
<td>1.3-8</td>
<td>141</td>
</tr>
<tr>
<td>2.5-9</td>
<td>96</td>
<td>1.3-9</td>
<td>201</td>
</tr>
<tr>
<td>2.5-10</td>
<td>111</td>
<td>1.3-10</td>
<td>91</td>
</tr>
</tbody>
</table>

4.3.3 Comparison of experimental results and numerical predictions

During the tensile test, it was observed that some of the specimens broke with little elongation and the fracture was not in the middle of the gauge section (a comparison of the failed and successful case is shown in Figure 4.20). This was probably caused by the stress concentration during the manufacturing of tensile specimen. Thus, the data for these specimens were omitted in the following analysis.
As presented in Section 4.2.3, the relative density of all these specimens are above 0.97, and it was considered that the difference of their densities (e.g. between 0.98 and 1.00) had little impact on the results of tensile strength. Thus, holding time is the predominant factor that influences the tensile strength (i.e. bonding between powders) of each specimen in the present work.

The tensile testing results in terms of the two groups of specimens are shown in Figure 4.21 and 4.22 (red dots). A similar trend could be captured for the two figures: the tensile strength increases with the holding time, while it tends to be a stable value as the holding time approaches a certain critical value. This critical holding time was termed the bonding time under a specified forging condition.

![Figure 4.21 Experimental results and numerical prediction for bonding time under the forging condition of 2.5 kN (198.9 MPa), 1150 °C.](image)
The theoretical bonding model for powders proposed in the present work was established based on a constant pressure and temperature. The ideal method to validate this model is to conduct a compression test on two single powder particles where the pressure and temperature on the contact surface can be regarded as constant after compression. The nanoindentation technique is a possible solution to this idea [102], however, compressing single powder at a high temperature (e.g. 1000 °C or above) is not feasible under current conditions. Thus, a miniature forged component which was introduced in this chapter was employed as a substitution, of which the temperature distribution can be considered as uniform, and the contact pressure between powders during the holding time was assumed to equal the applied pressure.

According to the analysis in Chapter 3, the initial size of micro-void and the creep and diffusion data has been determined for FGH96 powder. Thus, the bonding time for this material under the forging conditions of 2.5 kN (198.9 MPa), 1150 °C and 1.3 kN (103.5 MPa), 1150 °C were predicted using the proposed bonding model as 39 s and 73 s respectively. The simulation results were illustrated using a green dashed line in Figure 4.21 and 4.22. It could be found that the numerical prediction shows a good agreement.
with the experimental results, indicating that the bonding model established in the present work is applicable for FGH96 powders (or powders with a similar surface morphology).

### 4.4 Summary

Temperature, applied pressure and holding time are three key process parameters of DPF which influence the bonding between powders. The critical holding time under a given temperature and pressure is termed the bonding time. The combination of temperature, pressure and corresponding bonding time is the bonding condition for a specific powder material.

In this chapter, FGH96 powders were DPFed to produce miniature components using a graphite tool in a dynamic testing machine (Gleeble 3800). Considering the mechanical strength of the graphite tool, the miniaturized components were manufactured under relatively low pressures (198.9, 159.2 and 103.5 MPa) at 1150 °C, and with different holding times for each pressure. The use of a miniature component makes it easy to maintain a uniform temperature during the DPF process, thus the only changing variable between the DPFed components under a given temperature and pressure is the holding time. In order to investigate the bonding conditions for the FGH96 powder, the tensile strengths of the miniature components were measured. From the experimental results, it was found that the tensile strength tends to approach a stable value with the increase of holding time, thus the corresponding critical holding time was identified as the bonding time under a given temperature and pressure. A numerical prediction of bonding time was also made using the bonding model proposed in Chapter 3, which shows a good agreement with the obtained experimental results. Thus, the applicability of the proposed bonding model for FGH96 powder (or other powder materials which have a similar surface morphology as FGH96) was validated.
Chapter 5  Methodology for Implementing the Theoretical Bonding Model in Finite Element Simulation

5.1 Introduction

The theoretical bonding model in the present work was developed to predict the extent of bonding for a certain contact area between two powder particles where the temperature and contact pressure can be regarded as constant. Since the contact condition between powders (including temperature and contact pressure) varies during the powder forging process, a single analytical model is insufficient to achieve real-time bonding results [87]. Finite Element (FE) modelling is effective for investigating interactions between objects [27, 103–105], through which the contact results between powders can be updated during the forging process. Once these contact results are derived in an FE model, they can be applied in the theoretical bonding model to calculate the bonding results for a particular area. Thus, a combined analytical and FE model has the potential to predict the real-time bonding status for all contact surfaces.

In this chapter, a hemisphere geometry was used to demonstrate the feasibility of implementing the theoretical bonding model in an FE simulation. The hemisphere model aims to mimic the contact behaviour between two curved surfaces under uniaxial compression, and it was assumed that the compression is performed at a constant temperature. Figure 5.1 shows the schematic of the hemisphere model.

Since the contact pressure is a nodal output in the FE simulation, it was presumed that a micro-void exists at each node of a contact segment, and the micro-void begins to shrink when the corresponding node starts establishing contact. According to the concept illustrated before, the bonding of a domain around this node is regarded as the shrinkage of corresponding micro-void. With the nodal contact pressure, the reduction of void size can be calculated using the theoretical model, and a bonding parameter $\omega$, ranging between 0 and 1, was defined to describe the extent of bonding, where 0 represents the
initial state and 1 means the surrounding area of this node has been fully bonded. The definition formula of $\omega$ is shown as Equation (5.1).

$$\omega = 1 - \left( \frac{a_c}{a_i} \right)^3$$  \hspace{1cm} (5.1)

where $a_c$ is the current radius of the micro-void; and $a_i$ is the initial radius of the micro-void, which is determined based on a given powder material.

Consequently, the nodal bonding results for a contact segment can be obtained in terms of a simulation step, which is served as the input of the next step. Thus, the real-time bonding status in the hemisphere model can be predicted during compression. The details of this methodology are presented in the following sections.

![Figure 5.1 Schematic of the hemisphere model in compression.](image)

### 5.2 Incorporation of the theoretical bonding model in DEFORM

#### 5.2.1 Introduction to DEFORM

DEFORM is an engineering FE software developed for analysing metal forming, heat treatment, machining and mechanical joining processes [106]. One of the advantages of DEFORM is its ability to deal with large deformations effectively, which shows a great potential in modelling powder compaction. DEFORM also provides user-defined FEM routines (FORTRAN subroutines) to fulfil customized functions [107]. USRMSH is used as a general-purpose routine that has access to many internal variables including the
normal pressure of every node (i.e. contact pressure). This subroutine is called at the beginning and end of each simulation step. USRUPD is a subroutine for user-defined nodal and elemental variables. It allows the user to calculate special state variables and store them for each node or element. User elemental variables are updated at the beginning of each iteration and at the end of a converged step; while user nodal variables are updated only at the end of a converged step. These variables can be viewed in the post processor of DEFORM.

**5.2.2 The hemisphere model in DEFORM**

The two user subroutines mentioned above were applied with a hemisphere model in DEFORM. As shown in Figure 5.2, the two hemispheres are compressed by two rigid dies (all contacts are assumed to be frictionless), of which the top one is assigned a displacement (60% of the hemisphere radius) while the bottom die is fixed. Since this hemisphere model is only for verifying the feasibility of implementing the theoretical bonding model in FE simulation, the material was selected as copper of which the data was acquired from the material library in DEFORM, and a default tetrahedral (TET) mesh was adopted. The USRMSH was used to extract the nodal normal pressures (i.e. contact pressures for contacting nodes), and these data were then saved in a common block. The common block was invoked in the USRUPD, and the pressures were applied to calculate the evolution of the micro-void size for each contacting node. Thus, the bonding parameter \( \omega \) was produced as a user-defined nodal variable. The specific procedure is indicated in the following flow chart (see Figure 5.3), and the program for USRUPD is shown in Appendix 3.

![Figure 5.2 Hemisphere model in DEFORM.](image)
Figure 5.3 Flow chart of obtaining bonding results for a simulation step in DEFORM.
5.2.3 Results and discussion

Considering the symmetry of the hemisphere model, only one of the two hemispheres was utilised to illustrate the simulation results. Figure 5.4 shows the contact pressure and self-defined bonding parameter of the bottom hemisphere for both an intermediate and the final stage (indicated as step 30 and 60 respectively) of the compression. In terms of the bonding results, the red region represents the bonded area while the other colors indicate different extents of bonding.

In order to verify that the simulation results are consistent with the theoretical results, some typical nodes on the contact patch were selected, and the normal pressure histories of these nodes were extracted then applied in the theoretical bonding model which is programmed in MATLAB (see Appendix 1). The calculated bonding parameters for each simulation step were compared with the simulated ones from DEFORM, and the two results were found to be identical, which indicates that the theoretical bonding model can be successfully implemented into an FE simulation in DEFORM through user subroutines.

Figure 5.4 Contact (left) and bonding (right) results of the hemisphere model in DEFORM at two stages of compression.
However, some abnormal contours are observed in the simulation results as shown in figure 5.4. It becomes more obvious for the bonding parameter which reflects an accumulated error of the contact pressure. Considering the irregular distribution of the abnormal results, it was speculated that they were caused by the asymmetric mesh. Thus, a more symmetric hexahedral (HEX) mesh for the hemisphere was generated using the software TrueGrid then imported into DEFORM.

Through modifying the mesh, the simulation results have been improved as presented in Figure 5.5 (an early stage of compression, step 24), in which the contours become more symmetric. Also, it was observed that the bonding region was smaller than the contact region, which is a good proof of the standpoint that contact does not equate to bonding, i.e. densification of powders cannot guarantee the material strength of powder-forged components.

However, some unexpected local behaviors can still be observed, especially for the bonding results: the contours in the contact area are not circular. Decreasing the mesh size is a common method to deal with such loss of regularity during FE simulation in terms of a Hertzian contact problem [108]. Accordingly, the size of the HEX mesh was reduced but little improvement was found for the contact results. (The mesh cannot be ultrafine considering the capability of the processor.)

Except for the mesh size, another issue that has an influence on the contact results is the approach for defining contact. DEFORM uses a node-to-surface type contact so in extreme contact condition such as the Hertzian problem differences may be seen in the local stresses due to averaging. Consequently, DEFORM might not well be suitable for solving the current problem.

Figure 5.5 Simulation results using HEX mesh in DEFORM.
5.3 Incorporation of the theoretical bonding model in Abaqus

5.3.1 Introduction to Abaqus

Abaqus is an FEA software package that has been widely used for both scientific research and industrial applications. Abaqus provides more than one approach for defining contact: general contact, contact pairs and contact elements for Abaqus/Standard; general contact and contact pairs for Abaqus/Explicit. Although each approach has somewhat unique advantages and limitations, their formulations, in general, are all based on a choice of a contact discretization, a tracking approach, and assignment of “master” and “slave” roles to the contact surfaces [109]. Considering its robust functions, Abaqus has been used to solve various contact problems [110–112].

Abaqus also offers a wide range of user subroutines with different purposes. Based on the analysis in Section 5.2, the theoretical model can be incorporated with an FE model through user subroutines, thus a combined model can be established to produce bonding results for contacting surfaces. In order to fulfill the desired function, the subroutine should occupy the following features: (1) have access to the contact pressure and (2) allow use and update user-defined state variables. FRIC is a subroutine that meets the requirements mentioned above, and it is available in ABAQUS/Standard. The FRIC is provided to define the frictional behavior between contacting surfaces, however, in the present work, it is used as a dummy routine to calculate the diffusion bonding parameter.

5.3.2 The hemisphere model in Abaqus

Figure 5.6 shows the hemisphere model built in Abaqus, and a symmetric displacement was applied on the top and bottom surfaces (30% of the hemisphere radius each). Considering the model is only for verification, an elastic material model of copper was applied, and the HEX mesh generated in TrueGrid was used. A general contact algorithm was selected for modelling the interactions between the two curved surfaces. As shown in Figure 5.7, the FRIC extracts the contact pressures (PRESS) at the end of an increment, with which the bonding parameters for all contacting nodes are calculated. Since the user-defined nodal variables in FRIC cannot be displayed in the visualisation module, all information related to the contacting nodes were exported to a text file during every increment of the FE simulation. (The program of FRIC is shown in Appendix 4.)
Figure 5.6 Hemisphere model in Abaqus.

Figure 5.7 Flow chart of obtaining bonding results for an increment in Abaqus.
5.3.3 Results and discussion

As mentioned before, the bonding parameters for contacting nodes cannot be displayed in Abaqus, and all information related to these nodes were stored in a text file through the subroutine FRIC. The information includes step number, increment number, node number, coordinates of the node, contact pressure (PRESS), current increment in time, micro-void radius and bonding parameter (ω). With the location information of contacting nodes, the contact surface can be represented in MATLAB, and the bonding results on this surface can also be displayed. Figure 5.8(a) shows the contact pressure results of the hemisphere model while Figure 5.8(b) presents the corresponding bonding status for the contact surface (in MATLAB). It can be seen that the extent of bonding for the central part is larger than its surroundings, which is reasonable since the pressure in this area is higher and the contact time is longer. Also, it was found that Abaqus gave more symmetric results (as in Figure 5.8) as compared to those in DEFORM (shown in Figure 5.5).

In order to verify that the simulation results are consistent with the theoretical results, the contact pressure (PRESS) extracted from FRIC was transferred to the analytical bonding model programmed in MATLAB (see Appendix 1), and the theoretical bonding results were thus derived and compared with the bonding results obtained from the subroutine FRIC during the FE simulation. Through comparison, the two results were found to be identical, which indicates that the theoretical bonding model can be successfully implemented into an FE simulation in Abaqus through a user subroutine.

Figure 5.8 (a) Contact pressure (CPRESS) of the hemisphere model in the visualization module of Abaqus; (b) Bonding results of the contact surface shown in MATLAB.
5.4 Predictions of bonding both during and after compression

In the section above, the theoretical bonding model has been proven to be successfully implemented in the FE simulation through user subroutines. The analytical and FE model consist of a combined model, which is able to predict the extent of bonding for contacting surfaces during compression. As can be seen from the simulation results, some parts of the contacting surface are not fully bonded thus a holding time, as in the practical powder forging process, was required after compression to achieve a fully-bonded state for all contacting surfaces.

In this section, the predictions of bonding after compression was demonstrated based on the simulation results of the hemisphere model in Abaqus (using the material data of HIPed FGH96 as shown in Figure 5.9, provided by Beijing Institute for Aeronautical Materials, BIAM). Thus, a complete cycle of bonding, from initial contact, compression to holding, for powders during a forging process can be predicted.

![Graphs showing material behaviors at different temperatures and strain rates](image)

Figure 5.9 Material behaviors of HIPed FGH96 at temperature of 1000, 1050, 1100, 1120 °C and strain rates of 0.1, 1, 10 (/s).
Figure 5.10 shows the evolution of contact pressure (CPRESS) during compression at 1120 °C (including the initial, intermediate and final stage). Assuming the final state of a contact surface (e.g. contact pressure and temperature) was maintained after compression, the bonding results for this period of time were calculated using the theoretical model in MATLAB (Appendix 1), thus the prediction of a complete bonding cycle for any particular area on the contact surface could be obtained. Figure 5.11 presents the bonding results of three typical points (indicated in figure 5.10) both during and after compression. (The compression time was set to be 50 s.) Thus, the extent of bonding for these regions can be checked during the whole bonding process.

Figure 5.10 Evolution of contact pressure during compression at 1120°C.

Figure 5.11 Bonding results of three typical points for the whole bonding cycle.
The extent of bonding in terms of the entire contacting surface was evaluated using a parameter $\Omega_c$ which is the percentage of bonded nodes ($\omega=1$) among all contacting nodes, and the definition formula is shown as Equation (5.2).

$$\Omega_c = \frac{\text{Bonded nodes}}{\text{Total contact nodes}}$$  \hspace{1cm} (5.2)

Figure 5.12 shows the extent of bonding for the contact surface in the hemisphere model both during and after compression. The bonding status for three distinct stages during the whole process are also presented. It could be found that the central part of the contact surface bonded prior to the rim since the contact starts from the centre, and a longer contacting time produces a higher level of bonding. The bonding rate for the contact area became very slow during the late stage of bonding (as indicated in Figure 5.12) due to a low contact pressure at the edge of the contact surface, where a contact was just established when the compression was completed. The bonding proceeded after compression because a holding time was applied. Based on the curve obtained in Figure 5.12, a critical holding time, i.e. bonding time, for a contact surface to achieve fully bonded state (or a specified level of bonding) could be estimated.

Figure 5.12 Extent of bonding for the contact surface both during and after compression at 1120 °C.
In terms of the industrial application, the objective of bonding between powders is to acquire satisfied material properties. To achieve this goal, it may not be necessary to make all the contact areas reach the fully bonded state, while this conjecture still needs experimental verification. A parameter, \( \Omega_{cr} \), was set in the present analysis, which represents the critical value of extent of bonding for a contact area to obtain a qualified material strength. Assuming \( \Omega_{cr} = 0.95 \), Figure 5.13 shows the required bonding time at four different temperatures (1000, 1050, 1100 and 1120 °C). The amount of compression for these four cases are identical and the compression time are all 50 s.

![Figure 5.13 Bonding time for the hemisphere model at four different temperatures (1000, 1050, 1100 and 1120 °C).](image)

It could be seen that the bonding time required at 1100 °C is longer than that at 1000 °C or 1050 °C, which seems to contradict the general trend that a higher temperature usually accelerates the bonding process (as the results for 1100 °C and 1120 °C). However, this can be explained by the difference of contact pressures between these temperatures. Figure 5.14 shows the contact results during compression in terms of the three typical points (A, B and C as indicated in Figure 5.10) at 1000, 1050, 1100 and 1120 °C. It was found that the contact pressures for 1000 °C are between 600 and 800 MPa during most of the compression time, and for 1050 °C are between 400 and 600 MPa; both are higher than the contact pressures at 1100 °C (between 200 and 400 MPa). Since the effects of contact pressure on bonding time at different temperatures for FGH96 powders have already been discussed in Section 3.5.2 (shown in Figure 3.11), it could be found that when the contact pressure is 200 MPa at 1100 °C, the bonding time required is longer.
than that in the situation of 400 MPa and 1050 °C, which indicates that for a relatively low temperature (within a reasonable range considering the forming temperature of a specific powder material), once the contact pressure is high enough, the bonding time required can be less than that at a higher temperature (as exemplified by the results in Figure 5.13). In terms of the bonding time for 1100 °C and 1120 °C, since their contact pressures are very close, the temperature becomes a dominant factor and a shorter bonding time was required for 1120 °C as expected.

![Graph showing contact pressure evolution during compression for three typical points at four different temperatures: 1000, 1050, 1100, and 1120 °C.](image)

Figure 5.14 Evolution of contact pressure during compression for three typical points at four different temperatures: 1000, 1050, 1100, and 1120 °C ($t/t_f$ is the normalized time where $t_f$ is the final time for compression.).

### 5.5 Summary

Since the contact pressure between powders varies during a practical powder forging process, a single theoretical bonding model is not sufficient to predict real-time bonding information. Finite element (FE) modelling is able to simulate the interaction between
powders, from which the evolution of contact pressure can be obtained. Thus, in this chapter, a methodology was proposed to implement the theoretical bonding model (established in Chapter 3) in an FE simulation, which is to assume a micro-void existing at each node of a contact segment, and the bonding process is regarded as the shrinkage of these micro-voids. A bonding parameter, $\omega$, was defined to describe the extent of bonding for the area around a node in an FE model.

A hemisphere model was used to demonstrate the application of the combined theoretical and FE model. The combined model was first established in DEFORM via the user subroutines USRMSH and USRUPD, while some unexpected local behaviours were observed on the contact region which was due to the contact type adopted in DEFORM. Abaqus, which provides various approaches for defining contact, was used as an alternative to improve the validity of the contact results. The user subroutine FRIC was applied as a dummy routine to calculate the bonding parameter due to its accessibility to nodal contact pressure during the simulation. The material data for HIPed FGH96 was used as a substitution of single FGH96 powder in the FE model. Thus, the combined model was applied to predict the extent of bonding for both a particular point ($\omega$) and a whole contact region ($\Omega_c$). Also, the bonding time for a contact region (to reach a specified extent of bonding) was predicted in terms of four different forming temperatures (1000, 1050, 1100 and 1120 °C).
Chapter 6  Multiscale Modelling for Predicting Bonding Time in Direct Powder Forging Process

6.1 Introduction

As mentioned before, temperature, applied load and holding time are three main process parameters for manufacturing a powder-forged component, and a critical holding time for obtaining a qualified mechanical strength (i.e. all powders are bonded together) is termed the bonding time. Thus, it is of great significance to numerically predict the bonding time under a given forming temperature and applied load, through which the optimum process parameters can be determined.

In terms of a small-scale component produced by direct powder forging (DPF) (such as the miniature cylindrical component shown in Chapter 4), a full density is easy to achieve, and a uniform temperature can be presumed during the forming process. Thus, the bonding for all powders within this component are considered to occur under the same processing condition, and the bonding time can be estimated directly using the theoretical bonding model (proposed in Chapter 3). However, for a large-scale DPFed component, the temperature distribution is always not uniform during the DPF process (especially for an early stage), which can result in a different bonding time for different locations of the component.

In order to predict the bonding time for a specified location of a relatively large-scale DPFed component, a multiscale model was proposed in this chapter, which involves a continuum FE model, a micromechanical model and the theoretical bonding model (established in Chapter 3). Figure 6.1 shows the framework of the multiscale model. The continuum FE model aims to simulate the temperature and relative density distribution of a specific component during the DPF process. As the powders within the specified location of the forged component are represented by a corresponding area in the continuum model, the evolution of the temperature and relative density for this specified
location can be predicted, which has already been studied by some previous researchers [11,97]. The micromechanical model, as indicated in Figure 6.1, serves to represent the specific area in the continuum model. In the present work, a micromechanical model was established at the powder particle level in the FE software Abaqus. In order to ensure that the micromechanical model exhibits the same densification behaviour as the corresponding area does in the continuum model, the micromechanical model was designed to present the same relative density evolution as the corresponding area does by imposing an appropriate boundary condition. Since the micromechanical model can simulate the interactions between powders during the densification process, the theoretical bonding model is implemented in the micromechanical model to calculate the bonding results using the methodology introduced in Chapter 5. At the end of this chapter, the use of this multiscale model is demonstrated via a practical DPF process.

![Figure 6.1 Framework of the multiscale model.](image)

**6.2 Micromechanical model**

**6.2.1 Overview of the model**

In the present work, a micromechanical model was built to simulate the densification of powder assemblage, which employs the hexagonal close-packed (HCP) and face-centred cubic (FCC) structures [113] as a representative volume element (RVE). The model was established in the FE software Abaqus considering its robust functions in defining
contacts, which are favourable for solving Hertz contact problem as in the present model. The use of HCP and FCC structures assumes periodic arrangement of particles, which is practically not true. However, the physics gained from such modelling laid the foundation for a deeper insight into densification process associated with randomly-packed powder assemblage. Another drawback of the proposed RVE model rests with the assumption of unchanged coordination number (defined as the average number of contacts per particle), twelve in the RVE model, which contradicts with the general trend that coordination number increases with the progression of compaction process [114]. Nevertheless, it was found that the coordination number could exceed ten when the relative density was above 0.74 [69]. Considering that the initial relative density of HCP and FCC structures are 0.74, this assumption is somewhat reasonable in terms of modelling powder compaction using the present model. The proposed micromechanical model has already been utilised to investigate the plastic deformation of powders during compaction, and good agreements with existing models were reported in terms of the macroscopic yield behaviour [115].

Similar to multi-particle finite element model (MPFEM) [27], the present model simulated the actual interaction between particles and therefore was capable of predicting realistic contact pressure between powders during compaction. On the other hand, the periodic boundary condition (PBC) was imposed at the RVE faces where neighbouring RVEs join for the sake of eliminating the boundary effects [116], which have not been avoided in the MPFEM where an entire powder assemblage is employed [27]. Also, another advantage of the present model is the much lower computation cost as a result of considering only several particles in the RVE.

6.2.2 RVEs for HCP and FCC structures

Figure 6.2(a) shows the unit cell for the HCP structure, characterised by alternating stacking of layers $\alpha$ and $\beta$ that consist of close-packed particles. The RVE for the HCP structure, shown in Figure 6.2(b), corresponds to the volume enclosed in the hexagonal prism in Figure 6.2(a). The relative density of the RVE, $\rho_r$, is defined by Equation (6.1) as the ratio of RVE density ($\rho_{RVE}$) to particle density ($\rho_p$), and is equal to the ratio of particle volume ($V_p$) to RVE volume ($V_{RVE}$).

$$\rho_r = \frac{\rho_{RVE}}{\rho_p} = \frac{V_p}{V_{RVE}}$$ (6.1)
For Figure 6.2(b), $V_p = 8\pi r_p^3$, $V_{RVE} = 24\sqrt{2}r_p^3$, where $r_p$ denotes particle radius; thus, $\rho_r = \pi/3\sqrt{2}$.

Likewise, Figure 6.3 depicts the unit cell and RVE for the FCC structure. The unit cell in Figure 6.3(a) is a cube and contains close-packed particles in \{1 1 1\} planes. Along the [1 1 1] direction, three neighbouring layers in \{111\} planes – $\alpha$, $\beta$ and $\gamma$, periodically stack, which can be treated as the insertion of an additional layer $\gamma$ into the unit cell for the HCP structure, as illustrated in Figure 6.3(b). In line with Figure 6.2, the RVE for the FCC structure is the volume within the hexagonal prism, and corresponds to $\rho_r$ equal to $\pi/3\sqrt{2}$.

It should be noted that the hexagonal prism describes the same pattern of particle arrangement as the cubic unit cell, and is used only to be consistent with Figure 6.2 in terms of the RVE geometry.

Although RVEs for HCP and FCC differ in the number of close-packed layers, both deal with periodic arrangement of the constituent layers along the stacking direction (i.e. the
direction normal to the close-packed layers), as indicated in Figures 6.4(a) and 6.4(b). However, stacking of close-packed layers that usually take the three forms - $\alpha$, $\beta$ and $\gamma$ is not limited to a periodic feature but can occur with an arbitrary manner as exemplified by Figure 6.4(c). It is noted that any arbitrary stacking pattern can be decomposed into the combination of the two periodic features represented by the RVEs for HCP and FCC. Therefore, a study on the compaction behaviour of the RVEs for HCP and FCC lays a foundation for understanding the compaction behaviour of an infinite number of close-packed layers arranged either periodically or arbitrarily.

Figure 6.4 (a) Periodic arrangement of layers $\alpha$ and $\beta$ in the stacking direction; (b) Periodic arrangement of layers $\alpha$, $\beta$ and $\gamma$ in the stacking direction; (c) An example of an arbitrary stacking of layers $\alpha$, $\beta$ and $\gamma$ that can be regarded as combinations of the RVEs for HCP and FCC.

### 6.2.3 Periodic boundary conditions (PBCs)

Figure 6.5(a) schematically shows the assemblage of an infinite number of RVEs that thus involves an infinite number of particles. Upon external loading, the assembled entity exhibits periodicity in relative displacement which is defined as the displacement of a point within a RVE relative to that of the RVE reference point [116,117], i.e. points at the same position in each RVE have the same relative displacements. Consider the two arbitrarily-chosen RVEs in Figure 6.5(a) that respectively take O and O’ as reference points (O and O’ have the same position in the respective RVEs), the relative displacement of an arbitrary point P in one RVE is equal to that of point P’ in the other RVE, which has the same position as P in the respective RVEs, mathematically expressed by Equation (6.2).
\[ u(P) - u(O) = u(P') - u(O') \]
\[ v(P) - v(O) = v(P') - v(O') \]
\[ w(P) - w(O) = w(P') - w(O') \] (6.2)

where \( u, v \) and \( w \) denote respectively the displacements along the axes \( X, Y \) and \( Z \).

Figure 6.5 (a) Schematic about projection of an infinite assemblage of RVEs in X-Y plane, where the points in each pair (\( O - O' \) and \( P - P' \)) have the same position in the respective RVE; (b) RVE1 and RVE2 in (a) isolated to illustrate constraints imposed on the six boundary faces where neighbouring RVEs adjoin. E1-E6 denote the edges of RVE1; the points in each pair (\( A - A' \) and \( B - B' \)) have the same position respectively in RVE1 and RVE2.

While a single RVE, e.g. the one shaded in Figure 6.5(a), is used to study the mechanical behaviour of the assembled entity, periodic boundary conditions (PBCs) are imposed at the three pairs of opposing boundary faces (parallel to the \( Z \) axis) of distance \( D_0 \) from one another where neighbouring RVEs adjoin, in order to maintain continuity of the displacement field [118,119]. To facilitate the expression of the constraint equations related to the PBCs, three local rectangular coordinate systems \( x_i y_i Z \) \((i = 1, 2, 3)\) are defined in Figure 6.5(b), with the respective axis \( x_i \) normal to the boundaries and \( x_i y_i \) related to the global system \( XY \) by Equation (6.3).

\[
\begin{bmatrix} x_i \\ y_i \\ z_i \end{bmatrix} = \begin{bmatrix} \cos \theta_i & \sin \theta_i \\ -\sin \theta_i & \cos \theta_i \end{bmatrix} \begin{bmatrix} X \\ Y \end{bmatrix} \quad (i = 1, 2, 3) \] (6.3)
where \( \theta_i \) equals 0\(^0\), 60\(^0\), 120\(^0\) respectively for \( i = 1, 2, 3 \).

The boundary constraints are illustrated through the two adjoining RVEs in Figure 6.5(b), which take A and A’ (having the same position in the respective RVEs, as with O and O’ in Figure 6.5(a)) as the respective reference points. B and B’ have the same position respectively in RVE1 and RVE2, and are located arbitrarily within the two paralleled boundary faces normal to axis \( x_i \), denoted respectively by \( x_i = D_0/2 \) and \( x_i = -D_0/2 \). According to Equation (6.2), the constraints on B and B’ are defined as:

\[
\begin{align*}
\mathbf{u}_1(B) - \mathbf{u}_1(B') &= \mathbf{u}_1(A) - \mathbf{u}_1(A') \\
\mathbf{v}_1(B) - \mathbf{v}_1(B') &= \mathbf{v}_1(A) - \mathbf{v}_1(A') \\
\mathbf{w}(B) - \mathbf{w}(B') &= \mathbf{w}(A) - \mathbf{w}(A')
\end{align*}
\]  

(6.4)

where \( u_i \) and \( v_i \) represent respectively the displacements along axes \( x_i \) and \( y_i \). The right-hand side of Equation (6.4) represents the relative displacement between reference points A and A’, which can be related to the macroscopic strain of the assembled entity by Equation (6.5) [117].

\[
\begin{align*}
\mathbf{u}_1(A) - \mathbf{u}_1(A') &= \mathbf{\varepsilon}_{x_1}(x_1(A) - x_1(A')) + \mathbf{\gamma}_{x_1y_1}(y_1(A) - y_1(A')) \\
&\quad + \mathbf{\gamma}_{x_1Z}(Z(A) - Z(A')) \\
\mathbf{v}_1(A) - \mathbf{v}_1(A') &= \mathbf{\varepsilon}_{y_1}(y_1(A) - y_1(A')) + \mathbf{\gamma}_{y_1Z}(Z(A) - Z(A')) \\
\mathbf{w}(A) - \mathbf{w}(A') &= \mathbf{\varepsilon}_Z(Z(A) - Z(A'))
\end{align*}
\]  

(6.5)

where \( \mathbf{\varepsilon}_{x_1} \), \( \mathbf{\varepsilon}_{y_1} \) and \( \mathbf{\varepsilon}_Z \) are normal strains respectively in the directions of axes \( x_i \), \( y_i \) and \( Z \), and \( \mathbf{\gamma}_{x_1y_1} \), \( \mathbf{\gamma}_{x_1Z} \), \( \mathbf{\gamma}_{y_1Z} \) denote shear strains.

The magnitudes of the macroscopic strain components depend on the loading path, the features of the die enclosing the assembled powder particles, and the platen that compresses the powder. Assuming the geometry of the die does not change and the platen remains flat, then \( \mathbf{\gamma}_{x_1y_1} = \mathbf{\gamma}_{x_1Z} = \mathbf{\gamma}_{y_1Z} = 0 \). Equation (6.5) therefore becomes:

\[
\begin{align*}
\mathbf{u}_1(A) - \mathbf{u}_1(A') &= \mathbf{\varepsilon}_{x_1}(x_1(A) - x_1(A')) \\
\mathbf{v}_1(A) - \mathbf{v}_1(A') &= \mathbf{\varepsilon}_{y_1}(y_1(A) - y_1(A')) \\
\mathbf{w}(A) - \mathbf{w}(A') &= \mathbf{\varepsilon}_Z(Z(A) - Z(A'))
\end{align*}
\]  

(6.6)
With reference to Figure 6.5(b), RVE2 can be thought of as the translation of RVE1 along the negative \( x_1 \) direction by \( D_0 \); hence, Equation (6.7) is obtained.

\[
\begin{align*}
x_1(A) &- x_1(A') = D_0 \\
y_1(A) &- y_1(A') = 0 \\
Z(A) &- Z(A') = 0
\end{align*}
\] (6.7)

Combining Equation (6.4) and (6.6) with (6.7) leads to:

\[
\begin{align*}
u_1(B) &- u_1(B') = D_0 \epsilon_{x_1} \\
v_1(B) &- v_1(B') = 0 \\
w(B) &- w(B') = 0
\end{align*}
\] (6.8)

As B and B’ are arbitrary points respectively within the two boundary faces, \( x_1 = D_0/2 \) and \( x_1 = -D_0/2 \), Equation (6.8) can be expressed as:

\[
\begin{align*}
u_1(x_1 = D_0/2) &- u_1(x_1 = -D_0/2) = D_0 \epsilon_{x_1} \\
v_1(x_1 = D_0/2) &- v_1(x_1 = -D_0/2) = 0 \\
w(x_1 = D_0/2) &- w(x_1 = -D_0/2) = 0
\end{align*}
\] (6.9)

Similarly, the constraints on the other two pairs of boundary faces, respectively normal to axes \( x_2 \) and \( x_3 \), are:

\[
\begin{align*}
u_2(x_2 = D_0/2) &- u_2(x_2 = -D_0/2) = D_0 \epsilon_{x_2} \\
v_2(x_2 = D_0/2) &- v_2(x_2 = -D_0/2) = 0 \\
w(x_2 = D_0/2) &- w(x_2 = -D_0/2) = 0 \\
u_3(x_3 = D_0/2) &- u_3(x_3 = -D_0/2) = D_0 \epsilon_{x_3} \\
v_3(x_3 = D_0/2) &- v_3(x_3 = -D_0/2) = 0 \\
w(x_3 = D_0/2) &- w(x_3 = -D_0/2) = 0
\end{align*}
\] (6.10)

where \( u_2, \ u_3, \ v_2 \) and \( v_3 \) represent respectively the displacements along axes \( x_2, \ x_3, \ y_2 \) and \( y_3 \) (Figure 6.5(b)); \( \epsilon_{x_2} \) and \( \epsilon_{x_3} \) are normal strains of the assembled entity respectively in the directions of axes \( x_2 \) and \( x_3 \).
The second assumption made in the present work is that the powder assembly shrinks or expands evenly in all directions across X-Y plane, then:

$$\varepsilon_{x_1} = \varepsilon_{x_2} = \varepsilon_{x_3} = \varepsilon_r$$ \hspace{1cm} (6.11)

where $\varepsilon_r$ is the radial strain. With Equation (6.11), the constraints on the three pairs of boundary faces, governed by Equation (6.9) and (6.10), can be unified into a single form, given by Equation (6.12).

$$u_i(x_i = D_0/2) - u_i(x_i = -D_0/2) = D_0\varepsilon_r$$
$$v_i(x_i = D_0/2) - v_i(x_i = -D_0/2) = 0 \hspace{1cm} (i = 1, 2, 3)$$
$$w(x_i = D_0/2) - w(x_i = -D_0/2) = 0$$ \hspace{1cm} (6.12)

6.2.4 Finite element model

1) Model construction

Geometric models of the RVEs for HCP and FCC were established via MATLAB programming and shown in Figure 6.6. As the RVE for HCP is symmetric with respect to the plane $Z = H_1/2$, half of the model as indicated is used for simulation. For finite element analysis within highly elastic and plastic structural domains, hexahedral mesh has a good performance of reducing error, decreasing element counts, and improving reliability [120]. However, considering the spherical powder particle is an ideal case which would be replaced by irregular shapes for further studies, the tetrahedral (TET) element, which is applicable for complex geometries, was adopted. Also, the remeshing for large deformation is often not feasible for the 3D hexahedral (HEX) mesh [121]. Thus, the tetrahedral element type C3D4 was employed in the present work to discretize the geometric entities and the number of elements was chosen to ensure convergence of the computed results. The nodes lying in each pair of opposing boundary faces, $x_i = \pm D_0/2$ ($i =1, 2, 3$) are symmetric with respect to the middle plane $x_i = 0$, which allows for imposition of the PBCs (Equation (6.12)) through equation constraints in the context of Abaqus software. Practically, the nodes for a pair of opposing faces were placed into two separate node sets, in a manner that each pair of symmetric nodes has the same position.
in the respective nodes sets. Equation constraints were applied to the node sets in one step, instead of to all pairs of symmetric nodes one by one.

Figure 6.6 Geometric models with symmetric nodes for (a) RVE for HCP and (b) RVE for FCC.

2) Boundary conditions

Boundary conditions for the RVEs of HCP and FCC are given respectively by Equation (6.13) and (6.14).

\[
\begin{align*}
&u = 0, \quad \text{at } Z = H_1/2 \\
&\dot{v} = \dot{v}_{HCP}, \quad \text{at } Z = H_1
\end{align*}
\]

\[
\begin{align*}
&u = 0, \quad \text{at } Z = 0 \\
&\dot{v} = \dot{v}_{FCC}, \quad \text{at } Z = H_2
\end{align*}
\]

where \( \dot{v} \) denotes the velocity along axis Z.

Integrating \( \dot{v} \) with respect to time, \( t \), the axial strains of the RVEs, \( \varepsilon_a \), for HCP and FCC, are calculated respectively by Equation (6.15) and (6.16).

\[
\varepsilon_a^{HCP} = \frac{2 \int \dot{v}_{HCP} \, dt}{H_1}
\]

\[
\varepsilon_a^{FCC} = \frac{2 \int \dot{v}_{FCC} \, dt}{H_2}
\]
\[ \varepsilon_{a}^{\text{FCC}} = \frac{\int \dot{\varepsilon}_{\text{FCC}} dt}{H_2} \]  

(6.16)

3) Stress state for RVEs

With references to Equation (6.11), (6.13) and (6.14), the macroscopic (or effective) stress state for the RVEs, \( \sigma \), is:

\[
\sigma = \begin{bmatrix} \sigma_r & 0 & 0 \\ 0 & \sigma_r & 0 \\ 0 & 0 & \sigma_a \end{bmatrix}
\]  

(6.17)

where \( \sigma_r \) represents the average stress in the direction of axis X and can be quantified by Equation (6.18):

\[
\sigma_r = \frac{\sum_{\text{node}} F_{x_i}^{x_i} \cdot A_{\text{Face}}^{x_i}}{A_{\text{Face}}^{x_i}}
\]  

(6.18)

where \( F_{x_i}^{x_i} \) denotes the force along axis \( x_i \) (\( i = 1, 2, 3 \)) acting on a node within the boundary face normal to \( x_i \), and \( A_{\text{Face}}^{x_i} \) is the area of that boundary face.

Due to the identical pattern of particle arrangement along axes \( x_1, x_2 \) and \( x_3 \), the resulting \( \sigma_r \) given by Equation (6.18) is expected to be the same along these directions; hence, any of the 6 boundary faces (Figure 6.6) can be used to determine \( \sigma_r \).

Similarly, the average stress in the direction of axis Z, \( \sigma_a \), is calculated as:

\[
\sigma_a = \frac{\sum_{\text{node}} F_{Z}^{Z} \cdot A_{\text{Face}}^{Z}}{A_{\text{Face}}^{Z}}
\]  

(6.19)

where \( F_{\text{node}}^{Z} \) represents the force along axis Z acting on a node within the planes, \( Z = H_1 \) and \( Z = H_2 \) (Figure 6.6) respectively for RVEs of HCP and FCC, where velocity boundary conditions are applied, and \( A_{\text{Face}}^{Z} \) is the area of that plane. \( A_{\text{Face}}^{x_i} \) is correlated to radial strain, \( \varepsilon_r \), and axial strain, \( \varepsilon_a \), by Equation (6.20), where \( H \) is equal to \( H_1/2 \) and \( H_2 \),
respectively for the half RVE of HCP and RVE of FCC, and \( \varepsilon_a \) is determined by Equation (6.15) and (6.16):

\[
A_{\text{Face}}^{x_i} = \left[ \frac{D_0}{\sqrt{3}} (1 + \varepsilon_r) \right] [H(1 + \varepsilon_a)]
\]

(6.20)

\( A_{\text{Face}}^{Z} \) is related to \( \varepsilon_r \) by Equation (6.21).

\[
A_{\text{Face}}^{Z} = \frac{\sqrt{3}}{2} D_0^2 (1 + \varepsilon_r)^2
\]

(6.21)

4) Material flow behaviour

The proposed RVE model has no restrictions on the flow behaviour of powder material. In this piece of work, linear elastic perfectly-plastic flow was used as an example to run the present model for the following analysis. In terms of the material, the elastic modulus, \( E \), and yield strength, \( \sigma_Y \), are assumed as 120 GPa and 1000 MPa respectively. The ratio of elastic modulus to yield strength is 120, which does not refer to a specific material but is an approximation of that for some metals and is in the same range of \( E/\sigma_Y \) values used in the literature [27].

6.2.5 Results and discussion

1) RVE anisotropy

In actual situation, powders are randomly distributed and hence the assembly is isotropic. However, the use of RVEs potentially leads to anisotropy because of the arrangements of particles, which is close-packed across the X-Y plane (Figure 6.6), but not within other planes. The degree of RVE anisotropy was assessed by subjecting the HCP RVE to isostatic compaction, i.e. \( \varepsilon_a = \varepsilon_r \), followed by comparison of the resulting axial and radial stresses, \( \sigma_a \) and \( \sigma_r \), as calculated by Equation (6.18) and (6.19). Evaluation of the FCC RVE in this regard is considered unnecessary, because the HCP RVE has the same pattern of particle arrangement across the X-Y plane as the FCC RVE, and the same distance along the Z axis for neighbouring close-packed planes. Figure 6.7 shows the axial and radial stresses obtained, with powder material assumed to follow linear elastic perfectly-
plastic flow; friction was neglected in these simulations. The difference between $\sigma_a$ and $\sigma_r$ is negligible over the full range of $\rho_r$ displayed. Particularly at $\rho_r$ higher than 0.9, $\sigma_a$ and $\sigma_r$ are expected to be nearly the same, because at full densification the powder assemblage behaves as a continuous block of isotropic material. The negligible difference between $\sigma_a$ and $\sigma_r$ indicates that the RVE models here are effectively isotropic when used to simulate the densification of a powder assemblage via inter-particle mechanical interaction. Although the RVE itself is anisotropic [122] from the crystallographic perspective, isotropic behaviour was obtained in the present model, which is mainly because the interatomic attractive or repulsive forces associated with the crystallographic structures are replaced by the mechanical interaction between particles.

![Figure 6.7](image)

Figure 6.7 Radial and axial stresses during isostatic compaction, obtained from the HCP RVE model with powder material taken to follow linear elastic perfectly-plastic flow.

2) HCP RVE vs. FCC RVE

The mechanical response of the RVEs for HCP and FCC were compared in terms of the axial stress during isostatic and closed-die compaction ($\varepsilon_r = 0$ and $\varepsilon_a \neq 0$). Figure 6.8 indicates that the two RVE models give almost the same values of $\sigma_a$ for both loading paths. Such identical responses can be interpreted from the perspective of RVE structures.
Figure 6.9(a) illustrates that the RVE for FCC can be thought of as the adjoining of three structures labelled S1, S2 and S3, while S1 and S3 both correspond to the RVE for HCP. S2, on the other hand, is essentially another form of the RVE for HCP. The solid and dotted circles in Figure 6.9(b) representatively depict two layers of close-packed particles, resembling the HCP structure. The RVE for HCP is not unique, and the region outlined in the two hexagons corresponds to two different RVEs as indicated by the 3D profiles, one of which is S3 and the other takes the form of S2. Although S2 and S3 are geometrically different, both refer to the same pattern of structure and hence should result in a common mechanical response. Since S1, S2 and S3 are all RVEs for HCP, their combination into the form of RVE for FCC, as seen in Figure 6.9(a), should generate the same mechanical responses as the HCP RVE, which has been demonstrated in Figure 6.8. In view of the identical responses between HCP RVE and FCC RVE, either one can be used to simulate the densification of a powder assemblage. In practical application, the HCP RVE should be preferentially adopted because it is computationally economic, due to the smaller number of particles (the number of particles included in HCP and FCC RVEs are respectively six and nine, as seen from Figure 6.2 and 6.3).

Figure 6.8 Axial stress computed for HCP and FCC RVEs subjected to two loading paths – isostatic and closed-die, with powder material assumed to be linear elastic perfectly-plastic.
Figure 6.9 (a) FCC RVE and the decomposition; (b) Two different RVEs for the common structure, composed of two layers of close-packed particles resembling the HCP structure.

3) Contact pressure during closed-die compaction

Contact pressure during the closed-die compaction ($\varepsilon_r = 0$ and $\varepsilon_a \neq 0$) was exemplified by the six nodal points numbered sequentially in Figure 6.10(a), of which the point 1 is close to the particle initial contact point. Figure 6.10(b) shows the variation of contact pressure during the densification process. For all six points, an initial rapid increase in contact pressure is observed, and the rate of initial increase appears slightly higher at larger relative density, $\rho_r$. Following the initial rapid increase, the contact pressure tends to vary insignificantly around a value equal to twice of the material yield strength (for $\rho_r < 0.92$), i.e. $2\sigma_Y = 2000$ MPa, as indicated in Figure 6.10(b); while (for $\rho_r > 0.92$) it increases dramatically with $\rho_r$ in a manner identical with the macroscopic hydrostatic pressure, $p$. Beyond $\rho_r$ of 0.95, the contact pressure for all points coming into contact in different stages converge and could be approximated by $p$.

Regarding elastoplastic materials involved in the present work, Figure 6.10(b) suggests that the contact pressure can be taken as a specific value and $p$ for $\rho_r$ less than 0.92 and more than 0.95 respectively, while an interpolation of contact pressure can be applied in between of the two bounds. In terms of materials exhibiting different mechanical behaviours, the present model can also be used to determine the evolution of contact pressure during powder compaction.
Statistical data on the contact pressure is depicted by the histograms in Figure 6.11, where the ordinate and abscissa represent, respectively, the number of nodes in contact and associated contact pressure. The sum of ordinate value for all columns is equal to the total number of contacting nodes. As can be seen, this value increases with $\rho_r$, which agrees
with the fact that contact area increases during densification. A normal distribution function was fitted to the histograms as illustrated in Figure 6.11. The prevalent contact pressure for $\rho_r = 0.86$ is around 2000 MPa, which becomes higher when $\rho_r$ increases to 0.95. It could be found that the magnitude and tendency of the prevalent contact pressure are consistent with the results obtained in Figure 6.10(b).

![Figure 6.11 Statistical representation of contact pressure for contacting nodes at (a) $\rho_r = 0.86$ and (b) $\rho_r = 0.95$.](image)

6.3 Multiscale Modelling

In this section, the use of the proposed multiscale model to predict bonding time was demonstrated via a practical direct powder forging (DPF) process. Figure 6.12 shows the cross-section of a component manufactured by DPF, during which the FGH96 powders are filled into a cylindrical container (92 mm in diameter and 90 mm in height) and the vacuumed and sealed container is heated in a furnace to 1070 °C then DPFed using a single-acting hydraulic machine. This work has been conducted in a companion project by Shuyun Wang et al. [97]. As presented in Figure 6.12, different locations of the forged component are distinguished using numbers from 1 to 30. In the following analysis, the bonding time for two typical locations, 3 and 28, were predicted using the multiscale model illustrated in Figure 6.1, and the specific procedure is detailed as follows.
6.3.1 Continuum FE model

A continuum model has been proposed by Shuyun Wang et al. in the FE software DEFORM to simulate the above-mentioned DPF process, which is illustrated in Figure 6.13(a) [97]. In their model, a thermal analysis has been conducted for the powders (including the container and the covers) during the transfer from the furnace to the hydraulic machine, and the prediction of the temperature distribution before the start of the forging test is presented in Figure 6.13(b). As can be seen, the temperature distribution of the powders is not uniform, which can lead to differences in bonding time for different locations of the component. In the following analysis, this temperature distribution was assumed to be maintained during the forging process.

Apart from the thermal analysis, their model has also been applied to predict the distribution of relative density during the DPF process. Figure 6.14 shows the evolution of the simulated relative density for different areas in the continuum model. Each curve represents a specific area which corresponds to a location of the DPFed component, and numbers shown in Figure 6.14 are consistent with those in Figure 6.12.

Figure 6.12 The cross-section of a DPFed component [97].
Figure 6.13 (a) The continuum FE model for DPF in DEFORM; (b) Temperature distribution of the powders before the start of the forging process [97].

Figure 6.14 Evolution of simulated relative density during DPF process for the locations indicated in Figure 6.12 [97].
6.3.2 Micromechanical (RVE) model

1) Determination of the boundary conditions

In order to predict bonding between powders (using the proposed bonding model), contact pressure is a necessary input, however, it cannot be derived in the continuum model. Thus, the micromechanical model established in Section 6.2, i.e. the RVE model, was introduced into the multiscale model to investigate the interaction between powders (according to Section 6.2.5, the HCP RVE was adopted in the present analysis).

As mentioned in Section 6.1, the RVE model serves to represent a specific area in the continuum model; thus, it is important to make sure that the RVE model exhibits the same densification behaviour as the corresponding area in the continuum model. Since relative density is a fundamental parameter to characterise a densification process, the RVE model was designed to present the same relative density evolution as the corresponding area does by imposing an appropriate boundary condition.

Considering the set-up for the DPF process shown in Figure 6.12, it is analogous to the hot-repressing process, of which the material flow during densification is mainly in the direction of compression [2]; thus, a closed-die compaction ($\varepsilon_r = 0$ and $\varepsilon_a \neq 0$) was assumed for the RVE model in the present analysis. In terms of a closed-die compaction, the vertical displacement imposed on the top plane of the HCP RVE model ($u_Z$) can be derived as a function of the relative density ($\rho_r$) based on Equation (6.1):

$$u_Z = \left( \frac{2\sqrt{3}\pi}{9} r_p \right) \frac{1}{\rho_r} - \frac{2\sqrt{\pi}}{3} r_p$$  \hspace{1cm} (6.22)

where $r_p$ is the radius of the particle in the RVE model.

As the relative density evolutions for location 3 and 28 have already been predicted in the continuum model (shown in Figure 6.14), based on which the displacements for their corresponding RVE models can be calculated using Equation (6.22). Figure 6.15 shows the calculated results in terms of the normalized displacement during compaction, which were imposed on the RVE models as the boundary conditions. Thus, each RVE model would exhibit the same relative density evolution as the predictions from the continuum model.
Figure 6.15 Displacement imposed on the top plane of the HCP RVE model (normalized by the initial height of the HCP RVE) during compaction ($t_F$ represents the total compression time in Abaqus) to obtain the same density evolution as the corresponding location in the continuum FE model.

2) Material flow behaviour

Since the particles in the RVE model represent the real powders, the material properties for the single powder particle are required to obtain more realistic simulation results. However, the material behaviour for a single FGH96 powder at high temperature (i.e. 1000°C and above) is not available. Thus, a linear elastic perfectly-plastic model was employed for the moment in the RVE model to approximate the powder behaviour at 1070°C. The elastic modulus and the yield strength are 140 GPa and 300 MPa respectively, which were interpolated from the data for HIPed FGH96 material (provided by BIAM). It should be noted that the rate effects were ignored in the present analysis, which could be reached by using a viscoplastic material model.

6.3.3 Modelling results

As the boundary conditions and the material behaviour were determined, the RVE model was applied to simulate the powder compaction process, and thus the contact results between different particles were obtained in terms of the two locations of the component. The hemispherical particle located in the center of the top plane of the RVE model was
used to demonstrate the simulated contact results, which contacts with nine other surfaces during the densification process; and the results of the contact pressure (CPRESS) obtained in the two RVEs (representing the two locations, 3 and 28) were presented at two distinct times as shown in Figure 6.15 ($t_1$ and $t_2$ were indicated in Figure 6.15). It was found that since the paths of the displacement for the two RVEs are different (as in Figure 6.15), the relative densities exhibited by the two models at the same specific time point are distinct, which then result in diverse contact results. Since the contact pressure has a crucial impact on the bonding process, the predicted bonding results during the compaction would be different in terms of the two locations.

Figure 6.16 Simulated relative density and corresponding contact results at two distinct times, $t_1$ and $t_2$, for (a) location 3 and (b) location 28.
Based on the simulated contact pressures, together with the temperature obtained in the continuum model, the bonding results for location 3 and 28 were calculated using the theoretical bonding model, which was incorporated with the RVE model using the methodology detailed in Chapter 5. Considering the compression time for direct forging the component shown in Figure 6.11(a) is 2.1 s [97], the extent of bonding for all the contacting surfaces ($\Omega_c$) in the RVE model was predicted based on Equation (5.2), and the predictions are shown in Figure 6.17. Thus, the bonding times for the two RVE models, corresponding to location 3 and 28, were estimated as 23 s and 17 s respectively, which indicated that the applied load and temperature should be held for at least 23 s and 17 s to ensure the powders within location 3 and 28 are fully bonded correspondingly.

![Figure 6.17 Predictions of the bonding time (i.e. critical holding time) for the two locations.](image)

Although the numerical predictions still need experimental verification, the proposed multiscale model shows a promising application for determining the critical value of an industrial process parameter, i.e. holding time, through which the mechanical strength of the forged components could be guaranteed.
6.4 Summary

In terms of a large-scale DPFed component (compared with the miniature DPFed component in Chapter 4), the temperature and relative density distribution are always not uniform during the forging process. Thus, the bonding time required for distinct locations of the component are different. In order to predict the bonding time for a specified location, a multiscale model was proposed in the present work, which consists of a continuum FE model, a micromechanical model and the theoretical bonding model (established in Chapter 3). The continuum model is usually established according to the specific shape of the DPFed component in an FE software, and is used to predict the temperature and relative density evolution of a specified location, which serve as the input of the micromechanical model. The micromechanical model is built to represent a specific area of the continuum model (corresponding to the specified location of the forged component), and is designed to exhibit the same densification behaviour as the continuum material of the specific area does through imposing an appropriate boundary condition. Thus, the theoretical bonding model can be implemented in the micromechanical model (via the methodology proposed in Chapter 5) to predict the bonding time, which is the critical holding time for the DPF process that allow all the powders in the specified location of the DPFed component to be fully bonded.

A continuum FE model has already been proposed by Wang et al. [97] in a companion project to simulate the DPF process of a relatively large-scale FGH96 component. In this chapter, the proposed multiscale model was applied to predict the bonding time for two typical locations of this DPFed component. The micromechanical model involved in the proposed multiscale model was built at the powder particle level, which employs the HCP and FCC structures as the representative volume element (RVE). The model applies the periodic boundary condition (PBC) to eliminate the boundary effects [116] during the simulation of the powder compaction process. The micromechanical model is able to predict more realistic particle interactions compared to DEM, while requires less computation time than the MPFEM. Since the RVE model was established in the FE software Abaqus, the theoretical bonding model was implemented in it (using the methodology in Chapter 5) to predict bonding time for the two specified locations, which is instructive for determining the critical holding time in terms of the practical DPF process.
Chapter 7  Conclusions and Suggested Future Work

7.1  Conclusions

7.1.1  Theoretical bonding model for powders

The bonding between powders during a powder forging process was regarded as the shrinkage of micro-voids formed by the observed indents on the powder surface. This concept was proposed based on the previous work of solid-state diffusion bonding (DB) between two prepared surfaces, where the DB process is considered as the elimination of voids originated from the roughness of two adjoining surfaces.

A mechanism-based analytical model was established for modelling the bonding process between powders during powder forging using the methodology by Derby and Wallach (the methodology is originally proposed for modelling DB between prepared surfaces). The main difference between the present model and the existing DB models is the assumed shape for the interfacial void: discrete spheres and parallel long strips respectively, which results in the model to be built in three-dimensional (3D) state and two-dimensional (plane strain) state correspondingly.

The assumption of the micro-void shape in the present work was made based on the SEM observation of the surface of argon atomised FGH96 powder. Thus, the proposed bonding model is applicable for any powder materials that have a similar surface morphology as FGH96. The model can be used to predict the extent of bonding for a contact region also to identify the bonding conditions for this region (i.e. a combination of time, pressure and temperature) provided that the two inputs of the model are known or can be derived in terms of a specific powder material: (1) creep and diffusion data, and (2) initial size of the micro-void (determined by the size of the observed indents on the powder surface).

The proposed bonding model was applied to predict the extent of bonding of two prepared copper surfaces; the results obtained from the present model was found to share a similar trend with two previous DB models (Guo [34] and Derby [48]), and the prediction is in a
reasonable range of the experimental results produced by Derby [48], through which the rationality of the model was validated. However, a gap was noted for the predicted results between the present model and the previous DB models, which is mainly due to the assumed void shape (the spherical shape in the present model is assumed for powders, not for prepared surfaces).

Since the present bonding model was established based on constant temperature and pressure, an ideal experimental method to verify the model is to conduct tests on two single powders. However, compression of two particles at high temperature (e.g. 1000 °C or above) is not feasible currently. A miniature component which was manufactured by direct powder forging (DPF) using FGH96 powders was utilised as a substitution, of which the temperature distribution can be considered as uniform and the contact pressure between powders was assumed to equate to the applied pressure after the powders were compressed to full density. The components were DPFed under relative low applied loads at 1150 °C considering the mechanical strength of the graphite tool. For each condition, the components were fabricated with different holding times, and the tensile strength of each component was obtained and compared. It was found the tensile strength, under a given temperature and pressure, tends to approach a stable value with the increase of holding time, and the critical holding time corresponding to the stable value was identified as the bonding time. The bonding times for two forming conditions (198.9 MPa, 1150 °C and 103.5 MPa, 1150 °C) were determined, and the corresponding numerical prediction derived from the present bonding model shows a good agreement with these experimental results, which indicating the model is applicable for bonding between powders.

7.1.2 Implementation of the theoretical bonding model in FE simulation

A methodology was proposed to implement the theoretical bonding model in an FE simulation, which is to assume a micro-void located at each node of a meshed contact surface and thus the nodal information (e.g. contact pressure) can be applied in the theoretical model to calculate the bonding results. A bonding parameter, \( \omega \), was defined to represent the extent of bonding of a node (fractional volume bonded of a micro-void), while another parameter, \( \Omega_c \), was proposed to describe the extent of bonding of a contact surface (the percentage of bonded nodes among all contacting nodes).
The methodology was demonstrated via a hemisphere model, which aims to mimic the contact between two curved surfaces. The hemisphere model was first established in the FE software DEFORM, and the theoretical bonding model was incorporated in the user subroutines USRMSH and USRUPD to calculate the bonding parameter \( \omega \) for every contacting node during the simulation. However, since DEFORM is not that effective in modelling the contact between two curved surfaces, Abaqus was used as a substitution in which the theoretical bonding model was implemented via the user subroutine FRIC. Thus, the real-time bonding information was derived for every contacting nodes during the whole bonding cycle (i.e. from initial contact to fully bonded state). Also, the bonding time for the contact surface in the hemisphere model was predicted under different temperatures (using the material flow behaviour of HIPed FGH96). The bonding results obtained in the hemisphere model demonstrated that the theoretical bonding model could be appropriately implemented in an FE simulation using the methodology proposed in the present work.

### 7.1.3 Micromechanical model for powder densification

A micromechanical model was established at the particle level in Abaqus to simulate the powder densification process, which employs the hexagonal close-packed (HCP) and face-centred cubic (FCC) structures as a representative volume element (RVE). The relative density covered by the present RVE model is in the range from 0.74 (i.e. initial relative density of HCP and FCC structures) up to nearly full density, which extends the upper limit of DEM (i.e. 0.82). Although the use of HCP and FCC structures give unchanged coordination number as 12, it is considered to be acceptable since the coordination number has been reported in the range of 10-14 during stage II compaction (relative density larger than 0.8) [69].

The RVE model exhibited negligible anisotropy during compaction although the RVE itself is anisotropic in nature [122]. This is because the interaction of adjacent particles in the present model was accomplished through contact and subsequent mechanical deformation at particle interfaces, rather than the interatomic attractive or repulsive forces as with crystallographic structures. Therefore, the present RVE model can be considered as isotropic when it is used to simulate densification of powder assembly.
Similar to MPFEM, the present RVE model simulated the actual interaction between particles and therefore is capable of predicting realistic contact pressure between powders (temperature effect is not considered in the present work), through which the bonding results can be calculated using the theoretical bonding model; however, the RVE model requires less computation time compared to MPFEM, and applies the periodic boundary conditions (PBCs) to avoid the boundary effects, which have arisen in the MPFEM where an entire powder assemblage is employed [27].

Although HCP and FCC structures produced the same results, HCP structure is recommended because it saves computational cost due to the inclusion of smaller number of particles. The regular packing of particles in the present model is practically not true, but the physics gained from such modelling laid the foundation for a deeper insight into the densification process associated with randomly-packed powder assemblage.

### 7.1.4 Multiscale model for predicting bonding time in direct powder forging

The temperature and relative density distribution for a large-scale DPFed component (compared to the miniature component introduced in Chapter 4) are always not uniform, which will lead to a difference in bonding results for distinct locations within the component. Thus, a multiscale model was proposed to predict the bonding time, i.e. critical holding time in terms of a practical DPF process, for a specified location of a DPFed component, which consists of a continuum FE model, the micromechanical (RVE) model and the theoretical bonding model.

The continuum model is built according to the specific shape of the component, of which the main function, in the multiscale model, is to predict the temperature and relative density evolution in terms of a specific area in the FE model (which representing the specified location of the component). The numerical results derived from the continuum model then serve as the input of the RVE model, which is to make sure the RVE model exhibits the same densification behaviour as the continuum material of the specific area does in the FE model. The theoretical bonding model is incorporated in the RVE model using the methodology detailed in Chapter 5. Thus, the bonding time for the RVE model (i.e. the specific area in the continuum FE model or the specified location of the component) can be predicted.
In the present work, the use of the proposed multiscale model was demonstrated via a practical DPF process (for which the continuum model has already been established in a companion project). The bonding times for two specified locations of a DPFed component were predicted; while the temperature effect was ignored in the RVE model and a simplified material model (linear elastic perfectly-plastic model) was used for the powder particle, which might well influence the accuracy of the numerical results. However, the scheme of the multiscale model shows a promising application for determining the critical value of an industrial process parameter, i.e. holding time, through which the mechanical strength of the forged components can be guaranteed.

### 7.2 Suggestions for future work

Several areas related to the present work require further investigations and some suggestions are as follows.

- In the present work, the numerical predictions for FGH96 powders using the proposed theoretical bonding model were experimentally verified at only relatively low pressures, which is limited by the mechanical strength of the graphite tool. In order to increase the reliability of the model, i.e. to validate the model at higher applied pressures, an outer protection could be added to the graphite die, e.g. a capsule made of high-strength material.

  Also, the proposed bonding model can be applied to other powder materials provided that the required inputs are known or can be derived. Thus, the applicability of the model could be further validated.

- As powders produced by different methods would results in various surface morphologies, other micro-void shapes (proposed based on specific powder surface morphologies) can be investigated in terms of the theoretical bonding model.

- A micromechanical (RVE) model was established in this thesis, of which the main function in the multiscale model is to obtain the simulated contact pressure between powders. In order to increase the accuracy of the predicted results, following improvements could be made in terms of the RVE model.
i. The material flow behaviour of a single particle at the forming temperature needs to be obtained. Thus, more realistic material model could be developed for the RVE model.

ii. The temperature effect can be considered in the RVE model. Thus, more practical contact results could be derived through a thermo-mechanical analysis.

iii. The frictional behaviour between powder particles can be involved in the RVE model to obtain more realistic contact results.

iv. To have a more general representation of simulation results, equal-sized spherical particles in the present model can be replaced by those of different sizes and shapes mimicking real powders.

- During an FE simulation, once a node is judged to be bonded, it should be fixed to the opposite surface (i.e. no relative motion is allowed). This will provide more realistic contact situations for the following simulation also increase the reliability of the simulation results; while this is not achieved in the present work. A possible solution could be the use of the user subroutine FRIC of Abaqus, in which a relative motion flag (LM) is built and the flag can be set manually to control the contact condition of two contacting surfaces: ‘0’ represents the relative motion is allowed; ‘1’ means no relative motion is allowed; ‘2’ assumes that frictionless sliding occurs.
References


[78] C.L. Martin, D. Bouvard, G. Delette, Discrete Element Simulations of the


[96] Speciality graphite materials for sintering, (n.d.).


[105] I.R. McColl, J. Ding, S.B. Leen, Finite element simulation and experimental


[118] Z. Xia, F. Ju, K. Sasaki, A general finite element analysis method for balloon


Appendix 1  MATLAB program for the theoretical bonding model

clear;
% 
%========================================== 1. Input Data ==
% 
% $Tc=1120;\%$ Bonding temperature (C)
% $T=Tc+273.15;\%$ Bonding temperature (K), $Tc=Tk-273.15$
% 
% $S=1;\%$ Sign of stress
% $Pi=0;\%$ pressure within the microvoid
% $Pc=100;\%$ contact pressure (MPa)
% 
% The micro-void radius is determined by powder surface
% morphology
% The value below is for %% FGH96 %%
% 
% $a0=0.55*10^{-6};\%$ initial micro-void radius (m)
% $b0P=10*a0;\%$ initial shell outer radius (m)
% 
% Set initial Value 

$bI=b0P;\%$ used in Interface source diffusion model
$b=b0P;\%$ used in Power-law creep model
$a=a0;\%$ used in Power-law creep and Interface source diffusion model

$Pe=Pc;\%$ used in Power-law creep model
$P=Pc;\%$ used in Interface source diffusion model

%========================================== 2. Creep and Diffusion Data for MAR-M200 ==
% 
% Constants 

$k=1.38*10^{-23};\%$ Boltzmann's constant (m^2*kg/s^2/K)
$R=8.314;\%$ Gas constant (J/mol/K)

% Crystallographic and thermal data

$SE=1.71*10^{-6};\%$ Surface energy (MJ/m^2)

$W=1.1*10^{-29};\%$ Atomic volume (m^3)
$bv=2.5*10^{-10};\%$ Burgers vector (m)
$Tm=1700;\%$ Melting temperature (K)
% Modulus %
\[ u_0 = 8 \times 10^4; \]
\[ \% \text{Shear modulus at 300K (MPa)} \]
\[ T_T = -0.5; \]
\[ \% \text{Temperature dependence of shear modulus} \]
\[ u = u_0 \times (1 + (T - 300)/T_m \times T_T); \]
\[ \% \text{Shear modulus at give temperature} \]

% Diffusion %
\[ D_{0v} = 1.6 \times 10^{-4}; \]
\[ \% \text{Volume diffusion pre-exp coefficient (m^2/s)} \]
\[ Q_v = 285 \times 1000; \]
\[ \% \text{Volume diffusion activation energy (J/mol)} \]
\[ D_{0b} = 2.8 \times 10^{-5}; \]
\[ \% \text{Boundary diffusion pre-exp coefficient (m^2/s)} \]
\[ B_L T = 10^{-10}; \]
\[ \% \text{Boundary layer thickness (m)} \]
\[ Q_b = 115 \times 1000; \]
\[ \% \text{Boundary activation energy (J/mol)} \]

\[ D_B = D_{0b} \times \exp(-Q_b/R/T); \]
\[ D_V = D_{0v} \times \exp(-Q_v/R/T); \]

% Power-law Creep %
\[ n_c = 7.7; \]
\[ \% \text{Power law creep exponent} \]
\[ A_p = 5.3 \times 10^{34}; \]
\[ \% \text{Power law creep pre-exp coefficient (s^{-1})} \]
\[ Q_{cr} = 556 \times 1000; \]
\[ \% \text{Power-law creep activation energy (J/mol)} \]
\[ A_A = A_p \times \exp(-Q_{cr}/R/T); \]
\[ \% \]
\[ n_c = 7.2; \]
\[ A_{pc} = 1.5 \times 10^{11}; \]
\[ A_A = A_{pc} \times u \times b v / k / T \times D_{0v} \times \exp(-Q_v / R / T); \]

% 3. Main program %
\[ a = \text{zeros}(1, 1); \]
\[ a_{aa} = \text{zeros}(1, 1); \]
\[ t = \text{zeros}(1, 1); \]
\[ n = 0; \]
\[ t = 0; \]
\[ d a I = -1; \% \text{make the while loop start} \]
\[ d a F = -1; \% \text{make the while loop start} \]
\[ \text{while } a > 0 \&\& d a I < 0 \&\& d a F < 0 \]
\[ n = n + 1; \]
\[ a a(n) = a; \]
\[ a_{aa}(n) = (1 - (a/a_0)^3) \times 100; \% \text{bonding parameter} \]
\[ t t(n) = t; \]
\[ d t = 100; \]
\[ K = -S \times A_A / 2 \times (a^3 b^3 / (b^3 / n c - a^3 / n c)) ^ n c \times (3 / 2 / n c / u \times (P e - P i + 2 \times S E / a)) ^ n c; \]
daP=K/a^2;
%dbP=K/b^2;
daI=-3*W/k/T*2/pi/a/(bI-a)*((P*bI-SE)/(bI-a)-SE/a)*(BLT/2*DB+a*DV)*10^6;
da=daP+daI;
%b=b+dt*dbP;
a=a+dt*da;
t=t+dt;
end
$
$
$================================= END  ==================================$
Appendix 2  Material data for different nickel alloys

<table>
<thead>
<tr>
<th>Material</th>
<th>Structure</th>
<th>Grain size (μm)</th>
<th>Sintering pressure (MPa)</th>
<th>Sintering temperature (°C)</th>
<th>Density (g/cm³)</th>
<th>Porosity (%)</th>
<th>Relative density (%)</th>
<th>Vicker hardness (HV 300)</th>
<th>Vicker hardness (HV 15)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAR-250</td>
<td>G</td>
<td>10</td>
<td>150</td>
<td>1200</td>
<td>7.8</td>
<td>0.2</td>
<td>99.8</td>
<td>300</td>
<td>150</td>
</tr>
<tr>
<td>MAR-A200</td>
<td>G</td>
<td>20</td>
<td>150</td>
<td>1200</td>
<td>7.8</td>
<td>0.2</td>
<td>99.8</td>
<td>300</td>
<td>150</td>
</tr>
</tbody>
</table>

*Note: Data is compiled from various sources and may not be exhaustive.*

---

Figure A2.1 Material data for different nickel alloys [92].
Appendix 3  DEFORM user subroutine
USRUPD

Part of the subroutine USRUPD is shown as below, thorough which a user-defined bonding parameter was calculated for each node during the FE simulation.

C***************************************************************
C
C
C     Self-Built (YW)
C
C     User defined state variable calculations
C
C     Diffusion Bonding Parameter stored in USRN
C
C     USRE is not used in this subroutine.
C
C     USRN(1):Radius of the micro-void (um in postprocessor)
C     USRN(2):Omega (range: from 0 to 1)
C     USRN(3):Sum of the Normal Pressures (MPa)
C
C***************************************************************
C
SUBROUTINE USRSV3

IMPLICIT DOUBLE PRECISION (A-H,O-Z), INTEGER (I-N)

COMMON /USRCTL/ DTK,KOBJ,ISTATUS,KSTEP

COMMON /ELMCOM/ RZE(3,8),URZE(3,8),STSE(6),EPSE(6),EFEPSE,
EFSTSE, TEPSE, RDTYE, TEMPE(8), USRE1(100),
USRE2(100), DTMPE(8), NODEE(6), KELE, KONP

COMMON /ELMCOM3/ TEPS_NE(8), EFEPS_NE(8), DAMG_NE(8),
STS_NE(6,8)

COMMON /ELMCOM4/ YLDTRNE(6)

COMMON /NODCOM/ RZN(3), URZN(3), DRZN(3), TEMPN, DTMPN,
USRN1(100), USRN2(100), KNODE

COMMON /NODCOM3/ EFEPS_NN, TEPS_NN, DAMG_NN, STS_NN(6),
IELMNOD(3)

COMMON /SSTU/ DTMAXC, DUM2(4)

C
C     This global variable stores nodal normal pressures for all
C     objects, and they are extracted in user routine USRMSH.
COMMON /YIWANG/ YWPRESSURE(18018)

Constants defined in this subroutine

Power Law Creep (parameters)

PARAMETER (A=7.42E5)
PARAMETER (u=4.29E4)
PARAMETER (bv=2.56E-10)
PARAMETER (Bk=1.38E-23)
PARAMETER (T=1200)
PARAMETER (Dv=6.2E-5)
PARAMETER (Qv=207E3)
PARAMETER (R=8.314)

PARAMETER (S = 1)
PARAMETER (Pnc = 4.8)
PARAMETER (b0P = 0.5E-6)
PARAMETER (a0 = 0.05E-6)
PARAMETER (Pi = 0)
PARAMETER (SE = 1.75E-6)

Interface Source Diffusion (parameters)

PARAMETER (W = 1.18E-29)
PARAMETER (DV0 = 2E-10)
PARAMETER (DB0 = 5E-10)
PARAMETER (BLT = 1E-10)
PARAMETER (QBI = 104E3)
PARAMETER (QVI = 197E3)

PARAMETER (bI = b0P)
PARAMETER (b = b0P)

AA=A*u*bv/Bk/T*Dv*exp(-Qv/R/T)
DBI=DB0*exp(-QBI/R/T)
DVI=DV0*exp(-QVI/R/T)

User defined parameters for all nodes

IF (ISTATUS.EQ.1.AND.KNODE.GT.0) THEN
Pe=ABS(YWPRESSURE(KNODE))
P=ABS(YWPRESSURE(KNODE))*1E6
USRN1(3)=USRN1(3)+Pe
IF (USRN1(3).EQ.0) THEN
USRN2(1)=a0*1E6
USRN2(2)=0
USRN2(3)=USRN1(3)
ELSE
USRN2(3)=USRN1(3)
IF (USRN1(1).EQ.0) THEN
USRN2(1)=0
USRN2(2)=1
ELSE
    Va = USRN1(1) / 1E6
    y2 = (3 / (2 * Pnc * u) * (Pe - Pi + (2 * SE / Va))) ** Pnc
    y3 = (-S * AA / 2) * ((Va * b) ** 3) / (((b ** (3 / Pnc)) - (Va ** (3 / Pnc))) ** Pnc)
    daP = y3 * y2 / (Va ** 2)
    y1 = (P * bI - SE) / (bI - Va) + SE / Va
    daI = -6 * W / Bk / T / 3.14159265 / Va / (bI - Va) * y1 * (BLT / 2 * DBI + Va * DVI)
    da = daP + daI
    Omega = 1 - Va / a0
C
    IF (Va .LT. 0) THEN
        USRN2(1) = 0
        USRN2(2) = 1
    ELSE
        USRN2(1) = Va * 1E6! (um in postprocessor)
        USRN2(2) = Omega
    END IF
END IF
END IF
C
    DO I = 4, 100
        USRN2(I) = USRN1(I)
    END DO
C
    END IF
C
    IF (ISTATUS .EQ. 1 .AND. KELE .GT. 0) THEN
        DO I = 1, 100
            USRE2(I) = USRE1(I)
        END DO
        RETURN
    END IF
    RETURN
C
    END
C
    ***End of SUBROUTINE USRSV3***
C
C**************************************************************************************
Appendix 4  Abaqus user subroutine FRIC

subroutine fric(lm, tau, ddtddg, ddtddp, dslip, sed, spd, ddtddt, pnewdt, statev, dgam, taulm, press, dpress, ddpddh, slip, kstep, kinc, time, dtime, noel, ciname, slname, mname, npt, node, npatch, coords, rcoord, drot, temp, predef, nfdir, mcrd, npred, nstatv, chrlngth, props, nprops)

include 'aba_param.inc'

c character*80 ciname, slname, msname
dimension tau(nfdir), ddtddg(nfdir, nfdir), ddtddp(nfdir),
dslip(nfdir), ddtddt(nfdir, 2), statev(*),
dgam(nfdir), taulm(nfdir), slip(nfdir), time(2),
coords(mcrd), rcoord(mcrd), drot(2, 2), temp(2),
predef(2, *), props(nprops)

C Parameters defined in this subroutine

C Creep and Diffusion Data

C Material: FGH96 (MAR-M200) (Nickel-based superalloy)

C Input data

PARAMETER (T = 1373.15) ! Bonding temperature (K)
PARAMETER (b0P = 0.2E-6) ! Initial shell outer radius (m)
PARAMETER (a0 = 0.02E-6) ! Initial micro-void radius (m)
PARAMETER (Pi = 0) ! Pressure within the micro-void
PARAMETER (S = 1) ! Sign of stress
PARAMETER (SE = 1.71E-6) ! Surface energy (MJ/m^2)

C Crystallographic and thermal data

PARAMETER (W = 1.1E-29) ! Atomic volume (m^3)
PARAMETER (bv = 2.5E-10) ! Burgers vector (m)
PARAMETER (Tm = 1600) ! Melting temperature (K)

C Constants

PARAMETER (Bk = 1.38E-23) ! Boltzmann's constant
(m^2*kg/s^2/K)
PARAMETER (R = 8.314) ! Gas constant (J/mol/K)
Modulus

PARAMETER (u0 = 8E4)! Shear modulus at 300K (MPa)
PARAMETER (TT = -0.5)! Temperature dependence of shear modulus

Power-law creep

PARAMETER (Pnc = 7.7)! Power law creep exponent
PARAMETER (Ap = 5.3E34)! Power law creep pre-exp coefficient (s^-1)
PARAMETER (Qcr = 556E3)! Power-law creep activation energy (J/mol)

Diffusion

PARAMETER (D0v = 1.6E-4)! Volume diffusion pre-exp coefficient (m^2/s)
PARAMETER (Qv = 285E3)! Volume diffusion activation energy (J/mol)
PARAMETER (D0b = 2.8E-5)! Boundary diffusion pre-exp coefficient (m^2/s)
PARAMETER (BLT = 1E-10)! Boundary layer thickness (m)
PARAMETER (Qb = 115E3)! Boundary activation energy (J/mol)

u=u0*(1+(T-300)/Tm*TT)! Shear modulus

DB=D0b*exp(-Qb/R/T)
DV=D0v*exp(-Qv/R/T)

AA=Ap*exp(-Qcr/R/T)

Define contact property

Set LM equal to 0 if relative motion is allowed (either due to slip or elastic stick).
Set LM equal to 1 if no relative motion is allowed; a rigid sticking condition at the interface is enforced by a Lagrange multiplier method.
C Set LM equal to 2 if friction is ignored (frictionless sliding is assumed).

lm=2

User-defined solution-dependent state variables (for slave nodes in contact)

This subroutine will be called at points on the slave surface of a contact pair and at the integration points in a contact element (only when the contact point is closed) for which the contact interaction property model contains user-subroutine-defined friction

statev(1):Va (ma,a, radius of the micro-void, um in postprocessor)
statev(2):Omega (Diffusion bonding parameter, range from 0 to 1)

Default of initial values of statev(n) are Zero.

Assume there is only one step, and the simulation starts from increment 1

IF (statev(1) .EQ. 0 .AND. statev(2) .EQ. 0) THEN
statev(1)=a0*1E6!(um in postprocessor)
statev(2)=0

END IF

IF (statev(2).EQ.1) THEN
statev(1)=0
statev(2)=1
ELSE
P=ABS(press)! (MPa) Contact pressure at end of increment

y2=(3/(2*Pnc*u)*(P-Pi+(2*SE/Va)))**Pnc
y3=(-S*AA/2)*((Va*b)**3)/(((b**(3/Pnc))-(Va***(3/Pnc)))**Pnc)
daP=y3*y2/(Va**2)
 dbP=y3*y2/(b**2)
C

y1=(P*bI-SE)/(bI-Va)+(SE/Va)
daI=-3*W/Bk/T^2/3.14159265/Va/(bI-Va)*y1*(BLT/2*DB+Va*DV)

da=daP+daI

Va=Va+dtime*da
b=b+dtime*dbP

Omega=1-Va/a0

----------------------------------------

TEMP(2): Current temperature at the slave node and the opposing master surface, respectively.

----------------------------------------

IF (Va.LE.0) THEN
  statev(1)=0
  statev(2)=1
ELSE
  statev(1)=Va*1E6!(um in postprocessor)
  statev(2)=Omega
END IF
END IF

----------------------------------------

When judged bonded, set a sticking condition to this slave node

! IF (statev(2).EQ.1) THEN
  ! lm=1
! ELSE
  ! lm=2
! END IF

----------------------------------------

Write all required information in a text file

OPEN(130,FILE='~/home/yw1012/Hemisphere/Info_temp.TXT')
WRITE(130,*): kstep, kinc, node, coords, press, dtime, statev(1), statev(2)

RETURN
END

C************************** End of the program **************************
C

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