Development of Direct Powder Forging Process

A thesis submitted to Imperial College London for the degree of

Doctor of Philosophy

By

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Declaration of Originality

I declare that this thesis, Development of Direct Powder Forging Process, is entirely my own work under the supervision of Prof. Jianguo Lin. The work was performed in the Department of Mechanical Engineering at Imperial College London. All published and unpublished materials used in this thesis have been given full acknowledgement. This work has not been previously submitted, in whole or in part, to any other academic institution for a degree, diploma, or any other qualifications.

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Abstract

Direct powder forging (DPF) is an innovative powder metallurgy (PM) process that aims to manufacture nickel-based superalloy components within a very short time, compared with current hot isostatic pressing (HIPing) and other processes, by applying high temperature and pressure to metal powders. DPF process has been proposed to reduce the microstructural defects, e.g. prior particle boundaries (PPBs), possessed by the conventional HIPing process and achieve considerably high production efficiency with low cost and energy saving. The aim of this project is to study the powder consolidation and microstructure evolution of a nickel-based superalloy, FGH96, during DPF process. The work in this thesis is divided into two parts: experimental studies and numerical modelling.

The experimental studies reported in this thesis concentrate on the material characterisation of FGH96 and investigate the products of DPF process under different process conditions. Firstly, hot compression tests were conducted using Gleeble 3800 test station to obtain the mechanical properties of FGH96. Secondly, small size powder forging tests were designed to study the powder consolidation. Samples acquired from small size powder forging tests were examined with material density. Tensile tests were then carried out to evaluate the mechanical properties of FGH96 superalloy produced by DPF process, and microstructure observation was used to identify the microstructure features. Lastly, the DPF process was tested under both laboratory and industrial environment. Tests were conducted on a laboratory-based 250 kN hydraulic press machine and an industrial 20,000 kN hydraulic press machine to consolidate FGH96 powder which was encapsulated with a stainless steel container. Material density, hardness and microstructure of the FGH96 components were then examined to evaluate the feasibility of implementing DPF process to manufacture fully dense FGH96 superalloys with desired material properties and microstructure features.

The numerical modelling was used to model the material behaviours of FGH96 and investigate the powder density evolution during DPF process. Firstly, a unified viscoplastic constitutive model was developed for fully dense FGH96 based on the results
obtained from the hot compression tests, and calibration was carried out to achieve good agreement between numerical integrations and experimental results. Secondly, the constitutive model was modified with powder densification parameters and variables, providing a method to numerically describe the powder density evolution during DPF process. Thirdly, the modified constitutive model, i.e. the powder material model, was implemented into the commercial software DEFORM 2D/3D via user defined subroutines. Results were analysed in terms of stress state and powder density distribution, and the correlation between the material properties of DPFed components and process parameters were discussed. Lastly, future work suggestions were proposed to improve the DPF process and modelling technique.
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# Table of Contents

Abstract ......................................................................................................................... 1
Acknowledgements ........................................................................................................ 3
Table of Contents ........................................................................................................... 4
List of Figures ................................................................................................................ 9
List of Tables ................................................................................................................ 14
Nomenclature ............................................................................................................... 15

# Chapter 1 Introduction ......................................................................................... 18
  1.1 Research background ......................................................................................... 18
    1.1.1 Development of high performance materials .................................................. 18
    1.1.1.1 Development of nickel-based superalloys ................................................... 19
    1.1.1.2 Turbine disk ............................................................................................. 20
    1.1.2 Development of powder metallurgy ............................................................... 21
    1.1.3 Hot isostatic pressing .................................................................................. 22
  1.2 Aims and objectives ............................................................................................ 23
  1.3 Outline of the thesis ............................................................................................ 24

# Chapter 2 Literature Review ............................................................................. 25
  2.1 Introduction ......................................................................................................... 25
  2.2 PM technology ................................................................................................... 25
    2.2.1 The global market for PM products, superalloys and nickel-based superalloys in industries ........................................................................................................................................................................................................................................ 26
    2.2.2 Nickel-based superalloys manufactured by PM ............................................ 28
      2.2.2.1 Powder production ................................................................................. 30
      2.2.2.2 Powder consolidation .......................................................................... 34
      2.2.2.3 Characteristics and properties of PM nickel-based superalloys ............ 39
## Table of Contents

2.2.2.4 HIPing map ........................................................................................................................................................................... 44

2.3 Materials modelling ........................................................................................................................................................................ 45

  2.3.1 Dislocations ........................................................................................................................................................................... 46

  2.3.2 Modelling of dislocations ......................................................................................................................................................... 48

2.4 Summary ....................................................................................................................................................................................... 49

### Chapter 3 Experimental Techniques ........................................................................................................................................ 51

  3.1 Introduction .................................................................................................................................................................................. 51

  3.2 Test materials .............................................................................................................................................................................. 51

  3.3 Uniaxial compression tests ......................................................................................................................................................... 52

    3.3.1 Test equipment ..................................................................................................................................................................... 53

    3.3.2 Test preparation and set-up .................................................................................................................................................. 54

    3.3.3 Test operation ....................................................................................................................................................................... 55

  3.4 Small size powder forging .............................................................................................................................................................. 57

    3.4.1 Graphite tool-set .................................................................................................................................................................... 57

    3.4.2 Test preparation and set-up ................................................................................................................................................ 58

    3.4.3 Test operation ....................................................................................................................................................................... 59

  3.5 Laboratory feasibility study .......................................................................................................................................................... 62

    3.5.1 Test equipment and set-up .................................................................................................................................................. 62

    3.5.2 Test-piece preparation ......................................................................................................................................................... 63

    3.5.3 Test operation ....................................................................................................................................................................... 64

  3.6 Industrial trials .............................................................................................................................................................................. 65

    3.6.1 Test equipment and set-up .................................................................................................................................................. 65

    3.6.2 Test-piece preparation ......................................................................................................................................................... 66

    3.6.3 Test operation ....................................................................................................................................................................... 67

  3.7 Examination and analysis of the DPFed samples ......................................................................................................................... 68
3.7.1 Density measurement ................................................................. 68
3.7.2 Hardness test ............................................................................. 68
3.7.3 Tensile test ................................................................................ 69
3.7.4 Microstructure observation ............................................................. 70

**Chapter 4 Experimental Results** ........................................................................ 75

4.1 Introduction ...................................................................................... 75
4.2 Results for high temperature uniaxial compression tests .......... 75
  4.2.1 Stress-strain relationship for stainless steel AISI 304 ............ 75
  4.2.2 Stress-strain relationship for FGH96 ........................................ 77
4.3 Results of small size powder forging tests ........................................ 79
  4.3.1 Mechanical properties test ............................................................ 79
  4.3.2 Microstructure analysis ................................................................. 81
    4.3.2.1 Load holding time of 15 s ....................................................... 83
    4.3.2.2 Load holding time of 20 s ....................................................... 85
    4.3.2.3 Load holding time of 30 s ....................................................... 87
    4.3.2.4 Load holding time of 60 s ....................................................... 89
    4.3.2.5 Load holding time of 100 s ..................................................... 91
    4.3.2.6 Samples after chemical etching .............................................. 93
    4.3.2.7 Samples after electroetching .................................................. 95
4.4 Results of laboratory feasibility study ..................................................... 96
  4.4.1 Consolidation and hardness test .................................................. 96
  4.4.2 Microstructure analysis ............................................................... 97
4.5 Results of industrial trials ................................................................. 100
  4.5.1 Density and microstructure ......................................................... 100
  4.5.2 Grain structure .......................................................................... 101
4.5.3 Grain size distribution ................................................................. 102
4.6 Conclusions .................................................................................. 103

Chapter 5 Materials Models .................................................................. 105
  5.1 Introduction ................................................................................... 105
  5.2 Development of fully dense material model ..................................... 105
    5.2.1 Deformation mechanisms ......................................................... 106
    5.2.2 Unified viscoplastic constitutive model .................................... 107
      5.2.2.1 Flow rule ......................................................................... 107
      5.2.2.2 Dislocation density evolution ......................................... 109
      5.2.2.3 Isotropic hardening .......................................................... 109
      5.2.2.4 Unified viscoplastic constitutive equations for fully-dense material...... 110
    5.2.3 Calibration of the constitutive model ........................................ 111
      5.2.3.1 Constants of the constitutive model for stainless steel AISI 304 ......... 112
      5.2.3.2 Modification of the constitutive model with softening behaviour for HIPed FGH96 ........................................................................................................... 114
      5.2.3.3 Constants of the constitutive model for HIPed FGH96 ................. 115
    5.3 Development of powder material model ........................................ 117
      5.3.1 Densification rate ..................................................................... 117
      5.3.2 The modification of constitutive model with relative powder density .... 118
        5.3.2.1 The modification of fully dense material model ......................... 118
        5.3.2.2 The modification of powder material model ............................. 119
      5.3.3 Calibration of the powder material constitutive model .................. 122
    5.4 Summary ...................................................................................... 125

Chapter 6 FE Simulation ........................................................................ 126
  6.1 Introduction .................................................................................... 126
# Table of Contents

6.2 Implementation of fully dense material model ........................................... 126
6.3 Implementation of powder density evolution ............................................. 130
  6.3.1 Stress distribution .................................................................................. 132
  6.3.2 Material density distribution ................................................................. 134
  6.3.3 Stress triaxiality factor .......................................................................... 135
  6.3.4 Effect of forming speed on powder density .......................................... 137
6.4 Conclusions .................................................................................................. 140

Chapter 7 Conclusions and Future Work Suggestions ........................................ 141
  7.1 Conclusions .................................................................................................. 141
    7.1.1 Conclusions from experimental studies ............................................ 141
    7.1.2 Conclusions from numerical studies ................................................ 142
  7.2 Future work suggestions ............................................................................ 143

References ........................................................................................................ 145
List of Figures

Figure 1.1 A typical gas turbine engine and the turbine disk (Boyce, 2006).......... 21

Figure 2.1 PM products purchased by industrial customers and companies, demonstrated by GKN Plc., a worldwide engineering business company that provides systems and components for manufacturers around the world (GKN, 2015). 27

Figure 2.2 Production of high performance alloys and segmentation of superalloy products by type (Bedder et al., 2013). ......................................................... 28

Figure 2.3 Different types of elements used in an aircraft engine, presented with different colours as: 1) red for nickel; 2) blue for titanium; 3) orange for steel; 4) yellow for aluminium and 5) green for composites (Akca et al., 2015; Gessinger, 1984). ................................................................. 29

Figure 2.4 A general idea of the PM processes.......................................................... 30

Figure 2.5 Schematic of AA method for metal powder production (Hu et al., 2011). ..... 33

Figure 2.6 Schematic of PREP method for metal powder production (Liu et al., 2012). 33

Figure 2.7 Typical morphology of AA powder (Mostafaei et al., 2016)...................... 34

Figure 2.8 Typical morphology of PREP powder (Zhong et al., 2016)........................ 34

Figure 2.9 A schematic and picture of HIPing equipment (Akca et al., 2015).............. 35

Figure 2.10 A typical microstructure of PPBs and PPB networks in PM superalloys (Bai et al., 2015)......................................................................................... 36

Figure 2.11 Pictures of: a) HIF equipment, b) the process monitor, and c) an example of component in HIF process (courtesy of Ladish Co.).................................. 37

Figure 2.12 Pictures of: a) HEX equipment, and b) an example of component processed by HEX process (courtesy of Wyman Gordon). ......................................... 38

Figure 2.13 Aircraft engine applications of HIPed René 95 superalloy (Soares, 2015)... 40

Figure 2.14 Aircraft engine applications of 2nd generation superalloys (Asthana et al., 2006)........................................................................................................ 41

Figure 2.15 Aircraft engine application of FGH96 (courtesy of BIAM)...................... 43

Figure 2.16 The HIPing map and basic concept of DPF process: a) Arzt and Ashby’s HIPing map for a superalloy with a particle diameter of 50 µm at 1200 °C (Arzt et al., 1983); b) the required pressure and time for powder
materials to reach a relative density of 1.0, with red and blue symbols indicating the required process conditions (Bai et al., 2015). ........................................... 45

Figure 2.17 The concept of edge dislocation: a) an edge dislocation in a schematic cubic crystal structure; b) the dislocation has moved one interatomic distance along the slip plane under the action of the shearing force indicated by the arrows; c) the dislocation has reached the edge of the crystal, and a unit amount of slip has been produced (Berdichevsky, 2017). .................................................. 46

Figure 2.18 The relationship between edge and screw dislocations in a 3-D crystal lattice (Smallman et al., 2014). ................................................................. 47

Figure 2.19 The movement of a dislocation through a crystal lattice in a 2-D view under a simple shear deformation situation (Smallman et al., 2014). ...................... 47

Figure 3.1 Gleeble 3800 thermo-mechanical test station. ........................................ 53

Figure 3.2 The geometry and dimensions (mm) of the test-piece and the set-up of uniaxial compression tests. ................................................................. 54

Figure 3.3 Illustration of the 5 stages in uniaxial compression tests. ......................... 56

Figure 3.4 The graphite tool-set for the small size powder forging tests: a) overview of the graphite tool-set; b) longitudinal-section of the graphite tool-set. .............. 58

Figure 3.5 The set-up of the small size powder forging tests: a) set-up; b) illustration of the set-up. ......................................................................................... 59

Figure 3.6 Illustration of the test periods and stages in small size powder forging tests. 61

Figure 3.7 The 250 kN ESH single shot high rate test machine. .................................. 62

Figure 3.8 The set-up of the up-setting test for laboratory feasibility study. ............. 63

Figure 3.9 The geometry and dimensions (mm) of the stainless steel container, and a prepared test-piece coated with glass lubricant. .................................... 64

Figure 3.10 The test equipment and experimental set-up for industrial trails. .......... 66

Figure 3.11 The geometry and dimensions (mm) of the test-piece used in industrial trials. .......................................................................................... 67

Figure 3.12 The hardness test sample and the test surface. The red dot indicates the first test location. .................................................................................. 69

Figure 3.13 The geometry and dimensions (mm) of the ‘dog-bone’ shaped sample and the special grips designed for the small size tensile test. ....................... 69
Figure 3.14 The Instron 5980 floor model universal testing machine. .......................... 70
Figure 3.15 An example of the prepared microstructure observation sample. ............... 71
Figure 3.16 The procedures and machines for sample preparation. ................................. 72
Figure 3.17 The cross section of the forged samples and chosen locations for 
  microstructure observations: a) samples from laboratory feasibility tests; b) 
  samples from industrial trial tests ................................................................. 73
Figure 3.18 The Hitachi S-3400N SEM test machine. .................................................. 74
Figure 4.1 Stress-strain curves for stainless steel AISI 304 at different temperatures with 
  a strain rate of 1.0 s⁻¹ ..................................................................................... 76
Figure 4.2 Stress-strain relationships of stainless steel AISI 304 at 1000 °C with different 
  strain rates ....................................................................................................... 77
Figure 4.3 Stress-strain relationships of FGH96 at different deformation temperatures 
  with a strain rate of 1.0 s⁻¹ ............................................................................ 78
Figure 4.4 Stress-strain relationships of FGH96 at 1000 °C with different strain rates. ... 78
Figure 4.5 Tensile test fracture position: a) normal fracture position; b) fracture at grip 
  position ............................................................................................................. 80
Figure 4.6 Tensile strength results for the 1st group of tensile tests (conducted at 1150 °C, 
  1.1 kN). ........................................................................................................... 81
Figure 4.7 Tensile strength results for the 2nd group of tensile tests (conducted at 1150 °C, 
  2.5 kN). ........................................................................................................... 81
Figure 4.8 Microstructure of the fracture surface of forged FGH96 sample with 1150 °C 
  and load holding time of 15 s: red circle indicates the magnification location; 
  a) and b) are different sides of fracture surface; c) and d) are the magnified 
  pictures for the red circle location with different magnitude. ......................... 84
Figure 4.9 SEM micrograph for samples with load holding time of 20 s. .................... 86
Figure 4.10 SEM micrograph for samples with load holding time of 30 s. .................... 88
Figure 4.11 SEM micrograph for samples with load holding time of 60 s. ................... 90
Figure 4.12 SEM micrograph for samples with load holding time of 100 s. ............... 92
Figure 4.13 SEM observation of forged FGH96 samples after chemical etching with 
  different holding times: a) to e) indicate the different load holding time (15,
20, 30, 60 and 100 s). Red circle indicates the voids found in the SEM image.

Figure 4.14 SEM observation of forged FGH96 samples after electroetching with different holding times: a) to e) indicate the different load holding time (15, 20, 30, 60 and 100 s). Red arrow points out a typical PPB structure. .......................... 94

Figure 4.15 Distribution of HV5 hardness (solid line with square symbols) and relative density (round symbols) with different distances from the centre of the sample. .................................................................................................................. 97

Figure 4.16 Schematic of the sample section and corresponding SEM micrographs of samples prepared by the laboratory feasibility test at different locations. The marked ‘a’ to ‘f’ indicate the chosen locations for the examination of microstructure within the SEM sample. ................................................................. 99

Figure 4.17 Microstructure of (a) HIPed and (b) DPFed FGH96 alloy. .................. 100

Figure 4.18 Microstructure of the feature samples of powder billet after DPF. Colours indicate grain size range. ............................................................................................................................... 101

Figure 4.19 Typical grain size distribution of DPFed FGH96 samples.................... 103

Figure 5.1 Schematic of material behaviour and microstructure evolution during hot deformation processes. A, B, C and D indicate different recrystallisation conditions during the process, while P indicates the yield point of the material. .......................................................................................................................... 107

Figure 5.2 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for stainless steel AISI 304 at strain rate of 1.0 s\(^{-1}\) under different temperatures. ................................................................................................................. 113

Figure 5.3 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for stainless steel AISI 304 at 1000 °C with different strain rates. ......................................................................................................................... 113

Figure 5.4 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for HIPed FGH96 at strain rate of 1.0 s\(^{-1}\) under different temperatures. ................................................................................................................. 116

Figure 5.5 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for HIPed FGH96 at 1000 °C with different strain rates. .... 116
Figure 5.6 A general idea of the cell model. S and T are in-plane compressive loads (Duva et al., 1994).

Figure 5.7 Validation of modified powder material model by calculated data obtained from unmodified constitutive model for FGH96 at 1050 °C with strain rate of 1.0 s\(^{-1}\). \(\rho_0\) is the initial relative powder density.

Figure 5.8 Powder density evolution obtained from modified powder material model at 1050 °C with a strain rate of 1.0 s\(^{-1}\).

Figure 6.1 The communication paths within DEFORM 2D/3D software.

Figure 6.2 The one-element axisymmetric 2D FE model for verification simulation.

Figure 6.3 The strain and stress distributions for the verification simulation at different progression percentages: a) 33%, b) 66%, and c) 100% of the total process progression.

Figure 6.4 Comparison of strain-stress curves between numerical integration data (solid curve) and FE simulation data (symbols) for the fully dense FGH96 with a strain rate of 1.0 s\(^{-1}\) at 1000 °C.

Figure 6.5 The 3D FE model used in the simulation of DPF process.

Figure 6.6 Distribution of stresses for FGH96 powder during DPF process at the progression percentages of: a) 25%, b) 50%, c) 75% and d) 100%. P1 to P5 indicate the locations chosen for analysis.

Figure 6.7 Comparison of material density distribution between experimental data (symbols) and FE simulation data (solid line with symbols).

Figure 6.8 Evolution of stress triaxiality factor, effective strain and relative powder density at five locations P1 to P5 as shown in the figure.

Figure 6.9 FE simulation results of powder density distribution obtained at different forming speed: a) 75 mm/s, b) 100 mm/s, and c) 200 mm/s.
List of Tables

Table 2.1 Comparison of AA and PREP powder.................................................. 32
Table 2.2 Comparison between some processes of PM disk fabrication. ................. 37
Table 2.3 Typical 1st generation nickel-based superalloys and their characteristics ...... 40
Table 2.4 Typical 2nd generation nickel-based superalloys and their characteristics ...... 41
Table 2.5 Characteristics and mechanical properties of FGH96 superalloy. ............... 42
Table 2.6 Typical 3rd generation nickel-based superalloys and their characteristics...... 43
Table 3.1 Chemical composition of FGH96 (Wt%). ............................................. 52
Table 3.2 Uniaxial compression test conditions. ‘√’ indicates the selected test conditions
for FGH96 and ‘○’ represents those for stainless steel AISI 304. ....................... 55
Table 3.3 The small size powder forging test at 1150 °C. ‘√’ indicates the selected test
conditions. ........................................................................................................ 60
Table 4.1 The 1st group of tensile test results (samples prepared at 1150 °C, 1.1 kN). ... 79
Table 4.2 The 2nd group of tensile test results (samples prepared at 1150 °C, 2.5 kN). ... 80
Table 4.3 Statistical grain size data after DPF process. ......................................... 103
Table 5.1 Determined material constants within the unified viscoplastic model for
stainless steel AISI 304. .................................................................................... 112
Table 5.2 Determined material constants within the unified viscoplastic model for HIPed
FGH96. ........................................................................................................... 115
Table 5.3 Determined material constants within the powder material model for HIPed
FGH96. ........................................................................................................... 123
Table 6.1 Correlation between forming speed and process time. .............................. 137
Nomenclature

English Alphabet

- $A$: Material constant
- $B_0$: Material constant ($MPa$)
- $C_0$: Material constant ($s^{-1}$)
- $D$: Material constant
- $E_0$: Material constant ($MPa$)
- $K_0$: Material constant ($MPa$)
- $k_0$: Material constant ($MPa$)
- $m$: Material constant
- $n$: Material constant
- $r_0$: Material constant
- $B$: Temperature-dependent parameter ($MPa$)
- $C$: Temperature-dependent parameter ($s^{-1}$)
- $E$: Temperature-dependent Young’s modulus ($MPa$)
- $K$: Temperature-dependent strengthening coefficient ($MPa$)
- $k$: Temperature-dependent yield stress ($MPa$)
- $r$: Temperature-dependent dislocation parameter
- $a$: Powder density coefficient
- $b$: Powder density coefficient
- $b_v$: Burger’s vector
- $C_{ijkl}$: Stiffness tensor
- $d$: Instantaneous diameter recorded by C-gauge ($mm$)
- $d_0$: Initial diameter ($mm$)
- $K_1, K_2$: Coefficients characterising dislocation evolution
- $K_B$: Boltzmann constant ($m^2 \cdot kg \cdot s^2 \cdot K^{-1}$)
- $l$: Mean free path for dislocation
- $M$: Strain rate hardening exponent
Nomenclature

- \( M_g \) Mobility of grain boundaries
- \( N \) Strain hardening exponent
- \( P_1 \) Strain rate potential for fully dense material
- \( P_2 \) Strain rate potential for powder material
- \( Q_1, Q_2, Q_3, Q_4, Q_5 \) Activation energy \((J \cdot mol^{-1})\)
- \( R \) Isotropic hardening \((MPa)\)
- \( T \) Temperature \((K\) unless stated as °C\)
- \( T_m \) Melting point \((K\) unless stated as °C\)
- \( U_0 \) Activation energy \((J \cdot mol^{-1})\)

Greek Alphabet

- \( \beta \) Material constant
- \( \gamma \) Material constant
- \( \delta_1, \delta_2 \) Material constant
- \( \varepsilon_0 \) Material constant
- \( \sigma_0 \) Material constant
- \( \varphi \) Material constant
- \( \kappa \) Universal gas constant \( (J \cdot mol^{-1} \cdot K^{-1})\)
- \( \delta \) Elongation
- \( \delta_{ij} \) Kronecker delta
- \( \rho \) Relative powder density
- \( \rho_d \) Average dislocation density
- \( \rho_{di} \) Initial dislocation density
- \( \rho_{dm} \) Maximum dislocation density
- \( \bar{\rho} \) Normalised dislocation density
- \( \tau \) Average energy per unit length of a dislocation \((J)\)
- \( \phi \) Reduction of area
- \( \omega \) Mobile dislocation damage
Nomenclature

Stress-related terms

- \( \sigma \)  Stress (MPa)
- \( \sigma_b \)  Tensile strength (MPa)
- \( \sigma_e \)  Effective stress (MPa)
- \( \sigma_m \)  Mean stress (MPa)
- \( \sigma_y \)  Yield strength (MPa)
- \( \sigma_{es} \)  ‘Effective’ effective stress for powder material (MPa)
- \( \sigma_{ij} \)  Stress tensor (MPa)
- \( S_{ij} \)  Stress deviator (MPa)

Strain-related terms

- \( \varepsilon \)  Strain
- \( \varepsilon_e \)  Effective strain
- \( \varepsilon_m \)  Mean strain
- \( \varepsilon_p \)  Plastic strain
- \( \varepsilon_t \)  Total strain
- \( \varepsilon_{ee} \)  ‘Effective’ effective strain for powder material
- \( \varepsilon_{ij} \)  Strain tensor
- \( \varepsilon_{pe} \)  Effective plastic strain
- \( \varepsilon_{ij} \)  Strain deviator
- \( \varepsilon_{dr} \)  Dilatation rate (s\(^{-1}\))
Chapter 1

Introduction

1.1 Research background

The past decades have witnessed a great improvement in the performance of gas turbine engines thanks to the research and development of advanced engine materials. Generally, the parts and components used in a gas turbine engine are exposed to a number of severe service conditions, such as high temperatures, corrosive gases, vibratory and constant stresses, etc. (Boyce, 2006; Sahm et al., 1974). Therefore, the engine materials are required to possess excellent high temperature properties for manufacture, good elevated temperature properties for service conditions, and adequate room temperature properties for maintenance (Rolls-Royce, 1996). This leads to the significant research and development of high performance materials and their process technologies.

1.1.1 Development of high performance materials

In the early stage of aircraft and aerospace industries, steels were the first choice for gas turbine engine materials. Almost all the major engine components, such as disks, shafts, bearings, gears, blades, were made from either austenitic steels or martensitic steels, and steels were accounted for more than 60% of the weight for all engine components (Oaks et al., 1981). However, these components generally exhibited very short service life due to steels’ inadequate creep strength and poor resistance to fatigue and oxidation at high temperatures. Eventually, the use of steels was greatly reduced because of the introduction of other higher performance materials, i.e., titanium alloys and nickel-based superalloys.

In 1952, titanium alloys were first used for the compressor blades and disks of the famous Pratt and Whitney (P&W) J57 engine. Due to the high strength-to-weight ratios of titanium alloys, a weight savings of approximately 200 kg were achieved on a gas turbine engine. Since then, titanium alloys were widely adopted in gas turbine engines (Polmear,
2006). To this day, titanium alloys still make up around 30% of the weight of most modern engines and can be found in the applications of blades and disks in the low and intermediate sections of the compressors of modern jet engines. However, the problems of low strength and lack of resistance to oxidation and creep at elevated temperatures still existed in titanium alloys, which limited the components to be operated below 550 °C (Wu, 1996).

Nickel-based superalloys have been extensively developed during the past 60 years. Because of the outstanding room temperature strength, and adequate strength, excellent resistance to oxidation, corrosion and creep at elevated temperatures, nickel-based superalloys can be used in the hotter parts and components of gas turbine engines, such as turbine blades and disks, at temperatures of over 550 °C, which leads to a tremendous improvement in the performance of modern gas turbine engines (Boyce, 2006; Wu, 1996). There is still great potential in applications of nickel-based superalloys, and significant research and studies are being carried out to further push the limit of modern gas turbine engines (Garibov et al., 2014; Zhang et al., 2013).

1.1.1.1 Development of nickel-based superalloys

The development of nickel-based superalloys has gone through three generations, with the 4th generation being developed at the moment, and each new generation has been developed for the purpose of a good combination of mechanical properties, microstructural characteristics and service temperatures (Wu et al., 2012).

The 1st generation of nickel-based superalloys were developed to meet the increasing demand for improved engine efficiency and performance by using high strength superalloys, such as René 95, IN100, Astroloy, etc. These superalloys usually show considerably high strength levels but fast crack growth rates and relatively low service temperatures, which significantly impacts the service time of the gas turbine engine. As a result, the 2nd generation of nickel-based superalloys has been proposed, incorporating the concept of damage tolerance and aiming at developing superalloys with a combination of adequate yield strengths, lower crack growth rates and higher service
temperatures. The representative superalloys of 2\textsuperscript{nd} generation include N18, René 88DT, PM U720 LI and FGH96. The 3\textsuperscript{rd} generation of nickel-based superalloys has been developed to combine the advantages of both 1\textsuperscript{st} and 2\textsuperscript{nd} generation superalloys, and to further push the service temperatures. René 104, Alloy 10 and RR1000 can be categorised as 3\textsuperscript{rd} generation superalloys (Voice et al., 2004).

1.1.1.2 Turbine disk

Figure 1.1 shows a typical gas turbine engine and the turbine disk (Boyce, 2006). Turbine disk is one the key parts of a gas turbine engine that is responsible for holding the turbine blades in place, and is subjected to centrifugal force associated with rotation. There also exists a temperature gradient from the bore to the rim of the disk. High tensile strength, damage tolerance and creep resistance are all required mechanical properties for a turbine disk. In the bore of the disk, there are high stresses in the order of 1000 MPa but relatively low temperatures. At this position, it is desirable to achieve good tensile strength. The rim experiences lower rotational forces but much higher temperatures, so good creep properties are needed (Rolls-Royce, 1996). Although austenitic steels were first used in the turbine disks, nickel-based superalloys were then adopted and are still the only materials for turbine disks for their excellent mechanical properties that are required for modern gas turbine engines.
1.1.2 Development of powder metallurgy

Powder metallurgy (PM) is a materials processing technology that produces components by consolidating powdered materials to a certain shape and size. The demand for PM in applications of manufacturing critical gas turbine engine components with nickel-based superalloys, especially turbine disks, has emerged in the 1960s and 1970s when nickel-
based superalloys, such as Astroloy, René 95, MERL 76, etc., were found to be difficult to be produced via the ingot-route. Since then, PM of nickel-based superalloys has been widely studied and utilised in the aircraft and aerospace industries (James, 2015). At first, PM products were limited to engine applications for military purposes, for example, the Pratt & Whitney manufactured 11 parts and components with IN100 nickel-based superalloy, including compressor and turbine disks, to be used for the F100 engine for the F-15 and F-16 fighters in 1972. With the development of PM technologies, PM products were then applied to commercial aircrafts, such as CF6-80 engine manufactured by General Electric (GE) for the Boeing 747-400 and the GE90 for the Boeing 777. Nowadays, PM is the standard procedure of producing gas turbine engine components and becomes a critical technology in the modern aircraft and aerospace industries.

1.1.3 Hot isostatic pressing

In modern PM industries, hot isostatic pressing (HIPing) is the dominant processing technology to manufacture turbine disks, which can produce net-shape or near net-shape components. Compared to conventional casting process, HIPing can produce components without macrosegregation and casting defects, and provide the components with homogeneous microstructure and fine grain size, thus improving mechanical properties (Maurer et al., 1996). On the other hand, there are still some critical issues to be considered, especially prior particle boundaries (PPBs), which greatly affect the service life of HIPed components and limit the use of HIPing process in PM industries.

PPBs can be formed during powder production or HIPing process on the powder particle surface and has been the major issue of HIPed nickel-based superalloys which hinder the bonding between powder particles and become the sites for crack initiation and growth under loading conditions. This becomes more apparent at elevated temperatures and leads to significant degradation of high temperature tensile ductility, stress creep rupture properties and service life of the HIPed parts. Massive efforts have been carried out to eliminate or decrease PPBs, such as improving the powder production process of the raw materials to reduce the oxygen and carbon contamination, adjusting the process.
parameters to prevent PPBs from forming, and applying post-HIP procedures to break PPBs into the matrix. From the forming process point of view, adjusting process parameters may result in abnormal microstructural evolution and lead to undesirable mechanical properties of the components, while post-HIP procedures will inevitably increase the process cost and impact the production efficiency. Accordingly, a new powder forming process, i.e. the direct powder forging (DPF) process has been proposed to change the current situation of HIPing process (Bai et al., 2015).

1.2 Aims and objectives

As a 2nd generation superalloy, FGH96 has been well developed and widely used in the manufacture of turbine disks using HIPing process (Zhang et al., 2012). In this research, FGH96 has been chosen as the studied material for its good fabrication characteristics, strength, damage tolerance and durability at elevated temperatures, which makes it a viable choice to be implemented into DPF process for further investigation.

The aim of this research is to develop the DPF technique for industrial applications and study the powder density evolution of FGH96 powder during hot deformation process. To achieve this goal, five objectives are proposed as follow:

1) To identify the material characteristics and mechanical properties of FGH96 superalloy;
2) To study the effects of process parameters, e.g. strain rate, temperature, deformation time, etc., on the consolidation of FGH96 powder;
3) To establish the constitutive model for FGH96 powder during hot deformation process and model the powder density evolution;
4) To investigate the processing of FGH96 components using DPF technique in both laboratory and industrial situations;
5) To establish finite element model for the DPF process and optimise processing parameters.
1.3 Outline of the thesis

The outline of this thesis is organised as follow:

1) Chapter 1 introduces the background of this research and illustrates the aims and objectives of the thesis;
2) Chapter 2 presents a literature review on PM technologies and products, including powder production techniques, process techniques and material characteristics and mechanical properties of the PM superalloys;
3) Chapter 3 introduces the experimental methods used in this research, including the characterisation of the material by hot compression tests, the investigation of process parameters, the feasibility study of DPF test in laboratory and industry, and microstructural observation techniques;
4) Chapter 4 contains the detailed experimental results obtained from the experimental procedures described in Chapter 3 and analyses the features and discoveries of the results;
5) Chapter 5 establishes both the basic constitutive model for fully dense materials and the modified model for porous powder compact, with particular attention on powder density evolution. Materials constants are determined from experimental data;
6) Chapter 6 demonstrates implementation of the constitutive model for FGH96 powder, and the results of FE simulations are discussed;
7) Chapter 7 summarises the main conclusions from this research and proposes suggestions for future works.
Chapter 2

Literature Review

2.1 Introduction

This chapter reviews the development of PM technologies in industries and their applications, as well as typical PM. This chapter contains the following content:

1) Analysis of the global market of PM: an analysis of the global PM market is presented to gain a better understanding of the rapid development of PM technologies in industries;

2) Review of typical PM technologies: powder production, HIPing and HIP-based processes, properties of PM superalloys, and the effects of HIPing pressure and time on relative powder density are reviewed and discussed to provide a background for the development of DPF process;

3) Review of materials modelling: the mechanism for material behaviours during deformation, i.e. dislocation evolution, is identified and reviewed, and a dislocation density evolution model is presented for further development of viscoplastic constitutive model of FGH96.

2.2 PM technology

PM is a materials processing technology to manufacture components and parts by using powdered materials as raw ingredients. A wide range of products can be manufactured by PM as it is particularly outstanding in forming the following types of materials (Krsjak et al., 2014; Nadakudurua et al., 2011; Yang et al., 2013; Zabihi et al., 2013):

1) Alloys created from high melting point metals;

2) Composites made of metal and non-metal materials;

3) Composites formed by metals that do not dissolve into each other;

4) Porous materials.
PM can manufacture net-shape and near net-shape products, avoiding severe segregation associated with melting and solidification and achieving lower cost through a reduction of machining and welding costs and high usage of raw materials. In addition, mechanical properties of alloys are significantly improved as the desired microstructure can be achieved by adjusting the composition of powders and forming conditions. Furthermore, high economic efficiency is expected with PM processes since low cost and little waste of raw material can be achieved by optimising process parameters and procedures during the production (Gentzbittel et al., 2002; Zhang et al., 2013).

2.2.1 The global market for PM products, superalloys and nickel-based superalloys in industries

PM technologies are now widely used in various industries to manufacture high performance alloys and at the same time to achieve excellent economic efficiency. Recent studies show that over 90% of PM products are used in the transport market, including automobile, aircraft and aerospace industries, and the adoption of next generation applications in PM is strongly required in growing markets, such as information home appliances, sustainable energy and life science (McWilliams, 2016). Figure 2.1 demonstrates an example of the global market of PM products, featuring several famous industrial customers and companies (GKN, 2015). The global demand leads to a positive compound annual growth rate (CAGR) of PM product market at around 5%, and the market is estimated to reach 8.7 billion USD by 2020.
Figure 2.1 PM products purchased by industrial customers and companies, demonstrated by GKN Plc., a worldwide engineering business company that provides systems and components for manufacturers around the world (GKN, 2015).

Superalloys are a type of metallic alloys that exhibit excellent strength, creep resistance, significant surface stability, and corrosion resistance at high temperature. Such astonishing properties make superalloys the excellent candidates for aircraft applications, especially for aircraft engines. According to Rolls-Royce Civil Aerospace, one of the largest manufacturers of jet engines, the global market, which covers passenger and cargo jets, corporate and regional aircraft, will require 149,000 engines to be delivered over the 20-year period of 2012-2031, worth around 975 billion USD, and these engines will be required to power 68,000 commercial aircraft and business jets (INSG, 2013; Williams, 2012). This high demand for aircraft engines leads to the superalloys being one of the major segments of high performance alloy products, accounting for around 35% in 2012 and retaining its proportion in the following years.

The base alloying elements used for superalloys are nickel, cobalt and iron, among which nickel is the most popular base element in aircraft industries for the manufacture of turbine disks and blades. The reason behind such a high popularity is that nickel-based superalloys are resistant to extremely high temperatures and physical stress, thus, they can be utilised for the high-temperature strength, toughness, and resistant to degradation
in oxidising and corrosive environments (Akca et al., 2015). Therefore, nickel-based superalloys take a dominant place in the superalloy market, as shown in Figure 2.2, and will remain as the most demanded superalloys in aircraft industries.

![Figure 2.2 Production of high performance alloys and segmentation of superalloy products by type (Bedder et al., 2013).](image)

### 2.2.2 Nickel-based superalloys manufactured by PM

Nickel-based superalloys are the most complex of all superalloys and have been widely used for some of the hottest parts and components in aircraft industries, as demonstrated in Figure 2.3 by red colour, contributing over 50% of the weight to aircraft engines (Akca et al., 2015; Gessinger, 1984). The principal characteristics of nickel as an alloy base are the high phase stability of FCC nickel matrix and the capability to be reinforced by a variety of direct and indirect methods. Further, the surface stability of nickel-based superalloys is readily improved by alloying with chromium and aluminium (Qiu et al., 2013). Currently, there are 3 generations of nickel-based superalloys that have been developed to serve specific purposes, with each generation tending to have higher
temperature resistance, and the 4th generation is under significant research, incorporating more expensive alloying metals, such as rhenium and ruthenium, to achieve the desired properties.

Figure 2.3 Different types of elements used in an aircraft engine, presented with different colours as: 1) red for nickel; 2) blue for titanium; 3) orange for steel; 4) yellow for aluminium and 5) green for composites (Akca et al., 2015; Gessinger, 1984).

Apart from the material characteristics of nickel, the presence of the reinforcement $\gamma'$ ($\text{Ni}_3\text{Al}$) phase also contributes to the exceptional material properties of nickel-based superalloys. $\gamma'$ precipitates are coherent and remain stable to relatively high temperatures, thus improve the high-temperature performance of nickel-based superalloys (Sarosi et al., 2007; Tang et al., 2009). In the mid-1960s, alloy designers focused on increasing the volume fraction of $\gamma'$ to increase high-temperature strength, so nickel-based superalloys with the $\gamma'$ volume fraction of 60% were very common. Unfortunately, increasing the $\gamma'$ volume fraction often brought the issues of increased macrosegregation in large ingot castings and caused a large drop in the service life of superalloys. As a result, PM was then proposed as the solution to the macrosegregation problems (Tian et al., 2006).

Typical PM processes can be divided into 3 stages, as shown in Figure 2.4:
1) Powder preparation: the powder is prepared from an original material for PM processes by powder production process and examined for its characterisations and microstructure;

2) Consolidation: the powder is encapsulated as a pre-form, then the pre-form is delivered to the compacting and sintering procedure, followed by optional secondary operations to acquire specific properties;

3) Properties: the component is examined for its densification conditions, mechanical properties and other physical properties.

![Figure 2.4 A general idea of the PM processes.](image)

### 2.2.2.1 Powder production

There are two commonly used methods for the production of nickel-based superalloy powders in industries (Hu et al., 2013; Zhong et al., 2016):

1) Argon atomisation (AA);

2) Plasma rotating electrode process (PREP).
AA method involves a melted liquid metal being injected into a large chamber where the liquid metal is atomised into powder droplets under the high pressure caused by full-filled argon gas. AA could obtain rapidly solidified metal powder with fine particle sizes and high purity, but the sphericity of the metal powder is poor, and normally the argon gas cannot be fully eliminated, which results in the formation of hollow powder (Hu et al., 2011; Zhong et al., 2016).

PREP method involves a rotating consumable electrode which is made of the original material, and the rotating electrode is protected by inert gas, e.g. argon gas, and melted by plasma arc from the electrode tip. Liquid metal droplets jet off because of the centrifugal force and are atomised into powders. Compared to AA powder, PREP powder has better sphericity and no hollows, but particles are coarser and inclusions may be found within the prepared powder (Liu et al., 2012; Zhong et al., 2016).

Table 2.1 compares the properties of metal powders produced by the AA and PREP methods. Figure 2.5 shows the general process of AA and Figure 2.7 presents a typical morphology of AA powder, while Figure 2.6 demonstrates the PREP procedures and Figure 2.8 shows a typical morphology of PREP powder.
Table 2.1 Comparison of AA and PREP powder.

<table>
<thead>
<tr>
<th>Method</th>
<th>Property</th>
<th>AA</th>
<th>PREP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle</td>
<td>Poor sphericity,</td>
<td>Good sphericity,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>hollow powder in presence</td>
<td>no hollows</td>
<td></td>
</tr>
<tr>
<td>Particle size</td>
<td>Finer,</td>
<td>Coarser,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>average &lt; 50 μm</td>
<td>average &gt; 50 μm</td>
<td></td>
</tr>
<tr>
<td>Purity</td>
<td>Very small amount of inclusions</td>
<td>Large inclusions may be in</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>presence</td>
<td></td>
</tr>
<tr>
<td>Gas pollution</td>
<td>High (argon gas)</td>
<td>Very low</td>
<td></td>
</tr>
<tr>
<td>Powder</td>
<td>Usage of material is high,</td>
<td>Usage of material is low,</td>
<td></td>
</tr>
<tr>
<td>collection</td>
<td>more powder is collected</td>
<td>less powder is collected</td>
<td></td>
</tr>
</tbody>
</table>
Figure 2.5 Schematic of AA method for metal powder production (Hu et al., 2011).

Figure 2.6 Schematic of PREP method for metal powder production (Liu et al., 2012).
2.2.2.2 Powder consolidation

Generally, after the powder production process, the prepared powder is encapsulated into a container with a pre-defined shape and then delivered to the powder consolidation process (Ekbom, 1990).

Typically, HIPing is the most commonly used process for the manufacture of nickel-based superalloy by consolidating metal powder into a specific shape under high temperature and a certain level of pressure. Nevertheless, components manufactured by HIPing usually cannot be used directly and are processed by follow-up procedures to achieve desired properties (Pollock et al., 2006). Therefore, based on HIPing process, several powder consolidation processes have been developed to serve different purposes:
1) HIPing;
2) HIPing followed by hot isothermal forging (HIP+HIF);
3) HIPing followed by hot extrusion and hot isothermal forging (HIP+HEX+HIF).

HIPing process starts with heating the metal powder to a high temperature under a certain level of hydrostatic pressure. After a certain period of time, sintering and diffusion bonding of the powder under pressure produce a full dense superalloy component (Dellis et al., 1996; Epishin et al., 2013). A schematic and picture of equipment used during HIPing process are presented in Figure 2.9. Compared with HIP+HIF and HIP+HEX+HIF, HIPing creates near net-shaping products at a relatively low cost and high efficiency, and the absence of follow-up procedures leads to a relatively high usage of the original metal powder and avoids massive waste of the material (Bampton, 2005; Rezaie et al., 2017). However, a major issue found in HIPed nickel-based superalloy components is that PPBs and PPB networks are present within the final components due to long exposure at high temperature (Alniak et al., 2006). Many studies and researches suggest that PPBs and PPB networks are detrimental to nickel-based superalloys by forming undesired networks around powder particles, providing an easy fracture path that results in the failure of the components and leading to a weakening of strength, hardness and other properties (Buckingham et al., 2016; Ning et al., 2012). Figure 2.10 shows a typical microstructure of PPBs and PPB networks in PM superalloys.

![A schematic and picture of HIPing equipment](Akca et al., 2015)
Therefore, HIP+HIF and HIP+HEX+HIF are developed to compensate the insufficient deformation provided by HIPing and break PPB networks formed during the process by applying follow-up procedures, such as HEX and HIF, hence improving the mechanical properties (Galindo-Nava et al., 2015; Mignanelli et al., 2015). In addition, large deformation provides a better environment for recrystallisation, hence grain size of the HIPed component can be refined, improving strength and other related mechanical properties (Liu et al., 2014). On the other hand, the introduction of the follow-up procedures significantly increases the cost of manufacturing and influences the efficiency of production. A summary of the HIP-based powder consolidation processes is presented in Table 2.2. Figure 2.11 and 2.12 illustrate the HIF and HEX equipment and examples of component in process.
Table 2.2 Comparison between some processes of PM disk fabrication.

<table>
<thead>
<tr>
<th>Process</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>HIPing</td>
<td>Simple procedures, lower cost, higher efficiency</td>
<td>Insufficient deformation, PPBs in presence</td>
<td>Coarser grains</td>
</tr>
<tr>
<td>HIP+HIF</td>
<td>More deformation, elimination of PPBs</td>
<td>Higher cost, small amount of PPBs in presence</td>
<td>Refined grains, more recrystallisation</td>
</tr>
<tr>
<td>HIP+HEX+HIF</td>
<td>Large deformation, complete elimination of PPBs</td>
<td>Highest cost, lowest efficiency, complex procedures</td>
<td>Refined grains, complete recrystallisation</td>
</tr>
</tbody>
</table>

Figure 2.11 Pictures of: a) HIF equipment, b) the process monitor, and c) an example of component in HIF process (courtesy of Ladish Co.).
Despite that HIPing and HIP-based consolidation processes are well developed in industries, there is still room for improvement of these consolidation processes, especially for HIPing. HIPing takes relatively long time to create the final components and high energy cost is reported due to the powder material being maintained at high temperature for hours, leading to the appearance of PPB networks, while other processes contain multiple procedures and have a significantly higher manufacture cost (Liu et al., 2016). Therefore, a new process is required to improve the energy consumption, productivity and efficiency by reducing process procedures, as well as PPBs and recrystallisation issues.
2.2.2.3 Characteristics and properties of PM nickel-based superalloys

As mentioned in the previous section, there are currently 3 generations of PM nickel-based superalloys that have been developed and adopted in industrial applications. Based on the characteristics and properties, they can be categorised as follow:

1) The 1\textsuperscript{st} generation nickel-based superalloys: high strength superalloys, represented by René 95 (General Electric Company (GE), USA);

2) The 2\textsuperscript{nd} generation nickel-based superalloys: damage tolerance superalloys, represented by N18 (Safran Aircraft Engines (SNECMA), France). The studied material in this research is categorised as the 2\textsuperscript{nd} generation of nickel-based superalloy;

3) The 3\textsuperscript{rd} generation nickel-based superalloys: high strength and damage tolerance superalloys, represented by RR1000 (Rolls-Royce (R-R), UK).

The 1\textsuperscript{st} generation nickel-based superalloys exhibit the features of high strength and relatively low working temperature at below 650 °C. This is achieved by a high volume fraction of reinforcement $\gamma'$ phase (45% or higher) and highly refined grains (Singh et al., 2013). On the other hand, this type of superalloys has a relatively low resistance to crack propagation and durability, which is not preferred in the manufacture of large turbine disks. Table 2.3 shows a few typical 1\textsuperscript{st} generation nickel-based superalloys and their characteristics and Figure 2.13 shows examples of the applications on aircraft engines.
Based on the 1\textsuperscript{st} generation superalloys, the 2\textsuperscript{nd} generation of nickel-based superalloys have been developed to improve the resistance to crack propagation and durability, and this type of nickel-based superalloys is categorised as damage tolerance superalloys that feature a lower tensile strength but can be used at higher temperatures ranged from 650 °C to 750 °C, with improved service time (Nie et al., 2014; Xu et al., 2012). The volume fraction of reinforcement $\gamma'$ phase has been decreased to around 45\% and the grain size is larger than that of 1\textsuperscript{st} generation superalloys. Typical 2\textsuperscript{nd} generation nickel-based superalloys are presented in Table 2.4 and an example of the applications on
aircraft engines is shown in Figure 2.14. A representative for 2\textsuperscript{nd} generation superalloys is the René 88DT superalloy. Compared to René 95 superalloy, the tensile strength of René 88DT has been decreased by 10\%, but the propagation rate of cracks has been lowered by around 50\%, with an increased working temperature from 650 °C to 750 °C, which is a huge improvement over René 95 and greatly preferred for aircraft engines (MacSleyne et al., 2009).

<table>
<thead>
<tr>
<th>Superalloy</th>
<th>Manufacturer</th>
<th>(\gamma') phase</th>
<th>Density (g/cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>René 88DT</td>
<td>GE, USA</td>
<td>40</td>
<td>8.33</td>
</tr>
<tr>
<td>N18</td>
<td>SNECMA, France</td>
<td>55</td>
<td>8.00</td>
</tr>
<tr>
<td>PM U720LI</td>
<td>SMC, USA</td>
<td>46</td>
<td>8.13</td>
</tr>
</tbody>
</table>

FGH96 is categorised as a 2\textsuperscript{nd} generation superalloy and exhibits similar characteristics and mechanical properties to René 88DT (Zhang et al., 2011). The characteristics and
mechanical properties of FGH96 superalloy are listed in Table 2.5, and a picture of turbine disk made of FGH96 is presented in Figure 2.15.

<table>
<thead>
<tr>
<th>Superalloy</th>
<th>Manufacturer</th>
<th>$\gamma'$ phase</th>
<th>Density (g/cm$^3$)</th>
<th>Tensile properties</th>
<th>Stress rupture</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Volume fraction (%)</td>
<td>Solvus temperature (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2nd generation</td>
<td>BIAM, China</td>
<td>40</td>
<td>1130</td>
<td></td>
<td></td>
</tr>
<tr>
<td>nickel-based</td>
<td></td>
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<tr>
<td>superalloy</td>
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<tr>
<td>FGH96</td>
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<tr>
<td><strong>Temperature</strong></td>
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<tr>
<td>Room Temperature</td>
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<tr>
<td><strong>Yield strength</strong> &amp; <strong>Tensile strength</strong></td>
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<tr>
<td><strong>Elongation</strong></td>
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<tr>
<td><strong>Reduction of Area</strong></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td><strong>Working temperature</strong> &amp; <strong>Working Stress</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Service time</strong></td>
<td></td>
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</tbody>
</table>

Table 2.5 Characteristics and mechanical properties of FGH96 superalloy.
The 3\textsuperscript{rd} generation of nickel-based superalloys is developed to combine the advantages of both 1\textsuperscript{st} and 2\textsuperscript{nd} generation superalloys, aiming at creating better superalloys to meet higher requirements for the advanced aircraft engines. The 3\textsuperscript{rd} generation superalloys inherit the high strength features from 1\textsuperscript{st} generation superalloys and the better damage tolerance features from 2\textsuperscript{nd} generation superalloys, at the same time increasing the working temperature to up to 800 °C (Detrois et al., 2015). The volume fraction of reinforcement $\gamma'$ phase has been controlled between 45% and 55%, and the grain size can be adjusted according to specific requirements. Table 2.6 summarises the typical 3\textsuperscript{rd} generation nickel-based superalloys.

<table>
<thead>
<tr>
<th>Superalloy</th>
<th>Manufacturer</th>
<th>$\gamma'$ phase</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>René 104</td>
<td>GE, P&amp;W, NASA GRC, USA</td>
<td>50</td>
<td>1155</td>
</tr>
<tr>
<td>Alloy 10</td>
<td>Honeywell, USA</td>
<td>55</td>
<td>1180</td>
</tr>
<tr>
<td>LSHR</td>
<td>NASA GRC, USA</td>
<td>55</td>
<td>1160</td>
</tr>
<tr>
<td>RR1000</td>
<td>R-R, UK</td>
<td>46</td>
<td>1146</td>
</tr>
</tbody>
</table>
2.2.2.4 HIPing map

HIPing process is the dominant technology of producing FGH96 superalloy components, but it possesses productivity and cost issues, as well as PPB networks generated alongside the grain boundary during HIPing process, which provides an easy fracture path and is believed to be detrimental to mechanical properties of FGH96 superalloy.

The productivity and cost issues can be alleviated by optimising HIPing pressure and time. Many researches have been focusing on the relationship between HIPing pressure and time to process completion (DeLo et al., 1999; Kim et al., 2009; Zhou et al., 2013). Arzt and Ashby studied the effects of pressure and time on the powder densification for superalloys (Arzt et al., 1983). It is reported that the deformation mechanisms would change with the increasing of HIPing pressure, from diffusion at low pressure, to power-law creep at intermediate pressure, and then to yield at high pressure. As the HIPing pressure increases, less time is needed for the powder to achieve full density. The relationship between HIPing pressure and relative powder density with process time can be described with a HIPing map, as demonstrated in Figure 2.16(a).

Based on the understanding of Arzt and Ashby’s HIPing map, a diagram has been created to elaborate the basic concept of DPF process, as shown in Figure 2.16(b). The red and blue symbols indicate the required pressure and time for the relative powder density to reach 1.0, which can be acquired from the HIPing map (Bai et al., 2015). Therefore, if the pressure is high enough, the process time for obtaining a fully dense material could be significantly reduced following a logarithmic form.
2.3 Materials modelling

Material behaviours are usually modelled with constitutive equations. Numerous attempts have been made to model the microstructure evolution in materials during hot deformation (Cao et al., 2008; Dixit et al., 2011; Lin, 2003; Lin et al., 2005a; Lin et al., 2017). It is believed that the dislocation structure developed during plastic deformation is the driving force for microstructural changes, e.g. recrystallisation, recovery, grain growth, etc., and the mechanical properties of materials are dominated by the mobility and multiplication of dislocations. Therefore, an understanding of the dislocations is crucial to the development of constitutive model and the modelling of material behaviours. However, it is worth noticing that an accurate modelling of microstructure evolution and material behaviours requires a thorough understanding of all phenomena occurring during the hot deformation, and these are complicated and difficult to model. As a result, most constitutive models are empirical and specific, and can be used in limited situations.
2.3.1 Dislocations

There are two types of dislocations, i.e. edge dislocations which move by slip and climb, and screw dislocations which move by slip and cross slip (Hu et al., 2017; Smallman et al., 2014; Stricker et al., 2015). Typically, the mobility of dislocations is affected by obstacles, such as other dislocations, grain boundaries, and precipitates. Figure 2.17 and 2.18 present the two types of dislocations, and Figure 2.19 shows an example of the movement of dislocations (Smallman et al., 2014). The movement of dislocations determines the material behaviours, and the strain fields around the dislocations control the mobility and multiplication of dislocations. The strain fields serve to impede the dislocation movement, for example, two edge dislocations with the same sign and slip plane are moved apart by their strain fields, but dislocations developed on the same slip plane with opposite signs will be drawn together and annihilated.

![Image of dislocations](image_url)

**Figure 2.17** The concept of edge dislocation: a) an edge dislocation in a schematic cubic crystal structure; b) the dislocation has moved one interatomic distance along the slip plane under the action of the shearing force indicated by the arrows; c) the dislocation has reached the edge of the crystal, and a unit amount of slip has been produced (Berdichevsky, 2017).
Figure 2.18 The relationship between edge and screw dislocations in a 3-D crystal lattice (Smallman et al., 2014).

Figure 2.19 The movement of a dislocation through a crystal lattice in a 2-D view under a simple shear deformation situation (Smallman et al., 2014).

For hot deformations ($T > 0.5T_m$), the deformation is highly temperature and strain rate dependent. At high temperatures, the thermal vibration of the lattice increases, thus the strength of obstacles to prevent dislocation movements decreases, and dislocations are generated at a faster rate (Carrick, 2009). The dislocations will glide, multiply and pile up into tangles, which results in an increase in the stress needed to maintain plastic flow.
Since more dislocations are generated and the dislocation density rises, the annihilation of dislocations becomes more effective. The flow stress continues to increase until the dislocation annihilating rate and dislocation generating rate are balanced, where the flow stress ceases to increase and the deformation proceeds at a steady state. The strain rate will also impact the annihilation and generation of dislocations (Carrick, 2009). The annihilation of dislocations is relatively rapid at lower strain rates and the steady-state of deformation is achieved at a larger subgrain size and lower flow stress, while the generation of dislocations is more effective and the steady-state of deformation is reached at a smaller subgrain size and higher flow stress (Ashby, 1972).

2.3.2 Modelling of dislocations

The modelling of dislocation evolution is usually by modelling the average dislocation density, $\rho_d$, with the consideration of strain hardening, and the static and dynamic recovery of dislocations. The dislocation density rate, neglecting recrystallisation, can be expressed as (Sandström et al., 1975):

$$\dot{\rho}_d = \frac{\dot{\varepsilon}_p}{b_v l} - 2M_g \tau \rho_d^2$$  \hspace{1cm} (2.1)

where $\dot{\varepsilon}_p$ is the plastic strain rate, $b_v$ the Burger’s vector, $l$ the mean free path for dislocation, $M_g$ the mobility of grain boundaries and $\tau$ the average energy per unit length of a dislocation. The first term $\frac{\dot{\varepsilon}_p}{b_v l}$ indicates the development of dislocations and the second term $2M_g \tau \rho_d^2$ describes the static recovery due to annihilation at high temperatures. By considering the dynamic recovery, the dislocation density model can be expressed as (Djaic et al., 1972):

$$\frac{d\rho_d}{d\varepsilon_p} = K_1 \rho_d^{0.5} - K_2 \rho_d$$  \hspace{1cm} (2.2)

where $K_1$ and $K_2$ are coefficients that characterise the dislocation storage and annihilation respectively. In particular, $K_2$ represents a thermally activated process of dynamic
recovery by dislocation cross slip (at low temperature) or dislocation climb (at high temperature). By combining both recovery models, the dislocation density evolution model is described as (Estrin, 1998):

\[
\dot{\rho}_d = (K_1 \rho_d^{0.5} - K_2 \rho_d) |\dot{\varepsilon}_p| - r 
\]  

(2.3)

where

\[
r = r_0 \exp \left( \frac{-U_0}{K_B T} \right) \sinh \left( \frac{\beta \rho_d^{0.5}}{K_B T} \right)
\]  

(2.4)

as \(r_0\) and \(\beta\) are material constants, and \(T\) is temperature. \(K_B\) represents the Boltzmann constant, while \(U_0\) stands for the activation energy. Equations 2.3 can be used to model the dislocation density evolution throughout the deformation process. Based on the dislocation density model, the constitutive model for nickel-based superalloys can be developed and expressed in terms of dislocation evolution, which will be discussed in Chapter 5.

2.4 Summary

The global market for PM technologies and products have been reviewed in this chapter. For a better understanding of the rapid development of PM technologies in industries, the global demand for PM products and superalloys has been analysed. It has been concluded that the high demand for PM products and superalloys has been a result of the rapid growing of aircraft engine applications which require a large amount of PM products and superalloys to be delivered and used in the manufacture of aircraft engines. The PM technologies have been reviewed for three major aspects, i.e. powder production, HIPing and HIP-based processes and properties of PM superalloys. The review has been used to analyse the current HIPing and HIP-based processes, aiming at identifying the features and usage of commonly used processes and the issues within those processes. Different generations of PM superalloys have been studied and reviewed to understand the characteristics and properties of the studied material in this research, i.e. FGH96 superalloy. Based on the understanding of the mechanism for material behaviours during
hot deformations, the materials modelling has been reviewed to provide a background for the development of viscoplastic constitutive equations. Dislocation evolution has been identified as the dominant mechanism for plastic deformation, and the dislocation generation and annihilation have been considered to cause the changes in flow stress. The viscoplastic constitutive model based on the dislocation evolution will be introduced in Chapter 5.
Chapter 3

Experimental Techniques

3.1 Introduction

This chapter presents the experimental methods utilised in this study. The experimental can be classified into three groups:

1) Characterisation of the test materials: uniaxial compression tests were carried out to characterise the stress-strain relationship in hot deformation conditions for HIPed nickel-based superalloy FGH96 and stainless steel AISI 304 used for capsule of powder ingot;

2) Development of the DPF process: tests were designed to investigate the effect of process parameters on DPFed samples and study the feasibility of DPF in both laboratory and industrial conditions, which was completed by small size powder forging tests, laboratory feasibility study and industrial trials;

3) Investigation of the microstructure and properties of DPFed components: tests were conducted to examine the qualities of DPFed components manufactured under different conditions and investigate the microstructure evolution within formed parts, including density tests, hardness tests, tensile tests and microstructure observations.

3.2 Test materials

In this study, nickel-based superalloy FGH96 was selected as the process material. FGH96 is a second-generation damage-tolerance superalloy with high working temperature and low crack growth rate (Liu et al., 2009). The chemical composition of FGH96 is shown in Table 3.1.
Table 3.1 Chemical composition of FGH96 (Wt%).

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
<th>Ta</th>
<th>Nb</th>
<th>Al</th>
<th>Ti</th>
<th>Zr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>15</td>
<td>13</td>
<td>4</td>
<td>4</td>
<td>&lt;0.2</td>
<td>0.8</td>
<td>2.2</td>
<td>3.7</td>
<td>0.035</td>
</tr>
<tr>
<td>Element</td>
<td>Si</td>
<td>Fe</td>
<td>Mn</td>
<td>N</td>
<td>C</td>
<td>S</td>
<td>P</td>
<td>B</td>
<td>Ni</td>
</tr>
<tr>
<td>Wt%</td>
<td>&lt;0.2</td>
<td>&lt;0.5</td>
<td>&lt;0.15</td>
<td>≤0.005</td>
<td>0.04</td>
<td>0.015</td>
<td>0.015</td>
<td>0.1</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Two forms of FGH96 were employed. One was fully dense HIPed FGH96 and the other was FGH96 powder. The HIPed FGH96 was prepared by HIPing process which was undertaken at around 1160 °C and 120 MPa for 2 to 4 hours and was machined to the shape of cylinders for uniaxial compression tests (Bai et al., 2015). The FGH96 powder was prepared with argon atomisation method and used in DPF-related tests. Because the DPF process involved a stainless steel container, cylindrical stainless steel AISI 304 samples were also prepared for the uniaxial compression tests to determine the mechanical behaviour. All materials were supplied by Beijing Institute of Aeronautical Materials (BIAM).

3.3 Uniaxial compression tests

In order to characterise the material behaviour of FGH96 during hot deformation process, uniaxial compression tests were carried out on HIPed FGH96 test-pieces at elevated temperatures using a Gleeble 3800 test station. Ideally the compression tests should be conducted on a single FGH96 particle to acquire the mechanical properties of the raw material for DPF experiments. Nano-indentation could be used for such purpose, but this technique could not be implemented at temperatures over 900 °C (Park et al., 2016; Zhang et al., 2017). Therefore HIPed FGH96 test-pieces were used to approximate the mechanical properties of a single FGH96 particle.

The tests were focused on the stress-strain relationship of FGH96 at different temperatures and strain rates, and the results were used to calibrate and validate the material constitutive model developed for DPF process.
3.3.1 Test equipment

A Gleeble 3800 test station (Dynamic Systems Inc., USA) was used to perform hot compression tests and is shown in Figure 3.1. The Gleeble is designed to carry out thermo-mechanical tests and physical simulation programmes, with a maximum static force of 20 tons in compression, a heating rate of up to 10,000 °C/s and a displacement rate of up to 2000 mm/s. A moving jaw, which moves along a horizontal axis and provides mechanical displacement to deform the test-piece, and a static jaw are pre-installed in the Gleeble test chamber. The Gleeble uses resistance heating to provide the heating power for the test-piece. Electrical current will be generated once the circuit is closed by installing the test-piece between two jaws and the programme is started.

![Gleeble 3800 thermo-mechanical test station.](image)

Thermocouples were used to monitor the real-time temperature of the test-piece and control the heating power. A C-gauge was used to record the diameter change of the test-piece, and the longitudinal strain could be calculated from C-gauge reading as:

\[ \varepsilon = -2 \cdot \ln \frac{d}{d_0} \]  

\[ (3.1) \]
where $\varepsilon$ is the longitudinal strain, $d_0$ is the original diameter and $d$ is the instantaneous diameter recorded by C-gauge.

### 3.3.2 Test preparation and set-up

Two tungsten carbide anvils were installed on the two jaws in the Gleeble test chamber. Graphite foils were placed onto the anvils to stabilise and enhance electric current flow and also to avoid direct contact between the test-piece and the anvils. A high temperature lubricant gel for nickel materials was applied to glue the graphite foils to the anvils, as well as to reduce friction. The HIPed FGH96 test-piece was a cylindrical specimen with a length of 12 mm and a diameter of 8 mm, as shown in Figure 3.2. It was mounted between the anvils and stabilised by applying a small compressive force of around 100 N. The thermocouples were spot welded to the centre of the test-piece, providing accurate temperature feedback. The C-gauge was installed at the centre of the test-piece to monitor the changes in diameter. Once the set-up has been completed, the test chamber was closed and vacuumed to around $4 \times 10^{-1}$ Pa to reduce the oxidation of the test-piece at high temperatures. Figure 3.2 shows the geometry and dimensions of the test-piece and the set-up of uniaxial compression test.

![Figure 3.2 The geometry and dimensions (mm) of the test-piece and the set-up of uniaxial compression tests.](image)
3.3.3 Test operation

Tests were carried out at different temperatures and strain rates. The deformation temperatures of uniaxial compression test were chosen between 900 °C and 1100 °C. The test temperatures were selected to be compatible with practical HIPing conditions with the consideration of temperature distributions. The test temperatures for FGH96 were chosen to be in the range from 1000 to 1100 °C, while those for stainless steel were selected between 900 and 1100 °C considering the heat loss and heat transfer of the container. In addition, the maximum strain rate of the uniaxial compression test was limited at 10 s\(^{-1}\) to allow adequate control for the Gleeble to keep the strain rate stable. As shown in Table 3.2, 10 sets of uniaxial compression tests were selected, with each set to be repeated once.

Table 3.2 Uniaxial compression test conditions. ‘√’ indicates the selected test conditions for FGH96 and ‘○’ represents those for stainless steel AISI 304.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Strain rate (s(^{-1}))</th>
<th>0.1</th>
<th>1.0</th>
<th>10.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td></td>
<td></td>
<td>○</td>
<td></td>
</tr>
<tr>
<td>1000</td>
<td>√</td>
<td>○</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1050</td>
<td></td>
<td></td>
<td></td>
<td>√ ○</td>
</tr>
<tr>
<td>1100</td>
<td></td>
<td></td>
<td>√</td>
<td>○</td>
</tr>
</tbody>
</table>

Every compression test cycle was divided into 5 stages, as shown in Figure 3.3:

i) Fast heating stage: the test-piece was heated at 20 °C/s to 100 °C below the deformation temperature;
ii) Slow heating stage: the test-piece was heated at 2 °C/s to the deformation temperature to prevent overheating;

iii) Soaking stage: the test-piece was held at the deformation temperature for 180 seconds;

iv) Deformation stage: the test-piece was deformed at a certain temperature and strain rate;

v) Cooling stage: the test-piece was cooled to room temperature.

Figure 3.3 Illustration of the 5 stages in uniaxial compression tests.

The progression of the compression test was first determined by the programmed temperature and a force control mode was used with a small compressive force of 0.2 kN during heating and holding cycle, to accommodate thermal expansion of the test-piece. When the deformation stage started, the controlling was switched to stroke control. After the deformation stage, the test programme was stopped and the test-piece was cooled to room temperature.

For each stage, the real-time temperature and displacement were recorded. The recorded experimental data were checked against the testing programme to find out any unexpected heating or strain rate errors. Repetition test was then conducted if necessary.
3.4 Small size powder forging

The small size powder forging was employed to investigate the effects of process parameters, i.e., deformation temperature, loading force and holding time, on the density, mechanical properties and microstructure evolution of DPFed components. Compared to laboratory feasibility study and industrial trials, a small size powder forging test uses much less FGH96 powder, hence produces a relatively small cylindrical component, with a length of less than 5 mm and a diameter of 4 mm.

The small size powder forging tests were also conducted on the Gleeble test station, and similar testing procedures were adopted. Three major changes were made. First, instead of using HIPed FGH96 test-pieces, FGH96 powder was used and filled into a special graphite die. Second, the controlling method was changed at the deformation stage, which was loading force control rather than stroke control. Third, the C-gauge was removed from the test set-up since the diameter of the FGH96 powder forging was determined by the cavity size of the graphite tool.

3.4.1 Graphite tool-set

A specially designed tool-set was employed in small size powder forging tests. A cylindrical die was prepared to hold FGH96 powder, with a small hole drilled in the middle part of the die for thermocouples, and two punches were manufactured to be compatible with the die. An illustration of the graphite tool-set is presented in Figure 3.4. Graphite was chosen as the tool-set material for its high thermal stability, electrical and thermal conductivity, which was crucial for the tool-set to be utilised on the Gleeble test station, since the heating was completed by electrical current and a more uniformed temperature distribution was of high importance to generate desirable results. In addition, graphite was valued for its lubricating properties to prevent the friction within the tool-set from affecting the stress conditions (Zhao et al., 2015; Zulkipli et al., 2015).
Figure 3.4 The graphite tool-set for the small size powder forging tests: a) overview of the graphite tool-set; b) longitudinal-section of the graphite tool-set.

One problem with the tool-set was that the strength of the graphite material used in small size powder forging tests were much lower than that of FGH96. Therefore, the maximum loading force was limited at 2500 N in the tests to avoid tool-set failures during the test.

3.4.2 Test preparation and set-up

Similar to the uniaxial compression test, the small size powder forging tests were conducted on the Gleeble test station. Anvils, graphite foils and nickel high temperature lubricant gel were applied for the same purposes. The graphite tool-set was prepared in advance, with FGH96 powder filled in the die and two punches placed at two ends of the die to keep powder from spilling out. Thermocouples were inserted into the small hole to track temperature changes. The tool-set was then mounted between the two anvils and stabilised by applying a small compressive force of around 100 N. Figure 3.5 is a demonstration of the test set-up.
3.4.3 Test operation

Three key factors, namely deformation temperature, loading force and load holding time, were believed to affect the properties of DPFed components. In this study, tests were designed to investigate the effect of loading force and load holding time, with each group being carried out at 1150 °C with different loading forces and load holding times. The test conditions are shown in Table 3.3.
Table 3.3 The small size powder forging test at 1150 °C. ‘√’ indicates the selected test conditions.

<table>
<thead>
<tr>
<th>Loading force (kN)</th>
<th>15</th>
<th>20</th>
<th>30</th>
<th>60</th>
<th>100</th>
<th>160</th>
<th>190</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>√</td>
<td>√</td>
<td></td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>√</td>
</tr>
<tr>
<td>2.5</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The small size powder forging tests were divided into 6 stages: fast heating, slow heating, soaking, loading, load holding and cooling, as shown in Figure 3.6:

i) Fast heating stage: the tool-set was heated at 20 °C/s to 100 °C below the deformation temperature;

ii) Slow heating stage: the tool-set was heated at 2 °C/s to the deformation temperature;

iii) Soaking stage: the tool-set was held at the deformation temperature for 300 seconds to ensure temperature homogenisation;

iv) Loading stage: the FGH96 powder was deformed at the temperature to the required load level;

v) Load holding stage: the load was maintained at the deformation temperature for a certain period of time;

vi) Cooling stage: the tool-set was cooled to room temperature.
Similar to the compression test, the progression of the small size powder forging test in the heating period was determined by temperature with force control mode. The controlling was then continued with force control in the loading and load holding period where the FGH96 powder was consolidated. The tool-set was held at a certain loading force level for a short period of time, followed by a cooling stage.

Different from the compression test, when the loading period started, it was still force control instead of changing to stroke control for the purpose of preventing more test failures. There were two main types of test failure in small size powder forging tests:

1) Unconsolidated components: when the total stroke was set to a small value, the powder could not be fully consolidated due to a relatively small jaw displacement that would lead to an early termination of the test programme;

2) Over-pressed tool-set: the test programme was not stopped when the powder was fully consolidated, so the test programme was still in operation and the tool-set was kept being pressed, which would cause severe tool-set failures.

![Illustration of the test periods and stages in small size powder forging tests.](image)
Therefore, loading force control was selected during the loading period to correctly stop the test programme and relatively small loading forces were selected to prevent tool-set from being damaged.

For each period and stage, the real-time temperature, loading force and time were recorded. The recorded experimental data were examined to find out any unexpected heating or loading errors. Repetition test was then conducted if necessary. Forged components were then prepared into samples for further tests of material properties and microstructure examinations.

### 3.5 Laboratory feasibility study

In order to further investigate DPF process, a laboratory feasibility study was carried out. An up-setting test was conducted to study the effect of stress on powder consolidation in DPF process (Bai et al., 2015).

#### 3.5.1 Test equipment and set-up

The set-up for the up-setting test was completed on a 250 kN ESH single shot high rate test machine. The test machine is shown in Figure 3.7.

![Figure 3.7 The 250 kN ESH single shot high rate test machine.](image)
Two flat dies were mounted on a pillar tool-set which was installed on the test machine. The test machine was connected to an oscilloscope to record stroke and loading parameters during the forging process. A separate furnace was employed to pre-heat the test-piece since no heating system was implemented within the test machine. The test-piece was designed to be in the shape of a cylinder and placed horizontally onto the fixed lower die. The movement of the upper die was driven by a punch installed on the test machine, which provided the compression force to deform the test-piece. The set-up of the up-setting test is shown in Figure 3.8.

![Figure 3.8 The set-up of the up-setting test for laboratory feasibility study.](image)

### 3.5.2 Test-piece preparation

A specially designed test-piece was used in the laboratory feasibility study. A cylindrical stainless steel container, with the outer diameter of 20 mm, outer height of 20 mm and thickness of 2 mm, was filled with FGH96 powder. Stainless steel AISI 304 was chosen as the container material because of its good weldability, high stiffness and strength at room temperature, and high ductility at elevated temperatures, which was ideal for maintaining shape and dimensional control at room temperature but soft and malleable at the deformation temperature. A tube was designed on the top of the cylindrical container for filling powder. It was then vacuumed to $1 \times 10^{-5}$ Pa and sealed to reduce the amount of
air, hence reduce the oxidation of the powder at high temperature. The container was coated with glass lubricant to reduce the friction during the forging test. Figure 3.9 shows the concept and design of the container and the test-piece.

![Figure 3.9 The geometry and dimensions (mm) of the stainless steel container, and a prepared test-piece coated with glass lubricant.](image)

### 3.5.3 Test operation

The up-setting test was divided into 4 stages:

i) Pre-heating stage: the test-piece was placed into the furnace and heated to the carbide super-solvus temperature of 1150 °C;

ii) Soaking stage: the test-piece was held at 1150 °C for 20 minutes to obtain a uniformed temperature distribution;

iii) Forging stage: the test-piece was transferred from the furnace to the test set-up and immediately compressed at a speed of 200 mm/s to a certain load;

iv) Cooling stage: the upper die was retracted and the test-piece was cooled at room temperature.

After the up-setting test, the forged test-pieces were cut along the black dashed lines as shown in Figure 3.8 and prepared into hardness test samples and microstructure analysis.
samples. Further investigations were undertaken to study the consolidation status and microstructure evolution of the DPFed components.

3.6 Industrial trials

The small size powder forging tests and laboratory feasibility study were both conducted at the laboratory level. Generally speaking, the testing circumstances are relatively ideal compared to industrial conditions, for example, the test parameters of laboratory experiment are less complicated than those of industrial productions, the compression speed of 200 mm/s in laboratory feasibility study is not readily available for most industrial machines (Wang et al., 2016). Therefore, industrial trials were proposed to further investigate the possibility of implementing DPF process into industrial applications.

3.6.1 Test equipment and set-up

The industrial trial tests were conducted on a single-acting hydraulic press machine, with a load capacity of 20,000 kN and a maximum speed of 20 mm/s. A cylindrical upper punch was fixed on the top of the tool-set and driven by the press machine to provide loading force onto the test-piece. An extrusion ring, with an inner diameter of 131 mm, was mounted on the bottom die to hold the test-piece, as well as to set a limit for the radial expansion of the test-piece and impose a radial stress towards the centre of the test-piece. The set-up parts, namely the upper punch, the extrusion ring and the bottom die, were manufactured from H13 steel. The test equipment and set-up are illustrated in Figure 3.10.
3.6.2 Test-piece preparation

The test-piece was a combination of two major parts:

1) Powder ingot: the powder ingot was prepared with a stainless steel container filled with FGH96 powder, similar to the test-piece used in laboratory feasibility study;

2) Protective covers: two stainless steel protective covers, namely top cover and bottom cover, were designed to provide better support for the powder ingot, e.g., protection of the filling tube, stabilisation of the temperature distribution, and assurance of the container stability.

The size of the powder ingot was designed to be compatible with that of the industrial work-piece used in HIPing process for better comparability. The complete test-piece, including the powder ingot and two protective covers, was designed to be fit into the test set-up, with a total height of 242 mm and outer diameter of 131 mm. The test-piece was also coated with glass lubricant to reduce friction during the forging process. Figure 3.11 shows the design of the test-piece for industrial trails.
3.6.3 Test operation

In the consideration of keeping experimental consistency, the industrial trail test was also completed in 4 stages, similar to the laboratory feasibility study, but with a few changes to the process conditions to be compatible with industrial applications:

i) Pre-heating stage: the test-piece was placed into the furnace and heated to 1070 °C, which was the recommended deformation temperature in industrial applications;

ii) Soaking stage: the test-piece was held at 1070 °C for 2 hours to obtain a uniformed temperature distribution;

iii) Forging stage: the test-piece was transferred from the furnace to the test set-up and immediately compressed at a speed of 17 mm/s to a certain load;

iv) Cooling stage: the upper die was retracted and the test-piece was cooled at room temperature.

After the test, the forged test-pieces were prepared into samples for the density measurement and microstructure analysis. Further investigations were undertaken to study the consolidation status and microstructure evolution of the DPFed components in industrial circumstances.
### 3.7 Examination and analysis of the DPFed samples

For a better understanding of the DPF process, the processed test-pieces obtained from the DPF-related tests were prepared into samples for further examinations. In this study, each set of samples was prepared for different purposes, thus the examination methods were distinct from one set to another. However, the main tests include density measurement, hardness and tensile tests, and microstructural examination. Examination results were then used to investigate the effects of DPF process parameters on material properties of the components, evaluate the feasibility of producing fully dense FGH96 parts, and assess the possibility of implementing DPF process into industrial applications, which was of great importance to the development of the DPF process.

#### 3.7.1 Density measurement

The density measurement was carried out on all samples based on Archimedes drainage method. The results obtained from the density measurement were analysed differently:

1) For small size powder forging tests: the material density was associated with process parameters to identify the relationship between deformation condition and consolidation status;

2) For laboratory feasibility study: the material density was used to investigate the connection between the stress state and the material density;

3) For industrial trials: the material density was measured as an evidence that DPF process was capable of producing fully dense components.

#### 3.7.2 Hardness test

Micro-hardness tests were carried out in a Zwick/Roell hardness testing system to study the hardness distribution of DPFed components in the laboratory feasibility study. The forged test-pieces were cut along the dashed lines shown in Figure 3.8 and then mounted into hardness samples. A total of 13 test locations were chosen alongside the x axis with a
spacing of 1 mm and the samples were tested with HV5 hardness. Figure 3.12 shows the surface of the hardness test sample and the first test location.

Figure 3.12 The hardness test sample and the test surface. The red dot indicates the first test location.

3.7.3 Tensile test

A small size tensile test was designed for samples acquired from the small size powder forging to further investigate the influence of process parameters on the consolidation of the FGH96 powder. The sample was machined to a small ‘dog-bone’ shape, with a length of less than 5 mm, a diameter of 1.5 mm for the test region and a diameter of 3.5 mm for both end regions. Because of the relatively small size of the sample, a special pair of grips was designed to hold the sample during the tensile test. Figure 3.13 shows the ‘dog-bone’ shaped sample and the special grips designed for the small size tensile test.

Figure 3.13 The geometry and dimensions (mm) of the ‘dog-bone’ shaped sample and the special grips designed for the small size tensile test.
The tensile tests were conducted on an Instron 5980 universal testing machine. The 5980 floor model is commonly used for testing high-strength metals and alloys, advanced composites, aerospace and automotive structures, etc. The machine features a loading capacity from 100 to 600 kN and a speed range from $5 \times 10^{-5}$ to 1016 mm/min. Figure 3.14 shows a picture of the Instron 5980 floor model.

![Figure 3.14 The Instron 5980 floor model universal testing machine.](image)

### 3.7.4 Microstructure observation

The obtained test-pieces were prepared into samples for microstructure observations. An example of the microstructure observation sample is shown in Figure 3.15 with hardness testing indents. The procedures and machines for sample preparation are presented in Figure 3.16. Generally, the samples were first cut into appropriate sizes to fit the
requirement for SEM samples. Then the cut samples were mounted with conductive materials into a cylindrical shape. After mounting, the samples were processed with grinding and polishing to get the required surface finish. Finally, etching was applied to different samples to serve specific purposes (Li et al., 2017). For FGH96 materials, two standard etching methods were selected as follow:

1) For the examination of porosity and grain structure: samples were etched with Kalling’s solution of 5 g CuCl<sub>2</sub>, 100 ml HCl and 100 ml ethanol for a better presentation of the porosity and grain structure;

2) For the examination of precipitates: samples were etched by electroetching, with a voltage of 3-5 V and a solution of 170 ml H<sub>3</sub>PO<sub>4</sub>, 10 ml H<sub>2</sub>SO<sub>4</sub> and 15 g CrO<sub>3</sub>, to obtain a better image of the precipitates.

![Image of a prepared microstructure observation sample](image)

**Figure 3.15** An example of the prepared microstructure observation sample.
For test-pieces obtained from small size powder forging tests, the size was relatively small, thus the samples could be prepared without the cutting procedure. On the other hand, the test-pieces obtained from laboratory feasibility study and industrial trials were covered with stainless steel container, thus they needed to be cut into half to show the FGH96 material. In addition, to investigate the microstructural distribution, each test-piece was divided into different locations to represent the examination point. Figure 3.17 demonstrates the chosen locations.
Figure 3.17 The cross section of the forged samples and chosen locations for microstructure observations: a) samples from laboratory feasibility tests; b) samples from industrial trial tests.

For laboratory SEM samples, six locations were selected and marked with ‘a’ to ‘f’, as shown in Figure 3.17(a), to represent the examination locations for the sample. For industrial SEM samples, the test-pieces were cut in the longitudinal section and marked with ‘1’ to ‘30’, as shown in Figure 3.17(b). Because the industrial test-pieces were too large to be directly mounted into SEM samples, the samples were prepared by obtaining a small cube with the size of $10 \times 10 \times 10$ mm from the chosen locations. Then these 30 small cubes were mounted separately into SEM samples.

Microstructure observation was undertaken on a Hitachi S-3400N scanning electron microscope (SEM) to investigate the consolidation status and microstructure evolution of
the DPFed samples obtained from the laboratory tests and industrial trails. The test
machine is presented in Figure 3.18. The machine features a fully eucentric 5 axis
motorised stage that allows samples up to 25 mm in diameter and an accelerating
voltages up to 30 kV. SEM tests were designed to investigate the porosity, powder
consolidation, and microstructure at the marked locations. The porosity was measured by
counting the pixels for the porosity to get a percentage, and the material density was
calculated by 1.0 minus the porosity percentage. Five images were taken at each location
for the analysis of microstructure. Electron Backscatter Diffraction (EBSD) was also
carried out on the SEM system equiped with the HKL software to study the grain
structure and grain size distribution, with a step size of 1.2 µm and an operating voltage
of 20 kV.

Figure 3.18 The Hitachi S-3400N SEM test machine.
Chapter 4

Experimental Results

4.1 Introduction

This chapter presents the experimental results obtained during this study, followed by analysis and discussion. These results are used to investigate the material properties of FGH96, the features and feasibility of DPF process. This chapter contains:

1) Mechanical properties for the test materials: the stress-strain relationships for both FGH96 and stainless steel AISI 304, providing a basic understanding of the test materials;

2) Effects of process parameters: the material characterisation and mechanical properties of test-pieces obtained from small size powder forging tests under different process conditions, and the microstructure observations for the prepared SEM samples, indicating the influence of process parameters on the DPFed components;

3) Feasibility study: the results of consolidation and hardness tests for the chosen locations of the test-pieces, and the connection between material characterisations and microstructure distributions, providing evidence that DPF process is capable of producing fully dense FGH96 superalloy;

4) Industrial trials: the results of the density for different test locations and microstructure observations for the SEM samples, including PPB conditions, grain structure and grain size distribution.

4.2 Results for high temperature uniaxial compression tests

4.2.1 Stress-strain relationship for stainless steel AISI 304

Figures 4.1 and 4.2 show the stress-strain curves for stainless steel AISI 304 at different deformation temperatures and different strain rates respectively. As mentioned in the previous chapter, stainless steel AISI 304 was chosen as the container material because
the mechanical properties of this material are ideal for both holding FGH96 powder at room temperature and maintaining the shape and dimensional control at deformation temperature. The results are for 900, 1000, 1100 °C with a strain rate of 1.0 s\(^{-1}\), and for strain rates of 0.1, 1.0 and 10.0 s\(^{-1}\) at 1000 °C. The test temperatures and strain rates were chosen to best reflect the DPF process conditions.

As can be seen from the figures, the stainless steel AISI 304 shows a good strength property, which is strong enough to keep the container from breaking during DPF process, but is soft and malleable under deformation conditions, which provides proper shape and dimensional control for the final components. The results show that strength increases with decreasing temperature and increasing strain rate. These results will be used to develop the constitutive equations for process simulations in the next chapter.

![Stress-strain curves for stainless steel AISI 304 at different temperatures with a strain rate of 1.0 s\(^{-1}\).](image)

**Figure 4.1** Stress-strain curves for stainless steel AISI 304 at different temperatures with a strain rate of 1.0 s\(^{-1}\).
4.2.2 Stress-strain relationship for FGH96

Figures 4.3 and 4.4 show the stress-strain curves for FGH96 alloy at different temperatures and different strain rates. A few conclusions can be drawn from the results. Firstly, as demonstrated by Figure 4.3, the flow stress decreases significantly with the increase of temperature from 1000 °C to 1050 °C, but shows a relatively small drop between 1050 °C and 1100 °C. This is mainly because the reinforcement γ’ phase starts to dissolve at around 1000 °C, and with the increase of temperature from 1000 °C to 1050 °C, the amount of dissolved γ’ phase will increase significantly, which results in a large decrease of flow stress. As the temperature approaches the γ’ solvus temperature of around 1120 °C, compared to the temperature range of 1000 °C to 1050 °C, less amount of γ’ phase remains in the material between 1050 °C and 1100 °C, which leads to less γ’ phase dissolving and a relatively small decrease in flow stress. Secondly, material softening occurs during the hot deformation. It is believed that dynamic recovery and recrystallisation are the dominant softening mechanisms of FGH96 during hot deformation process. Thirdly, as can be seen from Figure 4.4, the flow stress increases
with increasing strain rate, which shows the viscoplastic nature of the material at deformation temperatures and strain rate hardening effect.

Figure 4.3 Stress-strain relationships of FGH96 at different deformation temperatures with a strain rate of 1.0 s\(^{-1}\).

Figure 4.4 Stress-strain relationships of FGH96 at 1000 °C with different strain rates.
4.3 Results of small size powder forging tests

4.3.1 Mechanical properties test

Tables 4.1 and 4.2 show the density and tensile test results for forged samples prepared by small size powder forging tests. First, material density was measured for each sample before conducting tensile test and then converted to relative density. As can be seen from the tables, the relative density for forged samples ranges from 0.95 to 1.0, which indicates that the desired consolidation condition can be achieved by small size powder forging, and fully dense components can be acquired by DPF process. However, compared to standard DPF process or industrial experiment, the loading force used in small size powder forging test is relatively small, so relative density may not be adequate to reflect the powder bonding condition correctly, thus tensile tests and microstructure observation are carried out to provide further evidence of consolidation condition. Second, due to technical issues, the tensile test may result in abnormal fracture position of the forged samples, as shown in Figure 4.5, so only results with normal fracture position will be used to identify the tensile strength features of the forged FGH96 samples. Results marked by green in Tables 4.1 and 4.2 are used to generate Figures 4.6 and 4.7.

<table>
<thead>
<tr>
<th>No.</th>
<th>Holding Time (s)</th>
<th>Density (g/mm³)</th>
<th>Relative Density</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Notes</th>
</tr>
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<td>60</td>
<td>8.27</td>
<td>0.99</td>
<td>1412</td>
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<td>Fracture at grip position</td>
</tr>
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<td>60</td>
<td>8.14</td>
<td>0.98</td>
<td>1546</td>
<td>14.8</td>
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</tr>
<tr>
<td>3</td>
<td>100</td>
<td>8.20</td>
<td>0.98</td>
<td>1476</td>
<td>28.7</td>
<td>Fracture at grip position</td>
</tr>
<tr>
<td>4</td>
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<td>1.02</td>
<td>1558</td>
<td>-</td>
<td>No elongation due to unusual fracture surface</td>
</tr>
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<td>0.97</td>
<td>1542</td>
<td>-</td>
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<td>0.96</td>
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<td>0.98</td>
<td>1558</td>
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Table 4.2 The 2nd group of tensile test results (samples prepared at 1150 °C, 2.5 kN).

<table>
<thead>
<tr>
<th>No.</th>
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<th>Density (g/mm³)</th>
<th>Relative Density</th>
<th>Results</th>
<th>Notes</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Tensile Strength (MPa)</td>
<td>Elongation (%)</td>
</tr>
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<td>0.98</td>
<td>1571</td>
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</tr>
<tr>
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<td>0.98</td>
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<tr>
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</tr>
<tr>
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<td>0.98</td>
<td>1541</td>
<td>18.4</td>
</tr>
<tr>
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<td>1.00</td>
<td>1569</td>
<td>24.7</td>
</tr>
</tbody>
</table>

Figure 4.5 Tensile test fracture position: a) normal fracture position; b) fracture at grip position.

Figures 4.6 and 4.7 present the tensile test results for chosen forged samples. The results show that with a relatively longer holding time, the tensile strength increases slightly. With longer holding time, the powder bonding condition will be improved, hence a stronger component can be acquired, which results in higher tensile strength. Microstructure analysis will be used to further investigate the powder bonding condition and grain size evolution.

Compared to standard HIPed FGH96 superalloy, the tensile strength of forged samples is close to and even higher than the standard value of 1550 MPa, which proves that DPF process is capable of producing FGH96 components with required strengths.
4.3.2 Microstructure analysis

Figures 4.8 to 4.14 show the microstructure for the 2\textsuperscript{nd} group of forged FGH96 samples prepared by small size powder forging. The 2\textsuperscript{nd} group of forged FGH96 samples was prepared with a relatively higher loading force of 2.5 kN, which is more compatible with DPF process, and has a more regular fracture pattern during tensile tests compared to the 1\textsuperscript{st} group of forged FGH96 samples. As mentioned previously, all results in Figures 4.8 to
4.14 are from the samples prepared at 1150 °C with load holding time from 15 to 100 seconds.

As shown in Figure 4.5, each tensile test sample has two sides of fracture surface. For Figures 4.8 to 4.12, the red circles in (a) or (b) indicate the chosen side and location for the magnification and a more detailed observation, and (c) and (d) are the magnified view of the chosen location with different magnitude.

Figures 4.8 to 4.12 are the observation of samples without etching, while Figure 4.13 is the result after chemical etching, which aims for the investigation of porosity and grain structure, and Figure 4.14 is the result for electroetching, which focuses on the observation of precipitates.
4.3.2.1 Load holding time of 15 s

Figure 4.8 shows the microstructure for the sample prepared with load holding time of 15 seconds. From Figures 4.8(a), (b) and (c), it can be seen that most of the powder particles are still spherical, which suggests that the combination of loading force at 2.5 kN and load holding time of 15 s do not provide much plastic deformation to the FGH96 powder. Large voids can be found as shown in Figure 4.8(d), indicating that the consolidation for this sample is not sufficient.
Figure 4.8 Microstructure of the fracture surface of forged FGH96 sample with 1150 °C and load holding time of 15 s: red circle indicates the magnification location; a) and b) are different sides of fracture surface; c) and d) are the magnified pictures for the red circle location with different magnitude.
4.3.2.2 Load holding time of 20 s

Figure 4.9 is the microstructure for the sample prepared with load holding time of 20 seconds. For this sample, there is no obvious improvement on the deformation of powder particles compared to the first sample, as shown in Figures 4.9(a), (b) and (c). However, no large voids are found within this sample, indicating that the consolidation has been improved with the increase of load holding time.
Figure 4.9 SEM micrograph for samples with load holding time of 20 s.
4.3.2.3 Load holding time of 30 s

Figure 4.10 presents the microstructure for the sample prepared with load holding time of 30 seconds. From the macro-scale of the SEM Figures 4.10(a) and (b), it can be hardly seen that the powder particles are deformed, but Figure 4.10(c) shows an improvement on the particle shape and more deformed particles can be found in Figures 4.10(c) and (d). No large voids are found in these SEM images, but the sample will be chemically etched to examine the porosity.
Figure 4.10 SEM micrograph for samples with load holding time of 30 s.
4.3.2.4 Load holding time of 60 s

Figure 4.11 presents microstructure for the sample prepared with load holding time of 60 seconds. Although voids are found in the Figure 4.11(d), it is still concluded that the longer load holding time improves the deformation of the powder particle and benefits the powder consolidation. The porosity will be further examined with chemical etching for more details.
Figure 4.11 SEM micrograph for samples with load holding time of 60 s.
4.3.2.5 Load holding time of 100 s

Figure 4.12 presents microstructure for the sample prepared with load holding time of 100 seconds. With a relatively long load holding time, no obvious voids can be found and more powder particles are plastically deformed, thus the consolidation is promoted. It can be concluded that with a loading force of 2.5 kN at 1150 °C, it is possible to fully consolidate the FGH96 powder and still obtain a reliable microstructure as long as sufficient load holding time is applied. This can be very helpful for the design of the process parameters and provide guidance to the DPF process.
Figure 4.12 SEM micrograph for samples with load holding time of 100 s.
4.3.2.6 Samples after chemical etching

Figure 4.13 shows the SEM observation of samples after chemical etching. The chemical etching aims for a better observation of porosity and grain structure. It is quite obvious that the powder consolidation for (a), (b) and (c) is not enough that many voids can be found within the material. On the other hand, with an increased load holding time, the porosity has been reduced, as shown in (d) and (e), where no large voids can be found. The grain structure for (d) and (e) is also better than that of (a), (b) and (c), with a more uniformed grain size distribution.
Figure 4.13 SEM observation of forged FGH96 samples after chemical etching with different holding times: a) to e) indicate the different load holding time (15, 20, 30, 60 and 100 s). Red circle indicates the voids found in the SEM image.
4.3.2.7 Samples after electroetching

Figure 4.14 is the microstructure of samples after electroetching. The electroetching is more useful for the observation of precipitates and γ’ phase. It can be seen that PPBs and PPB networks can be observed from all five micrographs of Figure 4.14, especially for (a), (c), (d). In addition, there is no obvious connection between load holding time and PPB conditions. This is because the elimination of PPBs highly depends on the plastic deformation rather than load holding time, but the loading force used in these tests is not enough to provide sufficient large plastic deformation to break PPBs, hence a large amount of PPBs will be observed within the samples obtained from small size powder forging tests. This can be easily improved by increasing the loading force.
Figure 4.14 SEM observation of forged FGH96 samples after electroetching with different holding times: a) to e) indicate the different load holding time (15, 20, 30, 60 and 100 s). Red arrow points out a typical PPB structure.

4.4 Results of laboratory feasibility study

4.4.1 Consolidation and hardness test

Figure 4.15 shows the distribution of relative density and hardness with different distances from the centre line of the sample as indicated by ‘x’ in the insert. The results demonstrate that the relative density reaches 1.0 within a 7.5 mm from the centre line, then reduces gradually with the increase of distance from 7.5 mm to 10 mm. The relative density decreases greatly when the distance is larger than 10 mm, to 0.75 at 14 mm. The Vickers hardness has a similar pattern. The maximum hardness is observed near the centre line with a value of 450 HV. The minimum hardness is at the edge of the sample, with a value of 200 HV. The large difference indicates that powder consolidation has a great effect on the relative density, and will affect the material hardness. As the relative density decreases from 1.0 to 0.75, the value of hardness decreases by about 50%. To further investigate the relationships between powder consolidation and material properties, microstructure analysis was carried out to provide more understanding.
4.4.2 Microstructure analysis

Since the laboratory feasibility test is upsetting, the maximum stress will be found in the centre area, which is beneficial to powder consolidation, and the friction will result in the formation of a barrelling area with much lower stress and lower density. Figure 4.16 shows the microstructure of tested samples at different locations. As shown in Figures 4.16(a) and (b), within 7.5 mm from the vertical central line, the powder particles are almost fully consolidated. The grain size is less than 10 μm, which indicates recrystallisation occurred during the hot deformation. The PPB networks are broken sufficiently, and the PPB precipitate distribution is isolated and dispersed in this area. Figure 4.16(b) shows a micrograph with a higher magnification. It can be seen that the powder particles are bonded, and no obvious voids can be found in this area. At location c, which is 10 mm away from the vertical centre line, voids are present and the fraction of voids is less than 5% of the whole area. Recrystallisation (RX) can also be found because of large plastic deformation. However, at location d, which is on the edge of the sample, few powder particles are consolidated since particles in this area were subject to very low
stress due to the free end at the barrelling area. The particles are packed relatively loosely, and recrystallisation can hardly be observed inside the particles in Figure 4.16(d). In the vertical direction at the centre line, as shown in Figure 4.16(e) and (f), the powder particles are fully consolidated, and recrystallisation can be found in this area, similar to Figure 4.16(a).

These tests were focused on the feasibility of conducting DPF process to produce fully dense materials. Test results demonstrate the possibility of obtaining fully consolidated components and desired microstructure by adjusting process conditions and applying subsequent heat treatment.
Figure 4.16 Schematic of the sample section and corresponding SEM micrographs of samples prepared by the laboratory feasibility test at different locations. The marked ‘a’ to ‘f’ indicate the chosen locations for the examination of microstructure within the SEM sample.
4.5 Results of industrial trials

4.5.1 Density and microstructure

The samples were prepared and measured as mentioned in the previous chapter. Results show that material density for all samples ranges from 8.341 to 8.348 g/cm$^3$, which suggests that a fully dense component can be produced by DPF process under industrial environment. Figure 4.17 shows a comparison between the microstructures of a typical HIPed FGH96 material and a DPFed FGH96 component prepared in the industrial trials. The PPB networks can be clearly seen in the HIPed sample, while the PPB networks has been substantially reduced in the DPFed component. It has been well studied that the PPB networks are detrimental to the FGH96 component and will cause significant reduction in mechanical properties, such as elongation and stability, and much efforts have been made to reduce or eliminate PPB networks by applying post-production processes, such as rolling, extrusion and forging. The results obtained from these tests indicate that DPF process is capable of improving PPB conditions within the formed components, which can be an advanced technique compared to conventional HIPing process.

![Figure 4.17 Microstructure of (a) HIPed and (b) DPFed FGH96 alloy.](image)
4.5.2 Grain structure

Figure 4.18 shows the grain structure of the tested FGH96 samples. It features a rather heterogeneous and incompletely recrystallised microstructure with a scattered distribution of larger grains (red and yellow coloured) of about 10 to 20 μm, coexisting with a large amount of dynamically recrystallised (DRXed) grains (blue coloured) of about 3 to 5 μm. This may be caused by the very short forging time and the pinning effect of the fine secondary phases, which inhibits the growth of DRXed grains in the material. Meanwhile the coarse grains are dispersed in the microstructure. However, the sporadically distributed coarse grains could become a source for abnormal growth during post heat treatment depending on their micro energy state and overall distribution of grain size.

Figure 4.18 Microstructure of the feature samples of powder billet after DPF. Colours indicate grain size range.
4.5.3 Grain size distribution

The overall grain size distribution was acquired with a 1 μm observation interval of the grain size. The grain size distribution of all the samples is found to be log normal (not bimodal), and the breadth of the grain size does not have obvious difference at different locations. Typical examples of the grain size distribution are shown in Figure 4.19 and the statistical grain size data are presented in Table 4.3. It is shown that the samples have a mean linear intercept grain size of 4.4 to 5.2 μm with the standard deviation of the grain size distribution being 2.6 to 3.0. The maximum grain size is around 30 μm, which is the average size of the powder particles used for the DPF process. This shows that the grain size distributions at various locations does not necessarily have a clear half-peak width advantage that could lead to abnormal grain growth during subsequent solution heat treatment. Relative frequency for grain size of less than 5 μm always occupies an advantage for all samples, indicating that recrystallisation nucleation was in progress at the end of the DPF process. In addition, relative frequency for grain size of greater than 5 μm decreases with the increase of grain size, indicating that the recrystallised grains do not have sufficient time to grow during the DPF process because of the short deformation time.
Figure 4.19 Typical grain size distribution of DPFed FGH96 samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average (μm)</th>
<th>Standard deviation</th>
<th>Maximum (μm)</th>
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<td>2.97</td>
<td>29.48</td>
</tr>
<tr>
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</tr>
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### 4.6 Conclusions

Based on the experimental results presented in the previous sections, major conclusions are drawn as follow:

1) The stress-strain curves indicate that FGH96 exhibits the mechanical properties of viscoplastic material;

2) DPF process is capable of producing fully dense FGH96 components, which is the preliminary requirement for powder metallurgy processes;
3) Three key parameters, namely loading force, holding time and deformation temperature, will determine the material properties of the final component;

4) PPB network issues and microstructure of the forged component can be improved by sufficient deformation during DPF process;

5) The microstructure of DPFed component features a heterogeneous and incompletely recrystallised microstructure with an average size of 5 μm and a scattered distribution of coarse grains of around 10 to 20 μm.
Chapter 5

Materials Models

5.1 Introduction

This chapter presents the development of the constitutive models for both fully dense and powder FGH96 material, as well as the container material stainless steel AISI 304. The developed constitutive models will be used to predict the hot deformation behaviour of studied materials during DPF process. This chapter deals with:

1) Fully dense material model: a set of unified viscoplastic constitutive equations is developed to model the material behaviour of FGH96 and stainless steel AISI 304. The developed model is calibrated using Gleeble experimental data presented in Chapter 4 and the numerical integration results are discussed;

2) Powder material model: the fully dense material model is modified with powder density evolution, which is used to model the material behaviour of FGH96 powder during DPF. The modified model is calibrated and the numerical integration results are presented.

5.2 Development of fully dense material model

In order to model the material behaviour during DPF process, a unified viscoplastic constitutive model is developed for fully dense HIPed FGH96 material and stainless steel AISI 304. The fully dense FGH96 model is then modified, taking into account the powder density evolution for FGH96 powder.

DPF process is a hot deformation process which is carried out at high temperatures ($T > 0.5T_m$) and involves a large plastic strain (Zhuang et al., 2016). Typically, the plastic strain of the material during hot deformation processes is affected by different deformation mechanisms, i.e. dislocation nucleation, isotropic hardening, recovery and recrystallisation, etc. These deformation mechanisms are strain rate and temperature dependent, and contribute to the microstructural evolution, thus influence the
macroscopic mechanical behaviour of the material (Ashby, 1983). The development of constitutive model is based on the understanding of deformation mechanisms, however, accurate prediction of the material behaviour of FGH96 during DPF process is a difficult task due to the complex interactions of deformation mechanisms (Lin et al., 2005a).

5.2.1 Deformation mechanisms

Figure 5.1 shows a general material behaviour during hot deformation processes. The first stage is elastic deformation, where the material is deformed elastically and a linear stress-strain relationship is observed as indicated by Hooke’s law. The dislocation density does not change from its initial condition and no microstructural evolution takes place during this stage. After elastic deformation stage, the material starts to yield and plastic deformation can be observed. Dislocation starts to develop within the material matrix and dislocation density increases, which requires a higher stress to continue the plastic deformation (Berisha et al., 2008). This isotropic hardening leads to a significant increase in the flow stress.

Hot deformation is also accompanied by softening processes. Recovery and recrystallisation are two main softening processes that occur during and after hot deformation stage and contribute to the reduction of dislocation density (He et al., 2017). The recovery and recrystallisation which occur during hot deformation stage are defined as dynamic recovery and dynamic recrystallisation; otherwise they are defined as static recovery and static recrystallisation (Phan et al., 2017).

Dynamic recovery is usually a result of the movement of low angle boundaries generated by dislocation climb and glide, while dynamic recrystallisation takes place when plastic strain reaches a critical value and sufficient nucleation time is given, providing dislocation-free grains (Humphreys, 1999). Compared to dynamic recovery, dynamic recrystallisation leads to annihilation of a large number of dislocations because of the formation of dislocation-free grains, which is the dominant softening process for low stacking fault energy materials (Honeycombe et al., 1972; Pollock et al., 2006).
5.2.2 Unified viscoplastic constitutive model

Since the deformation mechanisms are highly affected by the dislocation accumulation and annihilation, the viscoplastic constitutive model has been developed to reflect the material behaviour based on dislocation density evolution. The model will be explained in terms of plastic strain rate, dislocation density evolution and isotropic hardening. Softening process will be discussed in the next section.

5.2.2.1 Flow rule

The relationship between flow stress and plastic strain can be described with flow rule by integrating factors such as yield stress, isotropic hardening and viscoplastic effect. Strain rate and deformation temperature are also of great importance to the flow stress and should be taken into account when modelling hot deformation processes.
When metals and alloys are deformed at low temperatures \((T < 0.4T_m)\), the plastic deformation is generated by dislocation motion and the stress-strain relationship can be given by:

\[
\sigma = K\varepsilon^N
\]  
(5.1)

where \(K\) is strengthening coefficient and \(N\) is strain hardening exponent. \(\varepsilon\) is strain and the term \(\varepsilon^N\) indicates the strain hardening caused by plastic deformation.

When the deformation process is carried out at elevated temperatures \((T > 0.5T_m)\), thermally activated mechanisms occur and the flow stress is also a function of strain rate, described as:

\[
\sigma = K\varepsilon^N\dot{\varepsilon}^M
\]  
(5.2)

where the dot indicates time derivative and \(\dot{\varepsilon}\) is the strain rate. The term \(\dot{\varepsilon}^M\) indicates the strain rate hardening effect and \(M\) is strain rate hardening exponent. Equation 5.2 can be then rearranged as:

\[
\dot{\varepsilon} = \left(\frac{\sigma}{K\varepsilon^N}\right)^{\frac{1}{M}}
\]  
(5.3)

However, to model the plastic flow of the material, Equation 5.3 needs to be modified with state variables \(R\) and \(k\) which represent isotropic hardening and yield stress respectively. As illustrated in Figure 5.1, during the elastic deformation stage, the stress potential that creates viscoplastic flow can be expressed as \(\sigma - k\), while the stress that leads to viscoplastic flow decreases to \(\sigma - R - k\) after the material starts to yield. This allows the flow rule to be modified as (Lin et al., 2005a):

\[
\dot{\varepsilon}_p = \left(\frac{\sigma - R - k}{K}\right)^n
\]  
(5.4)

where \(\dot{\varepsilon}_p\) is the plastic strain rate and \(n\) is material constant. \(K\) and \(k\) are temperature dependent parameters.
5.2.2.2 Dislocation density evolution

The dislocation density evolution is modelled with a normalised dislocation density term $\bar{\rho}$ which is defined as (Lin et al., 2005b):

$$\bar{\rho} = \frac{\rho_d - \rho_{di}}{\rho_{dm} - \rho_{di}}$$  \hspace{0.5cm} (5.5)

where $\rho_{di}$ is the initial dislocation density and $\rho_{dm}$ is the maximum dislocation density that can be generated within a fully saturated state material, while $\rho_d$ is the current dislocation density during the deformation process. Therefore, the dislocation density evolution can be described with the normalised dislocation density $\bar{\rho}$ as:

$$\dot{\bar{\rho}} = A(1 - \bar{\rho})|\dot{\varepsilon}_p|^{\delta_1} - C\bar{\rho}^{\delta_2}$$  \hspace{0.5cm} (5.6)

where $A$, $\delta_1$ and $\delta_2$ are material constants. $C$ is a temperature dependent parameter.

In Equation 5.6, the first term $A(1 - \bar{\rho})|\dot{\varepsilon}_p|^{\delta_1}$ is used to model the development of dislocation density led by plastic strain and dynamic recovery, while the second term $C\bar{\rho}^{\delta_2}$ indicates that static recovery also contributes to the dislocation density evolution. As it is difficult to accurately describe the interaction between these two mechanisms, dynamic recovery and static recovery have been modelled independently in the dislocation density evolution through Equation 5.6.

5.2.2.3 Isotropic hardening

As discussed in the previous sections, plastic deformation is highly affected by the formation and elimination of dislocations. As plastic deformation continues, dislocation density increases and isotropic hardening takes place and leads to an increase in flow stress. This is because some of the mobile dislocations is still locked in the material lattice and has a chance to interact with other dislocations, which forms new obstacles to dislocation slip.
The strain required to create dislocation slip is heavily influenced by the length over which a dislocation can move and the dislocation slip length. As dislocation density rises, the dislocation slip length reduces and a higher stress level is required to induce the plastic deformation. Based on this, the isotropic hardening is then directly related to the reciprocal of the average slip length through which a dislocation can move. On the other hand, the mean slip length is determined by $\rho_d^{-0.5}$, therefore, the isotropic hardening can be described with normalised dislocation density as:

$$\dot{R} = 0.5 B \rho^{-0.5} \dot{\rho}$$

(5.7)

where $B$ is a temperature dependent parameter.

### 5.2.2.4 Unified viscoplastic constitutive equations for fully-dense material

In summary, the set of unified viscoplastic constitutive equations is developed as (Lin et al., 2005c):

$$\dot{\varepsilon}_p = \left(\frac{\sigma - R - k}{K}\right)^n$$

(5.8)

$$\dot{\rho} = A (1 - \rho) |\dot{\varepsilon}_p|^{\delta_1} - C \rho^{\delta_2}$$

(5.9)

$$\dot{R} = 0.5 B \rho^{-0.5} \dot{\rho}$$

(5.10)

$$\sigma = E (\varepsilon_t - \varepsilon_p)$$

(5.11)

where $E$ is Young’s modulus and also a temperature dependent parameter. $\varepsilon_t$ and $\varepsilon_p$ are total strain and plastic strain respectively. The temperature dependent parameters, i.e. $K$, $k$, $C$, $B$ and $E$, are defined using the classic temperature-compensated parameters in Arrhenius relations as follow:

$$K = K_0 \exp\left(\frac{Q_1}{kT}\right)$$

(5.12)
\( k = k_0 \exp\left( \frac{Q_2}{\kappa T} \right) \)  
\( C = C_0 \exp\left( -\frac{Q_3}{\kappa T} \right) \)  
\( B = B_0 \exp\left( \frac{Q_4}{\kappa T} \right) \)  
\( E = E_0 \exp\left( \frac{Q_5}{\kappa T} \right) \)  

where \( K_0, k_0, C_0, B_0 \) and \( E_0 \) are material constants, and \( Q_1 \) to \( Q_5 \) are activation energy. \( \kappa \) is universal gas constant, \( \kappa = 8.31 \, J \cdot mol^{-1} \cdot K^{-1} \), and \( T \) is absolute temperature.

5.2.3 Calibration of the constitutive model

The proposed unified viscoplastic constitutive model is a set of non-linear ordinary differential equations, which can be solved by numerical integration techniques, for example, forward Euler method. The material constants within Equations 5.8 to 5.16 can be determined by using the experimental data obtained from the uniaxial hot compression tests.

The calibration method involved approximation of material constants and data approaching (Cao et al., 2008; Li et al., 2002). To start with, the boundaries of material constants were determined using an Evolutionary Programming (EP) based optimisation method. As a result, an approximate range for each constant was determined and implemented into a simple spread sheet as a reference for choosing appropriate values. The chosen values were then applied to the constitutive equations to plot calculated stress-strain curves, and these curves were compared with experimental data. By adjusting the applied values, good agreement was gradually achieved and the values of material constants were eventually determined.
5.2.3.1 Constants of the constitutive model for stainless steel AISI 304

The determined values of material constants for stainless steel AISI 304 are presented in Table 5.1, and the comparisons between numerical integration (plotted with solid lines) and experimental data (plotted with symbols) at different temperatures and strain rates are shown in Figures 5.2 and 5.3. Good agreement has been achieved, which indicates that the proposed constitutive model is capable of predicting the material behaviour of stainless steel AISI 304 during hot deformation process.

<table>
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<th>( K_0 ) (MPa)</th>
<th>( Q_1 ) (J · mol(^{-1}))</th>
<th>( k_0 ) (MPa)</th>
<th>( Q_2 ) (J · mol(^{-1}))</th>
<th>( C_0 ) (s(^{-1}))</th>
<th>( Q_3 ) (J · mol(^{-1}))</th>
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\( B_0 \) (MPa) | \( Q_4 \) (J · mol\(^{-1}\)) | \( E_0 \) (MPa) | \( Q_5 \) (J · mol\(^{-1}\)) | \( \delta_1 \) | \( \delta_2 \) |
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\( n \) | \( A \) |
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<td>3.80</td>
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Figure 5.2 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for stainless steel AISI 304 at strain rate of $1.0 \text{ s}^{-1}$ under different temperatures.

Figure 5.3 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for stainless steel AISI 304 at $1000 \, ^\circ\text{C}$ with different strain rates.
5.2.3.2 Modification of the constitutive model with softening behaviour for HIPed FGH96

Although good agreement has been achieved between calculated and experimental data for stainless steel AISI 304 without the modelling of softening mechanisms, it can be clearly seen from the experimental data of HIPed FGH96 that there is a significant drop in flow stress during the hot deformation process. This is a result of a series of complex interaction and cooperation of recovery and recrystallisation, and the recrystallisation processes are greatly affected by material composition, initial grain size, plastic deformation and process parameters such as temperature, strain, time, etc., therefore a simplified softening model is preferred.

The softening behaviour is similar to damage behaviour, so a typical damage model is used to approximate the softening behaviour of HIPed FGH96 during hot deformation. The damage model is described as (Lin et al., 2005b):

\[ \dot{\omega} = D (1 - \omega)^\varphi \dot{\varepsilon}_p \]  
(5.17)

where \( D \) and \( \varphi \) are material constants, and \( \omega = 1 - \rho_{di}/\rho_d \) is the mobile dislocation damage. Equation 5.8 is now modified with the damage model and the constitutive equations for HIPed FGH96 can be expressed as:

\[ \dot{\varepsilon}_p = \left( \frac{\sigma - R - k}{K} \right)^n \frac{1}{(1 - \omega)^\gamma} \]  
(5.18)

\[ \dot{\rho} = A(1 - \rho)\left| \dot{\varepsilon}_p \right|^{\delta_1} - C \rho^{\delta_2} \]  
(5.19)

\[ \dot{R} = 0.5 B \rho^{-0.5} \]  
(5.20)

\[ \dot{\omega} = D (1 - \omega)^\varphi \dot{\varepsilon}_p \]  
(5.21)

\[ \sigma = E (\varepsilon_t - \varepsilon_p) \]  
(5.22)

where \( \gamma \) is a material constant.
5.2.3.3 Constants of the constitutive model for HIPed FGH96

The material constants were determined using the calibration method mentioned above. The determined values of material constants for HIPed FGH96 are presented in Table 5.2, and the comparisons between numerical integration (plotted with solid lines) and experimental data (plotted with symbols) at different temperatures and strain rates are shown in Figures 5.4 and 5.5. The constitutive model is able to predict both hardening and softening behaviour of the material, showing a rapid flow stress increase initially and a large drop after peak flow stress. There is a good agreement in general, however, some discrepancies are observed. Potential improvement in future may be made by considering recrystallisation during hot compression.

| Table 5.2 Determined material constants within the unified viscoplastic model for HIPed FGH96. |
|-----------------------------------------------|-----------------|---------------|-----------|-----------------|-----------------|-----------------|
| $K_0$ (MPa)                                  | $Q_1$ (J mol$^{-1}$) | $k_0$ (MPa) | $Q_2$ (J mol$^{-1}$) | $C_0$ (s$^{-1}$) | $Q_3$ (J mol$^{-1}$) |
| 1.62$\times 10^{-6}$                          | 2.01$\times 10^5$    | 8.28$\times 10^{-8}$ | 2.33$\times 10^5$ | 1.90$\times 10^3$ | 1.42$\times 10^4$ |
| $B_0$ (MPa)                                  | $Q_4$ (J mol$^{-1}$) | $E_0$ (MPa) | $Q_5$ (J mol$^{-1}$) | $\delta_1$ | $\delta_2$ |
| 1.56$\times 10^2$                            | 1.05$\times 10^4$    | 5.82$\times 10^4$ | 1.00$\times 10^4$ | 0.80 | 2.00 |
| $n$                                          | $A$               | $D$             | $\varphi$ | $\gamma$        |
| 10.00                                        | 10.00             | 0.90            | 1.00      | 28.00            |
Figure 5.4 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for HIPed FGH96 at strain rate of $1.0 \text{ s}^{-1}$ under different temperatures.

Figure 5.5 Comparison of calculated (solid lines) and experimental (symbols) stress-strain curves for HIPed FGH96 at $1000 \degree \text{C}$ with different strain rates.
5.3 Development of powder material model

The consolidation process of FGH96 powder can be seen as the evolution of powder density which is determined by the densification rate. The densification rate is a function of many factors, such as material properties, stress-strain state, volume etc., and is greatly affected by other factors, for example, the initial powder compaction. Therefore, a simplified material model or a representative volume element (RVE) model is usually used to normalise the powder compaction conditions, providing an easier path to build the constitutive model (Cocks, 1989).

5.3.1 Densification rate

A cell model has been proposed to study the densification rate within the reinforced porous materials (Duva et al., 1994). The cell model suggests that the reinforced porous material can be treated as a two-phase material that contains a rigid reinforcing phase and a homogeneous porous creeping phase, which is a relatively simple alternative to the conventional three-phase model. Therefore, the densification rate of the porous material can be predicted by calculating the densification rate of the cell model, which is shown as Figure 5.6. In this model, a rigid circular cylinder fiber with infinite length is surrounded by a shell of porous creeping material, which indicates a plain strain condition. By connecting the constitutive relation between the two phases, the densification rate is then predicted and presented as a function of the reinforcing volume fraction, the material properties and the stress-strain state.
Figure 5.6 A general idea of the cell model. S and T are in-plane compressive loads (Duva et al., 1994).

5.3.2 The modification of constitutive model with relative powder density

The idea of calculating the densification rate by using the two-phase material model can be extended to FGH96 powder. FGH96 powder can be treated as a two-phase material that contains a solid, fully dense, incompressible, power-law phase, for which the constitutive model has been developed, and a macroscopically homogeneous, isotropic, powdered, power-law phase, for which the constitutive relation can be created. The strain rate potential proposed by Duva and Crow is used in this study (Duva et al., 1994).

5.3.2.1 The modification of fully dense material model

The strain rate potential for the fully dense material is expressed as:

\[ P_1 = \frac{\dot{\varepsilon}_0 \sigma_0}{m+1} \left( \frac{\sigma_e}{\sigma_0} \right)^{m+1} \]  \hspace{1cm} (5.23)

where \( \dot{\varepsilon}_0, \sigma_0 \) and \( m \) are material constants, and \( \sigma_e \) is the effective stress defined as:

\[ \sigma_e = \sqrt{\frac{3}{2} S_{ij} S_{ij}} \]  \hspace{1cm} (5.24)
\[ s_{ij} = \sigma_{ij} - \delta_{ij} \sigma_m \]  
(5.25)

\[ \sigma_m = \frac{1}{3} \sigma_{kk} \]  
(5.26)

where \( s_{ij} \) is the stress deviator, \( \sigma_{ij} \) the stress tensor and \( \sigma_m \) the mean stress. \( \delta_{ij} \) is the Kronecker delta. The strain rate tensor \( \dot{\varepsilon}_{ij} \) is then related to \( \sigma_{ij} \) through \( P_1 \):

\[ \dot{\varepsilon}_{ij} = \frac{\partial P_1}{\partial \sigma_{ij}} = \frac{3}{2} \frac{\dot{\varepsilon}_0}{\sigma_0} \left( \frac{\sigma_e}{\sigma_0} \right)^{m-1} s_{ij} \]  
(5.27)

The constitutive model for fully dense material is then modified and described as:

\[ \dot{\varepsilon}_{ij}^p = 3 \frac{\dot{\varepsilon}_0}{2 \sigma_e} \left( \frac{\sigma_e - R - K}{K} \right)^n \frac{1}{(1-\omega)^\gamma} \]  
(5.28)

\[ \dot{\varepsilon}_{pe} = \left( \frac{\sigma_{es} - R - K}{K} \right)^n \frac{1}{(1-\omega)^\gamma} \]  
(5.29)

\[ \dot{\rho} = A (1 - \rho) \left| \dot{\varepsilon}_{pe} \right|^{\delta_1} - C \rho^{\delta_2} \]  
(5.30)

\[ \dot{R} = 0.5 B \rho^{-0.5} \]  
(5.31)

\[ \dot{\omega} = D (1 - \omega)^\rho \dot{\varepsilon}_{pe} \]  
(5.32)

\[ \sigma_{ij} = C_{ijkl} (\varepsilon_{ij} - \varepsilon_{ij}^p) \]  
(5.33)

where \( C_{ijkl} \) is the stiffness tensor and \( \dot{\varepsilon}_{pe} \) is the effective plastic strain rate.

### 5.3.2.2 The modification of powder material model

The strain rate potential and strain rate tensor for the powder material are described as:

\[ P_2 = \frac{\dot{\varepsilon}_0 \sigma_0}{m+1} \left( \frac{\sigma_{es}}{\sigma_0} \right)^{m+1} \]  
(5.34)
\[ \dot{\varepsilon}_{ij} = \frac{\dot{\varepsilon}_0}{\sigma_0^m} \sigma_{es}^{m-1} \left( \frac{3}{2} a s_{ij} + \frac{1}{3} b \delta_{ij} \sigma_m \right) \]  \tag{5.35}

where \( \sigma_{es} \) is an ‘effective’ effective stress which is affected by the powder density evolution through coefficients \( a \) and \( b \):

\[ \sigma_{es} = \sqrt{a \sigma_e^2 + b \sigma_m^2} \]  \tag{5.36}

Coefficients \( a \) and \( b \) are powder density dependent parameters and described as:

\[ a = \frac{1 + \frac{2}{3} (1 - \rho)}{\rho \sigma_m^2} \]  \tag{5.37}

\[ b = [m(1 - \rho)(1 - (1 - \rho)\frac{1}{m})]^2 \sigma_m^2 \times \left[ \frac{3}{2m(1 - (1 - \rho)\frac{1}{m})} \right]^2 \]  \tag{5.38}

where \( \rho \) is the relative density of the powder compact and ranges from 0 to 1. When the powder is fully consolidated, as \( \rho = 1 \), then \( a = 1, b = 0 \) and \( \sigma_{es} \) reduces to \( \sigma_e \), and the material exhibits the behaviour of fully dense material.

According to Duva and Crow, the ‘effective’ effective strain rate of powder material is related to the ‘effective’ effective stress by \( P_2 \), which can be expressed as:

\[ \dot{\varepsilon}_{ee} = \frac{m + 1}{\sigma_{es}} P_2 \]  \tag{5.39}

Since \( \sigma_{es} \) is determined in Equation 5.36, the \( \dot{\varepsilon}_{ee} \) is then affected by powder density coefficients \( a \) and \( b \), and can be given as:

\[ \dot{\varepsilon}_{ee} = \sqrt{\frac{\dot{\varepsilon}_e^2}{a} + \frac{\dot{\varepsilon}_m^2}{b}} \]  \tag{5.40}

\[ \dot{\varepsilon}_e = \sqrt{\frac{2}{3} \dot{\varepsilon}_{ij} \dot{\varepsilon}_{ij}} \]  \tag{5.41}

\[ \dot{\varepsilon}_{ij} = \dot{\varepsilon}_{ij} - \frac{1}{3} \delta_{ij} \dot{\varepsilon}_m \]  \tag{5.42}
where $\dot{e}_e$ is the effective strain rate, $\dot{e}_{ij}$ the strain rate deviator and $\dot{e}_m$ the mean strain rate.

Similar to $\sigma_{es}$, when relative density reaches 1, $\dot{e}_{ee}$ reduces to $\dot{e}_e$.

The dilatation rate $\dot{e}_{dr}$ determines the densification rate, which is defined as:

$$\dot{e}_{dr} = \dot{e}_m = \sqrt{b\dot{e}_{ee}^2 - \frac{b}{a}\dot{e}_e^2}$$  \hspace{1cm} (5.43)

and is related with $\dot{\rho}$ as:

$$\dot{\rho} = -\rho \dot{e}_{dr}$$ \hspace{1cm} (5.44)

Equation 5.44 is used to model the powder density evolution. Thus, the constitutive model for powder material is then modified and described as:

$$\dot{e}_{ij}^p = \frac{3}{2\sigma_e}\left(\frac{\sigma_{es} - R - k}{K}\right)^n \frac{1}{(1-\omega)^{\nu}} \left(\frac{3}{2}aS_{ij} + \frac{1}{3}b\delta_{ij}\sigma_m\right)$$ \hspace{1cm} (5.45)

$$\dot{\rho} = -\rho \dot{e}_{dr}$$ \hspace{1cm} (5.46)

$$a = \frac{1 + \frac{2}{3}(1-\rho)}{\rho^{m+1}}$$ \hspace{1cm} (5.47)

$$b = \left[m(1 - \rho)\left(1 - (1 - \rho)^{\frac{1}{m}}\right)\right]^\frac{2}{m+1} \times \left[\frac{3}{2m(1 - (1 - \rho)^{\frac{1}{m}})}\right]^2$$ \hspace{1cm} (5.48)

$$\dot{\bar{\rho}} = A(1 - \bar{\rho})|\dot{e}_{pe}|^{\delta_1} - C\bar{\rho}^{\delta_2}$$ \hspace{1cm} (5.49)

$$\dot{\bar{R}} = 0.5B\bar{R}^{-0.5}\dot{\bar{\rho}}$$ \hspace{1cm} (5.50)

$$\dot{\omega} = D(1 - \omega)^\theta \dot{e}_{pe}$$ \hspace{1cm} (5.51)

$$\sigma_{ij} = C_{ijkl}(\varepsilon_{ij} - \varepsilon_{ij}^p)$$ \hspace{1cm} (5.52)
5.3.3 Calibration of the powder material constitutive model

There are three new material constants introduced in the modified fully dense and powder material model. The determination of $\dot{\epsilon}_0$, $\sigma_0$ and $m$ can be achieved by using calculated data obtained from unmodified Equations 5.18 to 5.22 and a rearranged form of Equation 5.26.

Considering uniaxial compression process, the Equation 5.27 can be expressed as:

$$\dot{\epsilon} = \frac{\dot{\epsilon}_0}{\sigma_0^m} \cdot \frac{3}{2} \sigma^{m-1} \cdot \frac{2}{3} \sigma$$

which is

$$\frac{\dot{\epsilon}_0}{\sigma_0^m} = \frac{\dot{\epsilon}}{\sigma^m}$$

The term $\frac{\dot{\epsilon}_0}{\sigma_0^m}$ is a constant, therefore the value calculated by $\frac{\dot{\epsilon}}{\sigma^m}$ would be constant as well.

In order to determine these constants, a spreadsheet is created and a set of initial values of the material constants are entered, then a curve of the calculated values is plotted against strain values. By adjusting the material constants, the result that best features Equation 5.54 can be used to determine the material constants.

The determined values of material constants for modified fully dense and powder material model for FGH96 are presented in Table 5.3, and an example of stress-strain curve validation and powder density evolution of the powder material model are demonstrated in Figures 5.7 and 5.8.
Table 5.3 Determined material constants within the powder material model for HIPed FGH96.

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<td>$10.00$</td>
<td>$10.00$</td>
<td>$0.90$</td>
<td>$1.00$</td>
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<tr>
<th></th>
<th>$\sigma_0$</th>
<th>$m$</th>
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<td>$5.00\times10^{2}$</td>
<td>$10.00$</td>
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Figure 5.7 Validation of modified powder material model by calculated data obtained from unmodified constitutive model for FGH96 at 1050 °C with strain rate of 1.0 s$^{-1}$. $\rho_0$ is the initial relative powder density.
Figure 5.7 shows a comparison of the calculated stress-strain curves with and without powder density evolution during uniaxial hot compression process and Figure 5.8 represents density evolution during the consolidation process. With the modification of powder density evolution, the initial flow stress of FGH96 powder is significantly lower than that of fully dense FGH96 material, which reflects the lower deformation resistance of powder material compared to fully dense material. As powder density increases, the flow stress of powder material rises rapidly, indicating the consolidation process takes place and the material becomes stronger as the volume fraction of consolidated powder increases. When the relative powder density reaches 1 at the strain of 0.532, the FGH96 powder is fully consolidated, therefore the material shows the mechanical behaviour of fully dense material and the flow stress is the same as that of fully dense material. It can be concluded that the modified constitutive model for FGH96 powder is capable of predicting the behaviour of powder density evolution during hot deformation process.
5.4 Summary

Dislocation evolution has been identified as the dominant deformation mechanism during the plastic deformation stage, while recovery and recrystallisation have been the main softening mechanisms for FGH96 and caused the large drop in flow stress. Based on the understanding of hot deformation mechanisms, the unified viscoplastic constitutive model for fully dense FGH96 and stainless steel AISI 304 has been successfully developed. The hardening has been well modelled for fully dense FGH96 material, while the modelling of softening due to recovery and recrystallisation has been simplified and modified using a damage parameter. The constitutive models have been calibrated using experimental data obtained from Gleeble test reported in the previous chapter. Material constants have been determined, and numerical integration results have been presented. Good agreement has been achieved between numerical integration results and experimental data for stainless steel AISI 304 and fully dense FGH96. For DPF process, the fully dense material model has been modified with powder density evolution to predict the material response of FGH96 powder. Promising results have been obtained, indicating the feasibility of implementing modified powder material model in FE simulation.
Chapter 6

FE Simulation

6.1 Introduction

This chapter presents the FE model built for the simulation of FGH96 powder in DPF process. The model was created in the commercial FE simulation software DEFORM 2D/3D and implemented with the developed constitutive equations using user defined subroutines. Contents discussed in this chapter are:

1) Implementation of fully dense material model: a 2D FE model is created with the fully dense material model. The 2D FE model is used for the verification to guarantee a successful implementation of the constitutive equations;

2) FE simulation with powder density evolution: a 3D FE model is built based on the feasibility experiments and implemented with the powder material model. The user defined subroutines are modified with the powder densification model. FE simulation is then carried out to predict the distribution of effective stress and mean stress, as well as density distribution of DPFed test-piece. The density distribution is validated by experimental data. The powder density evolution and stress-strain state are predicted and analysed, and the effects of forming speed on powder consolidation are investigated by the FE simulation.

6.2 Implementation of fully dense material model

The constitutive model for fully dense FGH96 was implemented into DEFORM 2D/3D via user defined subroutines USRMTR and USRUPD. The USRMTR was used to calculate the flow stress, while USRUPD were responsible for updating state variables, such as dislocation density, isotropic hardening, etc. Figure 6.1 shows the communication paths within the DEFORM 2D/3D software.
To verify the implementation of the fully dense material model for FGH96, a one-element axisymmetric 2D model was built to be compatible with the uniaxial compression test. The geometry of the model was set to be the same as that of the test-piece used in the uniaxial compression test, which was a cylinder with 8 mm in diameter and 12 mm in height. The FE model is axisymmetric, and the top and bottom dies were assumed to be semi-infinite and defined as rigid body. Figure 6.2 presents the FE model used for the verification simulation.

![Diagram](image)

**Figure 6.1** The communication paths within DEFORM 2D/3D software.

To verify the implementation of the fully dense material model for FGH96, a one-element axisymmetric 2D model was built to be compatible with the uniaxial compression test. The geometry of the model was set to be the same as that of the test-piece used in the uniaxial compression test, which was a cylinder with 8 mm in diameter and 12 mm in height. The FE model is axisymmetric, and the top and bottom dies were assumed to be semi-infinite and defined as rigid body. Figure 6.2 presents the FE model used for the verification simulation.
The total stroke for this verification simulation was set to be 6 mm, with a strain rate of 1.0 s\(^{-1}\) at 1000 °C. To simplify the simulation, some assumptions were made as follow:

1) No heat loss or heat source within the test-piece during the simulation process;
2) Friction coefficient was assumed to be uniform and constant throughout the deformation. Friction coefficient was set to 0.3 on the interfaces of the test-piece and both top and bottom dies.

The simulation results of strain and stress distributions are presented in Figure 6.3. As there is friction between the die and powder compact, some barrelling phenomena occurred, which was also observed in experiments. The strain-stress data were then obtained from the simulation and compared with numerical integration data. Comparison is shown in Figure 6.4. It could be seen that good agreement has been achieved between numerical and simulation results, which indicated that the fully dense material model has been correctly introduced into DEFORM and could be used to perform FE simulations for FGH96.
Figure 6.3 The strain and stress distributions for the verification simulation at different progression percentages: a) 33%, b) 66%, and c) 100% of the total process progression.
Figure 6.4 Comparison of strain-stress curves between numerical integration data (solid curve) and FE simulation data (symbols) for the fully dense FGH96 with a strain rate of 1.0 s⁻¹ at 1000 °C.

6.3 Implementation of powder density evolution

Plastic deformation dominates the DPF process because of the high deformation speed and short forming time. During the process, the powder material may be treated as a continuous medium and the constitutive equations that describe the deformation of the powder materials can be obtained by modification of plastic theory for solid materials (Duva et al., 1994; Ghorbanbeigi et al., 2016). Therefore, the modelling of powder density evolution for FGH96 powder in DPF process was carried out with the modification of fully dense material model by defining the powder densification in DEFORM porous material model and introducing the FGH96 powder material model developed in Chapter 5.

Based on the feasibility experiments, a 3D FE model was built to represent the process. Only half of the test-piece was modelled in the simulation and a quarter of the test-piece was analysed because of symmetry. Tetrahedral elements were used for container and FGH96 powder. The top and bottom dies were chosen to be the same as those used in the
simulation of fully dense material model. Figure 6.5 demonstrates the 3D model established in DEFORM.

![Figure 6.5 The 3D FE model used in the simulation of DPF process.](image)

To be compatible with the feasibility experiments, the simulation was carried out at 1150 °C, and the total stroke for this simulation was set to be 14.5 mm, which produced a test-piece with the thickness of 5.5 mm. Some test conditions and assumptions were as follow:

1) Effective thermal conductivity for fully dense FGH96 material and stainless steel container at 1150 °C were 28.75 and 22 W/m K respectively;

2) No heat loss or heat source within the test-piece, and the heat transfer coefficient between the test-piece and dies was 20 kW/m² K;

3) The initial relative powder density was set to 0.7;

4) The friction coefficient on the interfaces of both powder/container and container/dies were set to 0.3 and remained constant throughout the process.
6.3.1 Stress distribution

Figure 6.6 demonstrates the distributions of effective stress and mean stress for FGH96 powder at different progression percentages of the simulation. The progression percentage is the percentage of the current time from beginning of the process over the total time of the process. Four typical progression percentages were selected and analysed for the stress distribution during DPF process: 25%, 50%, 75% and 100% of the total process time.

The distribution of effective stress shows an obvious decrease as the locations are away from the centre of the test-piece at all progression percentages. For progression stage d, 5 locations were chosen as P1 to P5, and the effective stress will be used to calculate the stress triaxiality factor.

The mean stress determines the powder consolidation. The negative values of the mean stress show the compressive deformation that would promote the volume reduction and the consolidation of powder particles, hence increasing material density (Timothy et al., 2016). It can be seen that the mean stress maintains negative at locations P1 to P4, while mean stress at location P5 exhibits positive values at stages a to c and only becomes negative at the end of the process, which would result in a much lower material density.
Figure 6.6 Distribution of stresses for FGH96 powder during DPF process at the progression percentages of: a) 25%, b) 50%, c) 75% and d) 100%. P1 to P5 indicate the locations chosen for analysis.
6.3.2 Material density distribution

Figure 6.7 compares the material density distribution between experimental data and FE simulation data after the DPF process. Twenty FE simulation results were used to plot the solid curve shown in Figure 6.7, together with the experimental data obtained from the feasibility experiments. Good agreement has been achieved between these two sets of data, and the features of material density distribution were compatible with the characteristics of mean stress distribution. As the mean stress near the rim location of the test-piece changes from positive values to negative values only at later stages of the process, it hinders the consolidation of powder particles at such positions and results in the relatively low material density. In addition, the good agreement between experimental data and FE simulation data indicates the feasibility of predicting material density in DPF process using the powder material model.

Figure 6.7 Comparison of material density distribution between experimental data (symbols) and FE simulation data (solid line with symbols).
6.3.3 Stress triaxiality factor

It has been well documented that the stress triaxiality factor is one of the key aspects that can explain the annihilation of pores in porous materials (Danas et al., 2012). The stress triaxiality factor $\eta$ is the ratio of mean stress to effective stress and can be expressed as:

$$\eta = \frac{\sigma_m}{\sigma_e} \quad (6.1)$$

This indicates that when the hydrostatic stress is compressive, the stress triaxiality factor is negative and therefore promotes the reduction of porosity and the increase of powder density.

Figure 6.8 compares the stress triaxiality factor, effective strain and relative powder density at five different locations during the DPF process. As shown in Figure 6.6, the value of mean stress for locations P1 to P4 remains negative, therefore by calculating the Equation 6.1, the stress triaxiality factor for these locations is negative throughout the process, which indicates that the compressive deformation takes place and results in a quick climb in the relative powder density, as shown in Figure 6.8. On the other hand, at the rim position of the test-piece, i.e. location P5, the stress triaxiality factor starts at positive values and continues to be positive until the later stage of the process at 94%, where the factor drops below 0 and the relative powder density begins to increase at a much higher rate.

It can be concluded from the FE simulation results that the powder density evolution is considerably affected by the stress triaxiality factor and the effective strain. Locations with a large negative value of stress triaxiality factor experience a faster powder consolidation process, and larger effective strains are also beneficial for the elimination of porosity. Locations of P1 and P2 exhibit a faster increase of relative powder density compared to locations of P3 to P5, whereas P1 shows a more consolidated condition than P2 because of the larger effective strain.
Figure 6.8 Evolution of stress triaxiality factor, effective strain and relative powder density at five locations P1 to P5 as shown in the figure.
6.3.4 Effect of forming speed on powder density

Very short forming time is one of the most distinguished features of DPF process. For a fixed stroke, the strain rate is mainly determined by the forming time, which in practical applications is reflected by the forming speed. According to the powder density material model for FGH96, the densification rate is a function of stress-strain state, which suggests that forming speed will affect the consolidation of the powder particles. In addition, the forming speed is limited by the design of the forming equipment and is a process parameter that can be controlled under production conditions. Therefore, the effect of forming speed on powder densification is investigated through FE simulation, where other process parameters, e.g. temperature and stroke, remain constant.

Figure 6.9 presents the powder density distribution with different forming speed. The forming speeds of 75, 100 and 200 mm/s were selected. The correlation between forming speed and process time is presented in Table 6.1.

<table>
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<tr>
<th>Forming speed (mm/s)</th>
<th>Process time (s)</th>
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<tr>
<td>75</td>
<td>0.193</td>
</tr>
<tr>
<td>100</td>
<td>0.145</td>
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<tr>
<td>200</td>
<td>0.0725</td>
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It can be seen from the results that the relative powder density of the DPFed components shows a significant drop near the rim locations with the increase of forming speed, and the test-piece processed at 200 mm/s forming speed is not fully consolidated. Despite the simplified simulation conditions, such as constant friction coefficient, this can be attributed to the very high forming speed that leads to a high strain rate hardening effect, as shown in Figure 5.5. The great increase in the flow stress results in more difficulties in
consolidating powder particles. It can be observed that the thickness of the final powder components with higher forming speed is slightly greater than that of the components with lower forming speed, possibly due to less viscoplastic deformation in the very short time, which indicates that full consolidation cannot be acquired by simply increasing the forming speed when all other conditions are kept the same. On the other hand, more recrystallisation may take place and the microstructure of the components can be refined when higher forming speed is applied.

In this case, a forming speed within the range of 75 mm/s to 100 mm/s is preferred with the consideration of both powder consolidation and material properties. However, in practical applications, the forming speed is highly dependent on the design of the process equipment. Therefore, it is concluded that a reasonable range for forming speed needs to be determined, and the powder material model and FE simulation can be very helpful to achieve this goal.
Figure 6.9 FE simulation results of powder density distribution obtained at different forming speed:
a) 75 mm/s, b) 100 mm/s, and c) 200 mm/s.
6.4 Conclusions

In this chapter, an FE model for DPF process was established in the DEFORM 2D/3D software to predict and analyse the material behaviour of FGH96 powder, especially the powder density evolution. The model was first implemented with fully dense material model by using user defined subroutines, then validated by experimental data, followed by the modification of densification rate to model the powder density evolution. The model was proved to be capable of predicting material behaviours of FGH96 powder in DPF process, which provides useful feedbacks and suggestions on the designing of the process. Major conclusions from this chapter are drawn as follow:

1) Fully dense components can be acquired from DPF process under certain conditions. Fully consolidated material can be achieved within the component, while only the locations near the rim of the test-piece do not reach full density;

2) The powder density distribution has a similar pattern to the mean stress distribution. The influence of stress triaxiality factor is dependent on its signs: negative values of the factor indicate compressive deformation that promotes powder consolidation. Higher effective strain also contributes to the increase of relative powder density;

3) Increasing forming speed and hence the strain rate is good for material properties but impacts the powder consolidation. Higher forming speed leads to higher strain rate hardening effect that results in higher flow stress and more difficulties to consolidate the powder;

4) A reasonable range for the forming speed has been suggested for a specified DPF condition. It can be also concluded that the determination for other process parameters can be performed with this FE model.
Chapter 7

Conclusions and Future Work Suggestions

7.1 Conclusions

The main purpose of this research was to develop the DPF process for industrial applications and investigate the powder density evolution of FGH96 powder during hot deformation. Experiments were carried out to study the key factors in DPF process, material characteristics of FGH96 superalloy and mechanical properties of components manufactured by DPF process. Numerical methods were utilised to establish the constitutive model for fully dense FGH96 material, and modification was made to the fully dense material model for powder material considering powder density evolution and was implemented for numerical analysis and FE simulations. This chapter summarises the main conclusions drawn from this research.

7.1.1 Conclusions from experimental studies

Four sets of experiments, i.e. uniaxial compression tests, small size powder forging tests, laboratory feasibility tests and industrial trials, have been conducted on FGH96 materials, and subsequent microstructural observations have been carried out on specimens manufactured by FGH96 powder to examine microstructural conditions. The conclusions are drawn as follow:

1) The stress-strain curves of fully dense FGH96 material from uniaxial compression tests suggest that FGH96 is an elastic-viscoplastic material whose flow stress reduces with the increase of temperature and/or decrease of strain rate. In addition, softening behaviours of the material occurs when strain reaches a certain level, due to dynamic recovery and recrystallisation.

2) Acquirement of fully dense FGH96 specimens from small size powder forging tests, laboratory feasibility tests and industrial trials indicates that DPF process can produce fully dense components.
3) The material density and mechanical properties of the DPFed FGH96 specimens are very close to and even slightly better than those of HIPed FGH96 materials, which suggests that DPF process meets the basic requirements for PM applications.

4) The microstructure of FGH96 components, especially the PPB networks, is improved by DPF process. Applying large compression stress and plastic deformation contributes to the break-up of PPB networks, in addition to recrystallisation.

5) In industrial trial, the microstructure of DPFed component shows a heterogeneous and incompletely recrystallised microstructure due to very short deformation time.

7.1.2 Conclusions from numerical studies

1) A constitutive model has been developed for the fully dense FGH96 material, which takes into account the evolution of dislocation density, isotropic hardening and softening. Good agreement is achieved with experimental stress-strain curves.

2) Dislocation evolution is considered to be the dominant deformation mechanism during the plastic deformation and forms the model of isotropic hardening. It is also used to represent the softening behaviours for FGH96 with the introduction of mobile dislocation damage model, however, recovery and recrystallisation are suggested to be the main softening mechanisms.

3) A 2D verification model is used to examine the implementation of fully dense material model, which is confirmed by the good agreement between numerical integration and FE simulation results.

4) The powder material model is developed by the modification of fully dense material model with the introduction of powder density evolution using Duva’s porous material model, which is used to predict the material response during DPF process.
5) A 3D FE model is established with the implementation of powder material model. Results confirm that fully dense components can be acquired from DPF process under certain process conditions.

6) The powder density is dependent on the stress and strain history. The powder consolidation is promoted by the negative stress triaxiality factor and increased effective strain.

7) Forming speed is a crucial factor for the powder consolidation and material properties. Within the range of 75 mm/s to 200 mm/s, a lower forming speed is beneficial for the powder consolidation, however, higher forming speed is obviously more efficient in production. A reasonable forming speed can be determined from FE simulation.

8) The FE model is capable of predicting the material response for FGH96 powder in DPF process and is a useful tool for the determination of process parameters and industrial applications.

### 7.2 Future work suggestions

As a newly developed PM process, DPF process exhibits some advantages over traditional HIPing process for manufacturing net-shape or near net-shape components, while significantly improves the efficiency by reducing the process time to only a few seconds and the microstructure by introducing more recrystallisation. On the other hand, there are a few aspects that need to be considered for industrial applications. Further investigations and modifications are suggested as follow:

1) Mechanical properties of a single FGH96 powder: in this research, the mechanical properties of fully dense FGH96 superalloy is used to approximate a single FGH96 powder. It is highly recommended to perform compression tests on a single powder at elevated temperatures to identify the different deformation behaviours between powder and fully dense material. This will also contribute to the development of the powder material model.

2) Complex shape components: all experiments conducted in this research are designed for a simple cylinder shape test-piece to study the powder
compaction during DPF process. For further investigations, complex shape components, for example a ring component or an irregular polygon component, are proposed to be tested under both laboratory and industrial circumstances to investigate the powder consolidation condition in a more complicated stress-strain state.

3) Die and tool-set design: the applied forces in both experimental tests and FE simulations are parallel to the axis of the test-piece and in only one direction. When no constraint is applied to the rim, results show a relatively low powder density in the near rim locations of the test-piece. On the other hand, when constraint is applied, the powder consolidate is considerably improved. This needs to be taken into consideration for the design of the die and tool-set for DPF process.

4) Critical values for process parameters: it is believed that there exists a set of critical values for process parameters, i.e. temperature, loading force and holding time, to produce fully dense components. This will be of great importance to the design of DPF process and provide a valuable guidance to the industrial applications.

5) Softening mechanisms: the softening behaviour of FGH96 is modelled with a simplified damage equation in this research. Although a good agreement is achieved by the current model in the conditions investigated, it is recommended to modify the current empirical model to mechanism-based model by studying static/dynamic recovery and recrystallisation, which will further improve the accuracy of the material model and better describe the microstructure evolution.

6) Powder density evolution: the powder density evolution is currently predicted with Duva’s porous material model, which models the powder consolidation on a macro-scale level but could not explain the microstructural evolution within the powder material. A microstructural mechanism-based powder density evolution model should be developed for further implementation.
References


INSG (2013) INSG secretariat briefing paper.


