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**Characterisation of High Temperature Coatings by
Miniature Specimen Testing and Inverse Approaches**

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Abstract

Coatings, e.g. thermal barrier coatings (TBCs), have been widely used for many high temperature engineering applications to protect the components from such severe operating environments. An improved understanding of the mechanical behaviour of the coatings is required for assessing the reliability and performance of the critical components as well as developing novel TBCs.

Inverse methods were developed based on nonlinear optimisations to acquire the mechanical properties from experimental results in conjunction with analytical solutions or finite element (FE) modelling. They were proved to be capable of determining the elastic-plastic and creep properties of materials from various experimental data with different material constitutive models. For example, it was demonstrated that the inverse method was able to converge to the optimised mechanical properties within 5% error compared to the predefined values, taking the load-displacement curves and the surface profile of the indented specimen for the multi-objective optimisation. Some issues encountered during the development process, such as the scaling of the optimised quantities and the loading paths applied for FE modelling, were also investigated.

Theoretical studies were carried out for a two-material system (coating-substrate) regarding the individual and compound elastic-plastic/creep behaviour. The inverse method was adopted and demonstrated to be able to extract the material properties of an unknown material based on the known material behaviour and the experimentally determined compound material behaviour.

Experiments were carried out for a P91 steel (used as the substrate material for a coated system) and a thermally sprayed CoNiCrAlY coating (typically used as the bond coating of TBCs) using existing miniature specimen testing methods, such as the small punch tensile (SPT) and creep (SPC) tests and micro/nano-indentation tests. The experiments were carried out for the

temperature range from room temperature (RT) up to 700°C. The ductile-to-brittle transition temperature (DBTT) of the CoNiCrAlY was studied examining the variation of the mechanical properties, ductility and the failure patterns. The fracture surfaces of the tested SPT specimens of the CoNiCrAlY coating were studied using scanning electron microscopy (SEM). Brittle fracture was observed at temperatures from RT to 500°C while typical ductile fracture was observed at 700°C. Significant scattering was observed for the SPT tests at lower temperatures, indicating that the small punch test was less effective as a mechanical testing method for brittle materials due to early cracking and the complex multi-axial stress state during the deformation process.

A novel two-material miniature specimen testing method was developed for the applicable scale of coatings (total thickness of 0.5mm, minimum coating thickness of 0.15mm for the coating-substrate specimen). Single-material miniature tensile and creep tests were carried out for a P91 steel and the same CoNiCrAlY coating used for the SPT tests. Good agreements were achieved between the SPT and miniature tensile tests regarding the DBTT of the CoNiCrAlY coating. It should be noted that excellent consistency and repeatability were observed for the miniature tensile testing results at all temperatures, suggesting that this miniature testing method had the potential to become a universally applicable technique considering that it was also easier to interpret because the stress state in the uniform section was close to that in the conventional uniaxial tensile testing specimen. The load-displacement data obtained from the miniature tensile tests were used to extract the elastic-plastic properties of the CoNiCrAlY coating using the inverse method. The characterised stress-strain behaviours were applied for the FE modelling of SPT tests and some representative behaviours were obtained compared to the corresponding experimental results, such as the early cracking at the lower temperatures and the location of the ductile fracture at higher temperatures.

The results of this study provide miniature specimen characterisation methods with great potential for the applications for coatings. The numerical and experimental work has set a solid foundation for the future work of two-material and multi-material systems, e.g. characterisation of the elastic-plastic and creep behaviour of a coated system consists of a CoNiCrAlY coating and the substrate of a P91 steel or other coating-substrate systems.

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Nomenclature

A	Projected area of the indenter, stress constant, Creep damage model parameter, yield stress at reference temperature (Johnson-Cook constitutive model), steady-state creep constant
B	Creep damage model parameter, Johnson-Cook constitutive parameter
C	Curvature of the loading curve of indentation test, Johnson-Cook constitutive parameter
c, c_1, c_2	Scaling coefficients for the optimisation function
E, E_1, E_2	Elastic moduli
F	Force
F_e	Elastic-plastic yield load
h	Displacement
h_1, h_2	Thicknesses
H	Hardness
K	Strength coefficient
L	Length
n	Strain-hardening exponent, steady-state creep constant
N	Creep damage model parameter
P	Force
q_2	Creep damage model parameter
S	Initial unloading contact stiffness
S_1, S_2	Cross-sectional areas
t, t_f	Time, time to failure (creep)
t_0	Thickness of the specimen
T, T_r, T_m	Temperature, reference temperature and melting temperature, respectively
u, u_1, u_2	Displacements

α	Creep damage model parameter
β, η	Reference stress conversion factors
ε	Strain
$\dot{\varepsilon}$	Strain rate
$\dot{\varepsilon}^c$	Creep strain rate
θ	Indenter half angle
ν	Poisson's ratio
σ	Stress
σ_y	Yield stress
σ_{EQ}	Equivalent stress
χ	Creep damage model parameter
ϕ, ψ	Pre-straining parameters
$\omega, \omega_1, \omega_2$	Creep damage parameters
DBTT	Ductile-to-brittle transition temperature
FE	Finite element
HAZ	Heat-affected zone
HVOF	High velocity oxy-fuel
LVDT	Linear variable displacement transducer
NDT	Non-destructive testing
RT	Room temperature
SEM	Scanning electron microscopy
SPC	Small punch creep
SPT	Small punch tensile
TBC	Thermal barrier coating
TPB	Three-point bending

Chapter 1 - Introduction

1.1 Background

A high temperature environment is a common operating condition for many engineering applications, such as power plants, chemical plants, gas turbines and aero-engines. In many cases, it is desirable to assess the material behaviours and predict the future performance of certain components or structures for economic efficiency and risk management. For instance, creep strain and damage, which occur at elevated temperatures for extended periods of time, could lead to catastrophic failure of critical components or structures. To protect the engineering components and structures from the high temperature environment, coatings, e.g. thermal barrier coatings (TBCs), are widely applied to the surfaces to protect the oxidation damage and to lower the operating temperature of the substrate. The knowledge of the material behaviours of the coatings is crucial for evaluating and predicting the integrity and reliability of the engineering components. Due to the relatively small thicknesses of the coatings, conventional mechanical testing techniques, such as uniaxial tensile tests, are not suitable in this case. Non-destructive small specimen testing techniques have clear advantages under the stated conditions since the amount of material required for testing is small. There are different types of small specimen testing methods, such as micro-indentation testing or nano-indentation testing, small punch testing, small ring testing and two-bar testing and so on. Each type of these testing techniques has its own advantages and limitations. The choice of suitable testing methods should be made based on the environment, available amount of material and requirements for the sample geometry and output data. Inverse methods have been employed in many studies to characterise material behaviours in conjunction with various experimental methods. One of the strengths of inverse methods is that it is easy to implement, even for investigators with limited theoretical knowledge of the testing techniques. Also, most factors of influence are taken into

consideration by inverse methods since they are entirely based on the measurable experimental quantities. However, it also means that the validity of inverse methods greatly depends on the accuracy and consistency of the physical experimental data.

1.2. Aims and Objectives

This study aims to investigate the applicability of some existing small specimen testing techniques, i.e. indentation test and small punch tensile/creep test, for the characterisation of coatings. A novel miniature specimen testing method is to be developed to accurately acquire the elastic-plastic and creep behaviour of coatings. In addition, an inverse method framework is to be developed and applied to these testing methods to acquire the elastic-plastic and creep constitutive properties of the materials.

In summary, the objectives of the study include:

- 1) Develop an inverse approach to determine the mechanical properties of a material from the target data sets, obtained theoretically or experimentally and apply the inverse method to the indentation testing results obtained from experiments.
- 2) Develop a novel miniature specimen tensile and creep testing method for thin coatings and coating-substrate applications.
- 3) Carry out single-material tensile and creep tests on an industrial coating material (CoNiCrAlY) and a power plant steel pipe (P91) using the developed miniature specimen testing technique and compare to the uniaxial tests and small punch tests.
- 4) Develop an inverse method to determine the mechanical properties of the coating material of interest from a two-material system.

5) Determine the mechanical properties of the coating and the steel using the small specimen testing results in conjunction with the inverse method.

6) Carry out two-material (CoNiCrAlY coating-P91steel) small specimen tensile and creep tests and determine the material properties of the coating from the compound experimental results.

1.3. Thesis Outline

Chapter 1: the general introduction to the background and objectives of this study and the structure of the thesis.

Chapter 2: the literature review, including the state-of-the-art in material characterisation methods based on the miniature specimen testing techniques, such as the micro/nano-indentation testing, SPT testing and SPC testing, as well as the applications of inverse methods in the relevant studies.

Chapter 3: description of the numerical and experimental techniques used in this study. The numerical techniques include the FE modelling and the inverse approach involving nonlinear optimisation. The experimental techniques include the SPT and SPC testing, the micro/nano indentation testing, the novel miniature specimen testing and the SEM technique.

Chapter 4: development and applications of the inverse method. Some key factors of influence, such as the choice between the load-controlled and displacement-controlled FE models and the scaling coefficient for the multi-objective optimisation function. The applications of the inverse method to the theoretical indentation tests and realistic experimental SPC testing are also presented.

Chapter 5: a novel miniature specimen testing technique and the characterisation methods developed for the two-material systems. The inverse method is also applied for the characterisation of the two-material system.

Chapter 6: characterisation of a heat-treated P91 steel using the miniature tensile and creep testing. The heat treatment is performed for the specimens to replicate that typically received by the thermal barrier coatings during the manufacturing process. The heat-treated P91 steel will be used for the future two-material coted specimen experiments as the substrate material.

Chapter 7: characterisation of a thermally sprayed CoNiCrAlY coating using the SPT and miniature tensile testing. The experimental results from these two different testing methods are compared and discussed. This CoNiCrAlY coating will be used for the future two-material coated specimen experiments as the coating material.

Chapter 8: a summary of the conclusions of the study and the suggested and planned future work from the aspects of a) experiments and b) inverse method.

Chapter 2 - Literature Review

This chapter presents the literature review on bond coating of the thermal barrier coating (TBC) system, material characterisation using small specimen testing techniques and inverse method applications.

Bond coating is a critical part of the TBC system. It is important to have a good understanding of the mechanical properties of the bond coating in order to assess the overall integrity of the TBC system. Due to the small thickness of the coatings, conventional mechanical testing techniques are of very limited applicability in this case.

There are various types of small specimen testing techniques, such as the impression testing, the small ring testing, the small two bar testing, the indentation testing and the small punch testing. Regarding the applicability for coatings, the specimens for the indentation testing and the small punch testing have the advantages of small thickness (consider the typical case of thin coatings on a substrate material) and lower difficulty in manufacturing (it is more difficult to make specimens of complex geometries using coating materials due to their relatively lower ductility and higher hardness). Therefore, the focus of this literature review is on these two types of small specimen testing methods.

Inverse method could be a powerful tool for material characterisation when the interpretation of the experimental data is difficult, e.g. it is difficult to correlate the small punch testing data to the conventional uniaxial testing data. It is also useful for novel testing techniques with limited theoretical or empirical understanding.

2.1. Bond Coating of Thermal Barrier Coating System

Multiple layers of coatings are deposited to the surfaces of components to form the thermal barrier coating (TBC) to protect the components from the high temperature, e.g. the performance of the gas turbine is directly affected by the maximum operating temperature [1]. A TBC usually consists of three layers: 1) a ceramic top coating, e.g. Y_2O_3 stabilised ZrO_2 ; 2) a bond coating of MCrAlY alloy bond coating between the substrate alloy and the top coating, where M = Ni or Co; and a thin oxide layer forms between the top and bond coatings known as the thermally grown oxide (TGO) [2-4].

Bond coating is a critical part of the TBC system as it keeps the top coating attached to the component surface. It is crucial to have a good understanding of the mechanical behaviour of the bond coating, such as the ductility, plasticity and creep behaviour [4, 5].

Due to the small thickness of the coating, conventional mechanical testing techniques, such as the uniaxial tensile and creep testing, are not applicable. Small specimen mechanical tests are performed for coatings, e.g. micro tensile testing [6-8], small punch tensile testing [9] and small punch creep testing [10].

2.2. Material Characterisation Using Indentation Tests

2.2.1. Background

Indentation tests have been widely used for mechanical characterisation of single bulk materials and coated systems. Generally, a small sharp indenter penetrates a material test piece within a certain force or displacement range, by which they are categorized into three types: macro-indentation, micro-indentation and nano-indentation. This small specimen testing method has the advantages of being able to provide information related to local near surface mechanical properties of the test piece. This is especially important for thin films and coated systems since it is critical to separate the mechanical properties of the film and coating from the system. In recent decades, many

experiments, e.g. [11-29], and numerical simulations, e.g. [12-23], have been carried out to obtain displacement and load responses of many different bulk materials and coated systems. A well-known method to determine the hardness and elastic modulus using the indentation testing data is introduced by Oliver and Pharr [11]. Later, similar characterisations have been carried out by other researchers for different materials, such as metallic glass [12-13], bio-materials [14], metals [15, 20, 24, 26-28], coating [16, 21-23, 25, 29] and composites [29]. The load-displacement curves obtained from experiments and simulations are the main data used for the analysis. Other outputs include the surface profile and the stress and strain distributions around the indentation region. The focus of data interpretation has been to extract mechanical properties of the bulk materials or the films and coatings, such as Young's modulus, yield strength, Poisson's ratio and work hardening exponent for power law materials. Many analyses of indentation tests are based on the classic indentation theory developed by Olive and Pharr [11]. Different data interpretation methods for bulk materials [30-43] and thin film or coatings [44-46] have been proposed over the years. Usually, these methods involve dimensional analysis or inverse analysis using FE analysis (FEA).

Determining the mechanical properties of coatings is more challenging compared to single bulk material due to the substrate effect and indentation size effect. A review of the analysis and interpretation of nano-indentation tests discussing the common errors in using the indentation technique has been published by Fischer-Cripps [47]. The focus of that review is pointing out the possible mistakes and sources of error for the practitioners and provide a guidance for necessary correlations. The discussion on extracting material properties is limited to the elastic modulus and hardness. This present work aims to provide a comprehensive review of mechanical characterisation of thin films and coated systems using indentation tests from most aspects, including theoretical studies, experiments, and numerical studies. Regarding the determination of mechanical properties, elastoplastic properties, i.e. Young's modulus, Poisson's ratio, the yield stress and the work-hardening

exponent, are included. The developments and limitations of certain existing methods of determining the mechanical properties of materials using indentation tests are reviewed.

2.2.2. Classic Indentation Theory

To relate the mechanical properties of materials to indentation load-displacement data, there have been several theoretical studies, with the focus on the elastic modulus, E , and hardness, H . The classic theory of Oliver and Pharr [11] is the most commonly used one. Basically, the load (P) is described as a function of displacement (h). A typical P - h curve of an elastoplastic material from an indentation test is shown in Figure 2.1. Figure 2.2 shows the illustration of the indentation geometry at maximum force for an ideal conical indenter.

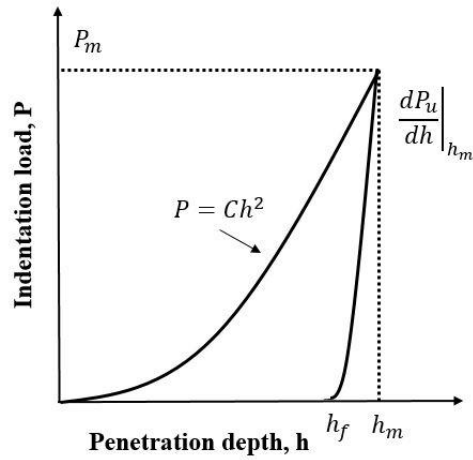


Figure 2.1. Schematic illustration of a typical P - h response of an elastoplastic material to instrumented indentation [11]

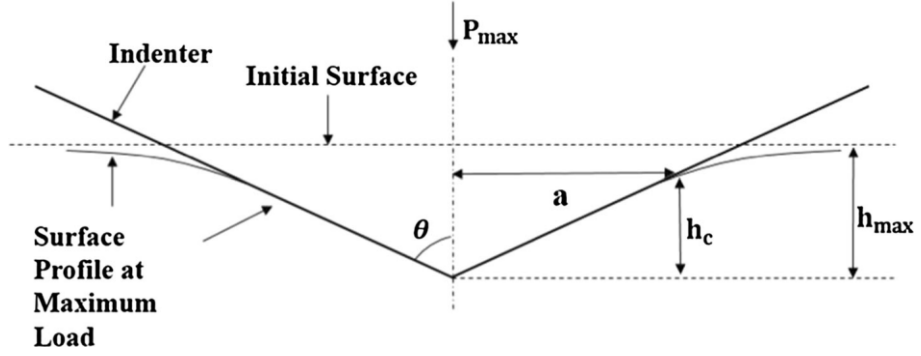


Figure 2.2. Illustration of the indentation geometry at maximum force for an ideal conical indenter.

The P - h curve from the loading process can be described by Kick's Law,

$$P = Ch^2 \quad 2.1$$

where C is the loading curve curvature. Oliver and Pharr's theory relates the measured initial unloading contact stiffness, $S = dP/dh$, to Young's modulus, E . This relationship is given by

$$S = \left(\frac{dP}{dh}\right)_{hm} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}$$

2.2

where $(dP/dh)_{hm}$ is the unloading slope at the maximum displacement h_m , E_r is the reduced modulus and is defined as follows

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E} \quad 2.3$$

where ν , E , ν_i , E_i are Poisson's ratio and Young's modulus of the test material and indenter, respectively. A is the projected contact area at the maximum load, $A = \pi a^2$, where a is the contact radius as shown in Figure 2.2. The approach above has been suggested to be applicable to any indenter that can be described as a body of revolution of a smooth function [48]. For non-axisymmetric polygonal indenters, such as Vickers and Berkovich indenters, a correction factor β can be introduced to Eq. 2.2 [48]:

$$S = \frac{dP}{dh} = \beta \frac{2}{\sqrt{\pi}} E_r \sqrt{A} \quad 2.4$$

where $\beta = 1.034$ for a Berkovich indenter and $\beta = 1.024$ for a Vickers indenter. The hardness of the test material can be easily calculated from:

$$H = \frac{P_{max}}{A} \quad 2.5$$

Therefore, the accuracy of this analysis depends on the accuracy of the estimation of the Poisson's ratio and the measurement of the projected contact area. The measurement of the projected contact area at the maximum load could be a major source of error because it is challenging to measure accurately in practice, since plastic pile-up or elastic sink-in occurs and affects the size of the real contact area. For example, without considering pile-up, the projected contact area is likely to be underestimated, leading to an overestimated elastic modulus and hardness according to Eq. 2.2 and Eq. 2.4. Although the influence of pile-up on measuring the contact area is only significant for materials exhibiting a relatively small amount of elastic recovery after unloading, i.e. $h_f/h_{max} > 0.7$, where h_f is the final indentation depth after unloading and h_{max} is the maximum indentation depth [48]. This analytical approach assumes an elastic-perfectly plastic bulk material. This means that the important work hardening behaviour of most alloys and metals cannot be taken into account. Also, it is not generally applicable to thin films and coatings on substrate materials.

2.2.3. Indentation Tests

Indentation range

For the discussion of indentation tests, it is important to clarify the definition of the indentation range, since it could be ambiguous in some studies. According to the ISO 14577 definition [49], the instrumented indentation tests are divided into three ranges: (i) macro range: $2 \text{ N} \leq P \leq 30 \text{ kN}$; (ii) micro range: $P < 2 \text{ N}$; $h > 0.2 \text{ }\mu\text{m}$; and (iii) nano range: $h \leq 0.2 \text{ }\mu\text{m}$. Different applications have different requirements for the scale of the indentation testing, e.g. small indentation depth is preferred for coating on a substrate to minimise the possible substrate effects while macro indentation could help rule out the size and local defect effects. The scale of the indentation tests is limited by the testing machine. In this review, this definition is adopted to classify the experiments in the literature. Some of the experimental studies conducted in the literature are in the nano range, mostly with bulk materials e.g. [11-13, 15]. Others are in the micro range e.g. [16-23, 44]. Experiments have been conducted with coatings on substrates e.g. [20-23, 44, 45, 50]. Very few indentation experiments have been performed in the nano range with coatings, e.g. [45].

Instrumentation

Generally, most instrumented indentation systems consist of an indenter, a displacement sensor and a frame for holding the sample. This type of system can be illustrated by Figure 2.3 [11]. There are some commercially available systems developed by different companies (such as MTS Systems Corp., Hysitron, Inc., Micro Materials Ltd., CSIRO, and CSM Instruments). Regarding certain features, there are some differences between these systems. For example, some of the devices have a vertical indenter axis (MTS Nano Instruments, Hysitron, CSIRO, and CSM Instruments systems) while it is horizontal in the Micro Materials system. The other major difference is the method of applying force and measuring displacement. In the MTS Nano Instruments, Micro Materials, CSIRO, and CSM Instruments systems, force application and displacement measurement are conducted separately, in different ways. In the Hysitron system, these two processes are performed by the same transducer at the same time [51].

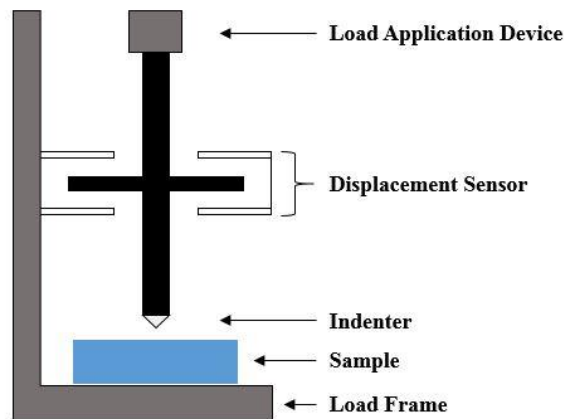


Figure 2.3. Schematic illustration of a typical instrumented indentation system [12].

Indenters

There are three main types of indenters used in experiments: spherical, Berkovich and Vickers indenters. Geometries of the Berkovich and Vickers indenter are shown in Figure 2.4 and Figure 2.5. The effects of using different indenter geometries have been investigated in a few studies using FE analysis and experiments, e.g. [52]. It is pointed out that the ratio of the residual indentation depth after the test to the maximum indentation depth is affected by the indenter geometry. [52]. It is also reported that the Berkovich indenter could reduce the substrate effect [52], therefore it is more suitable for the tests on the coating-substrate specimens. All Berkovich, Vickers and conical indentation tests are reported as not being sensitive to the friction coefficient [52], therefore it is reasonable to neglect the friction for the numerical simulation of the indentation tests. A spherical indenter allows the observation of purely elastic deformation changing to elastic-plastic deformation since the initial part of the loading process is within the elastic regime [53-55]. Also, spherical indenters are easier to control so that the substrate effects can be avoided or reduced compared to the sharp indenters, because they penetrate less with the same loading force. Another type of a less sharp indenter is the Knoop indenter, which is suitable for the material

characterization in narrow areas, e.g. the cross-section of thin films, and studying the direction-dependent properties due to its elongated geometry [56, 57]. It is also useful for indentation of brittle materials since it is less sharp and achieves a 30% lower indentation depth compared to the sharp indenters [52].

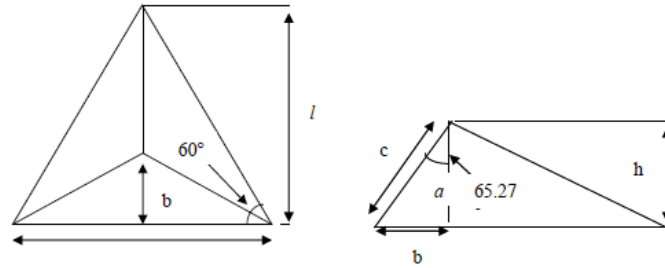


Figure 2.4. Berkovich indenter geometry.

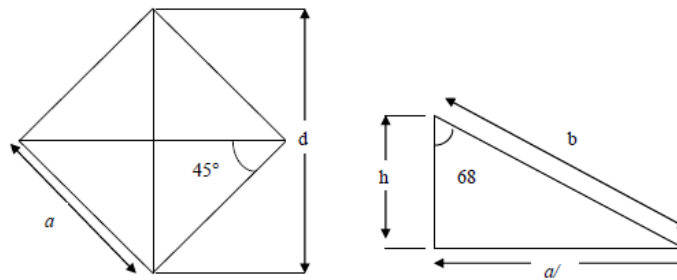


Figure 2.5. Vickers indenter geometry.

For indentation tests performed on a coating system, there are more factors of influence to consider compared to indentation tests on a single bulk material, such as the indentation depth, indentation size effect and surface conditions, such as the porosity and defects. This extra complexity mainly comes from the small coating thickness and the possible substrate effects. Some test method guidelines have been given by ISO 14577 [58] for single coating layers under the conditions where the substrate effects are not significant and where those effects are detected. Surface preparation should be minimised to

reduce the possibility of affecting the indentation responses [58]. Surface roughness is an important factor to consider since the thickness of the coating is usually small, and the allowed indentation depth is even smaller, which means the test results can be greatly affected by a rough surface within a very small penetration depth. The roughness R_a value, which represents the arithmetical average roughness calculated from the surface peaks and valleys from the mean line over the sampling length, should be less than 5% of the maximum indentation depth if possible [58]. To keep the roughness within an acceptable limit, a polishing process might be necessary. However, for metals, the residual stress and the work hardening state can be changed by the mechanical polishing process. Stress induced during polishing should be removed. Different models have been proposed to account for the influence of roughness and defects. The Proportional Specimen Resistance (PSR) model developed by Li and Bradt [59] allows the separation of the roughness effect, but it could not consider the indentation size effect satisfactorily since the relevant parameter is greatly influenced by the presence of roughness [46]. A model proposed by Chicot et al. [60] has been applied to characterise the indentation size effect and to obtain the hardness and the elastic modulus of a porous coating with good agreement achieved with values in the literature [60]. This relationship used for correction is given as follows:

$$P = 26.43 \cdot \left(HM_0^2 + \frac{H_{LSF}^2}{h_i - h_0} \right)^{\frac{1}{2}} \cdot (h_i - h_0)^2 \quad 2.6$$

where HM_0 is the dynamic Martens macro-hardness, H_{LSF} is the hardness-length-scale factor representing the indentation size effect of the material, h_i is the current indentation depth and h_0 is the depth shift. A lower value of hardness is given by this model when the indentation size effect is considered by the hardness-length-scale factor [60]. There are a few experimental studies of indentation on coated system, e.g. [21-23,44-46]. Most of the studies are carried out to determine the elastic properties and hardness of the materials [21-23, 45, 46] while the plastic properties are rarely investigated, e.g [44]. These investigations are mostly aimed at one or two specific coating materials

for a single layer of coating. More combinations of different materials for coatings and substrates and the case of multiple layer coated systems may be investigated in future experimental research.

2.2.4. Determination of Mechanical Properties of Coatings Using Indentation Tests

Dimensional analysis

Dimensional analysis has been used to extract mechanical properties from indentation tests for coating systems e.g. [61, 62]. As presented by Tunvisut et al [63], the loading process of the indentation on a coated system can be described by a functional relationship between the load force P and a series of material properties and parameters as follows

$$P = F_L(E_c, \sigma_{yc}, \nu_c, n_c, E_s, \nu_s, h, h_0, \theta) \quad 2.7$$

where the subscript c and s stand for coating and substrate, respectively and h_0 is the coating thickness.

With the assumption of knowing the substrate and coating thicknesses, the Buckingham π theorem can be applied for normalisation as follows

$$\frac{P}{h_0^2 E_s} = \Pi_\alpha \left(\frac{h}{h_0}, \frac{E_c}{E_s}, \frac{\sigma_{yc}}{E_s}, \nu_c, \nu_s, n_c, \theta \right). \quad 2.8$$

Similarly, the unloading process is expressed as follows

$$P = F_U(E_c, \sigma_{yc}, \nu_c, n_c, E_s, \nu_s, h, h_0, h_m, \theta) \quad 2.9$$

where h_m is the maximum indentation depth. Dimensional analysis of the above relationship gives

$$\frac{P}{h_0^2 E_s} = \Pi_\beta \left(\frac{h}{h_0}, \frac{h_m}{h_0}, \frac{E_c}{E_s}, \frac{\sigma_{yc}}{E_s}, \nu_c, \nu_s, n_c, \theta \right). \quad 2.10$$

Considering the initial unloading slope, the derivative of Π_β with respect to $\frac{h}{h_0}$ can be obtained as follows

$$\left. \frac{1}{h_0 E_s} \frac{dP}{dh} \right|_{h=h_m} = \Pi_\delta \left(\frac{h_m}{h_0}, \frac{E_c}{E_s}, \frac{\sigma_{yc}}{E_s}, \nu_c, \nu_s, n_c, \theta \right). \quad 2.11$$

The functional relationships described by Equ. 2.8 and 2.11 can be quantified using numerical analysis. From FEM analysis, the relationship shown in Equ. 2.11 is calibrated as follows [81]

$$\begin{aligned} \left. \frac{1}{h_0 E_s} \frac{dP}{dh} \right|_{h=h_m} = & \\ \begin{cases} 2.1 \left(\frac{E_c}{E_s} \right)^{0.86}, & \frac{E_c}{E_s} \leq 1, \\ -7 \left(\frac{E_c}{E_s} \right)^{-0.32} - 27.1 \left(\frac{E_c}{E_s} \right)^{0.01} + 13.9 \left(\frac{E_c}{E_s} \right)^{0.002} + 22.3, & \frac{E_c}{E_s} \geq 1, \end{cases} & 2.12 \end{aligned}$$

The general method of extracting the mechanical properties is finding the best match of the indentation curves describing the relationships between the normalised parameters obtained from the experimental tests and the numerical simulations. This approach of data interpretation can easily determine the Young's modulus with an acceptable 10% error for a work-hardening coating material. However, it is suggested [74] that an additional measurement of the final contact area is needed to tackle the issue of uniqueness for the determination of yield strength and work-hardening exponent, because it has been found that almost identical normalised load-

displacement curves can be obtained for materials with different combinations of yield strength and work-hardening exponent. This issue can limit the applicability of this interpreting method since accurately measuring the contact area is challenging in practice. The assumption that the properties of the substrate material are known in advance is sometimes not appropriate in a real situation when the information regarding substrate material may be limited. The other issue is that the indentation is performed to a depth of 33% of the coating thickness in the studies of Tunvisut *et al* [63, 64] while other studies report that the critical ratio of indentation depth to film thickness to separate the coating behaviour from the substrate effects is about 10% [56, 65, 66].

Analytical solutions

Analytical solutions have been proposed to predict the indentation response of coatings and have been used to extract the mechanical properties of the coating materials [67-72]. For example, an analytical solution to determine the Young's modulus of a coating layer, independent of the indentation depth and coating thickness, is given by Chudoba *et al* [68]. Analytical solutions proposed by Schwarzer *et al* [70-72] allow the stresses and deformations under a spherical indenter to be calculated. Good agreements have been achieved by comparisons between the analytical calculations, FE analysis and experiments [67-69]. However, the extraction of material properties is limited to elastic properties, such as Young's modulus, since the plastic properties are not covered by these analytical solutions. Comparison between different analytical models might provide a better insight, but no other analytical solutions for plastic behaviour have been seen in the literature.

FE method

Numerical simulation is another important method to extract material properties for coatings. An iterative FE technique was used by Knapp [61] to determine the Young's modulus and yield stress. Young's modulus and the yield stress are varied while Poisson's ratio and the work-hardening exponent

are fixed. Generally, the iterative procedures are aimed at finding a good fit to the experimental load-displacement curve. This method, however, does not attempt to extract the work-hardening exponent, which is important in many engineering materials. The uniqueness of the material property combination obtained in this study has not been discussed. When more mechanical properties are considered, the issue of uniqueness usually needs further investigation. Also, the computational work is expensive due to the iterative process involved. Tang and Arnell [73] also proposed a method to extract Young's modulus and hardness based on FEA. By introducing mathematical models involving the thickness index, defined as the coating thickness to indenter radius, the stiffness ratio, defined as the ratio of coating to substrate Young's modulus, and the hardness ratio, defined as the ratio of coating to substrate yield stress, the proposed method is able to extract Young's modulus and the hardness of the coating from the load-displacement data without expensive iterative computational work. Yet the hardening behaviour is not considered. The introduced parameter, i.e. the ratio of coating thickness to indenter radius, limits this method to be applied only to indentation tests with a spherical indenter.

Another method based on FE analysis involves a parametric study and measuring the size of the plastic zone [74]. The objective is to find the best fit of both the load-displacement curve and the plastic zone size simultaneously. Because of the need for measuring the plastic zone size, a larger indentation depth is preferred for a better identification and measurement. However, in the case of a thin film on a substrate, increasing the indentation depth raises a challenge in the separation of the indentation response of the coating layer from the substrate. Ma *et al.* proposed a hybrid method based on FE analysis to determine the yield strength and strain hardening exponent. The work-hardening exponent is related to a characteristic parameter, defined based on the load-displacement curve, and then related to the yield stress and the maximum load [75]. The main limitation of this method is the involvement of empirical equations.

2.3. Material Characterisation Using Small Punch Tensile (SPT) Tests and Small Punch Creep (SPC) Tests

2.3.1. Background

The small punch tensile (SPT) testing technique was developed in the 1980s. It has been widely applied for material characterisation when it is required to use a small specimen, e.g. for limited amount of available material or as a non-destructive testing method for critical operating components [76-83]. The earliest reported use of this type of testing technique can be tracked to the miniature disc bent test (MDBT) by Huang et al. [76] and Manahan et al. [79]. In the recent years, the small punch testing techniques have been accepted by more and more researchers, especially in Europe. A code of practice was proposed in 2006 by the European Committee for Standardisation (CEN) [84]. The standard dimensions of the small punch tests were proposed as a clamped disc specimen of 8mm in diameter and 0.5mm in thickness, the receiving hole of 2mm radius and punch diameter between 1-1.25mm. The application of the small punch testing technique has been extended to creep testing [85-94]. One important advantage of the small punch creep (SPC) tests is that they can provide the full-stage creep data, including the initial creep, the steady-state creep and the final stage creep. Since coatings are widely used for high temperature applications, e.g. power plants, chemical plants, aero-engines and gas turbines, it is important to review the studies on the creep behaviour of coated systems. However, it is difficult to obtain the elastoplastic and creep behaviour data through the conventional uniaxial tensile and creep tests due to the small thickness of most coatings. The small punch testing technique has the potential to be a useful material characterisation method for coatings because the specimen is a miniature thin disc. The schematic of a typical small punch testing rig is shown in Figure 2.6.

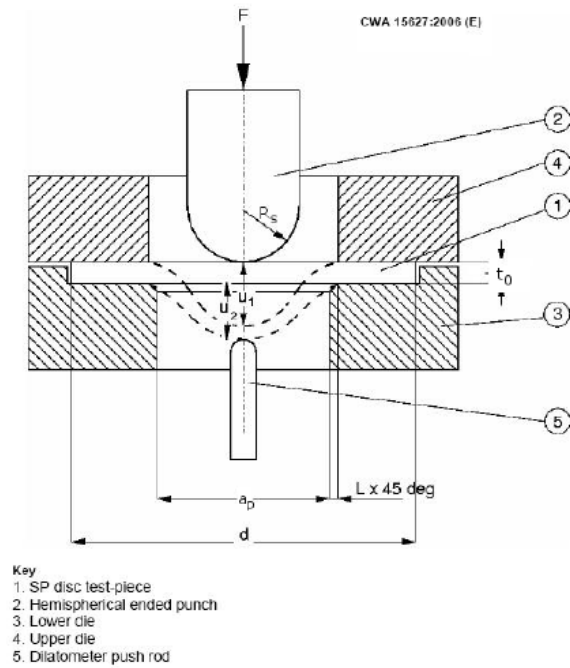


Figure 2.6. Standard geometry of the small punch (SP) test where a SP specimen is clamped between an upper and lower die and the load is applied through a hemispherical punch head [84].

Regarding the typical shape of a load-displacement curve from a small punch tensile test, there are usually 4 distinguishable regions as shown in Figure 2.7 for ductile materials [83, 99, 100]: (I): initial elastic stage reflecting the initial contact between the punch head and the specimen including a limited amount of yielding; (II): the transition stage from the domination of elastic bending to plastic bending with a relatively constant load-displacement rate; (III) the transition stage from plastic bending to membrane stretching starting from a point where the load-displacement rate increases to a higher but still relatively constant value; and (IV) the failure stage, in which the force reaches the maximum value and then decreases as a result of the initiation of localised thinning and crack.

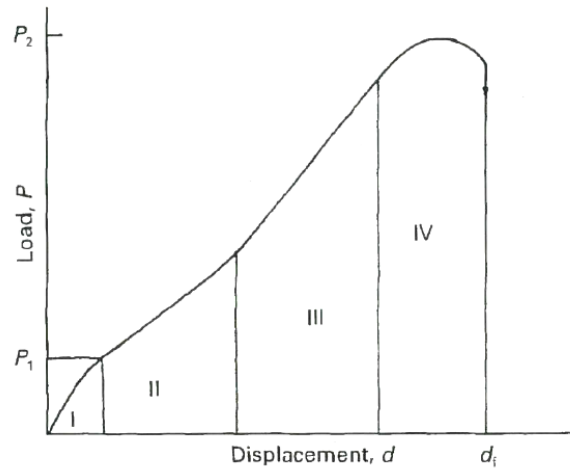


Figure 2.7. Schematic diagram of a typical SPT test load-displacement curve obtained for a ductile material featuring four regions [100].

2.3.2. Small Punch Tensile Test

2.3.2.1. Experimental Data

The main data obtained from the small punch tensile test are the load-displacement curves. For a displacement-controlled test, in which a constant displacement rate is applied to the punch, the load is the reaction force measured at the punch. Comprehensive data of the load-displacement behaviour of various types of steels has been obtained in many studies (e.g. [77, 79]). Such data has also been obtained for thermally sprayed NiAl coatings [95] and thermally sprayed CoNiCrAlY coatings [96-98].

2.3.2.2. Data Interpretation for SPT Test

The SPT test has been used to determine the material properties, such as the elastic modulus, yield strength, and fracture strength (e.g. [80,83, 95-97, 100]).

Elastic-plastic yield load

Regarding the elastoplastic behaviour of a SPT test specimen, the elastic-plastic yield load (F_e) is defined as the load at the transition point from elastic to plastic bending (the end of the stage I described in 2.3.1). The yield strength of a SPT specimen is usually directly related to the yield load. An offset method, in which F_e is defined as the intersect between the load-displacement curve and a line drawn parallel to the elastic region of the curve, being offset by 10% of the specimen thickness (t_0), was proposed by Okada et al. [80]. A smaller amount of offset, 1% of the specimen thickness, was suggested by Cheon and Kim [102] considering that there is usually early stage yielding in the SPT test. Figure 2.8 shows the offset method.

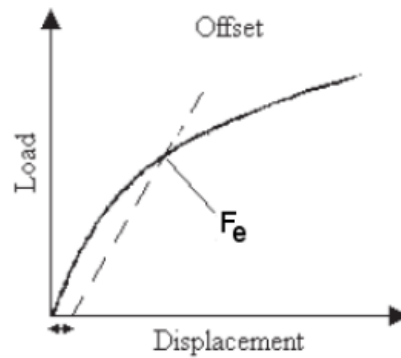


Figure 2.8. The offset method for the determination of the elastoplastic yield load [101]

A two tangents method was proposed by Mao and Takahashi [82]. F_e is defined as the intersect of two tangents drawn parallel to the elastic and plastic regions. A modified two tangents method is included in the CEN workshop agreement [84]. The two tangents method is shown in Figure 2.9.

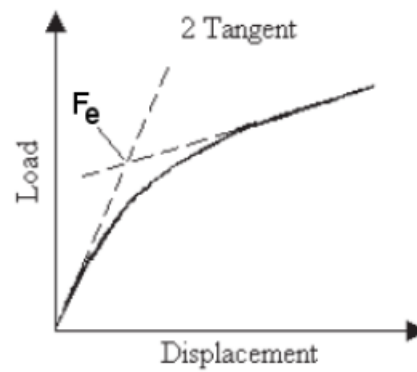


Figure 2.9. The two tangents method for the determination of the elastoplastic yield load [101].

Mak [101] proposed a point of non-linearity method where the yield load is defined as the load where the load-displacement curve transfers from linear to non-linear. It is suggested that the offset and two tangents methods are not suitable for materials with low ductility and/or undefined yield points [101]. The point of non-linearity method is shown in Figure 2.10.

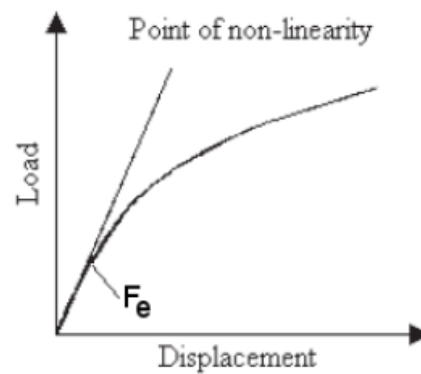


Figure 2.10. The point of non-linearity method for the determination of the elastoplastic yield load [101].

Yield strength and elastic modulus

An analytical method was developed by Roark [103] to calculate the yield strength from F_e . It is based on the bi-axial bending of a thin disc clamped at the circumference. The maximum bi-axial bending stress on the tensile surface of the specimen (σ_{max}) is given as follows:

$$\sigma_{max} = \frac{3F(1+\nu)}{2\pi t_0^2} \left(\ln \frac{a_p}{r'} \right) \quad 2.13$$

where F is the load in N, ν is Poisson's ratio, t_0 is the specimen thickness, a_p is the radius of the supporting jig and r' is the effective radius. The effective radius is calculated from the contact radius (r_c) between the punch head and the specimen:

$$r' = \sqrt{1.6r_c^2 + t_0^2} - 0.675t_0 \quad \text{for } r_c \leq 0.5t_0 \quad 2.14$$

or

$$r' = r_c \quad \text{for } r_c > 0.5t_0 \quad 2.15$$

However, this method is based on the assumption of a constant contact radius and is valid only for elastic bending. The elastic modulus of the specimen can be calculated as follows [103]:

$$E = \left(\frac{F}{\delta} \right) \frac{3a_p^2(1-\nu^2)}{4\pi t_0^3} \quad 2.16$$

where δ is the specimen displacement. Empirical relationships are developed to correlate the F_e/t_0^2 from the SPT test to the yield strength obtained from the uni-axial [82, 83, 104, 105]. However the applicability of these empirical relationships depends greatly on the amount of available uni-axial test data. Therefore, the empirical correlation methods have not been commonly applied to the SPT test on coating specimens, since it is difficult to obtain uni-axial tensile data for coating materials.

2.3.3. Small Punch Creep Test

An elevated operating temperature is unavoidable or preferred for many engineering applications, e.g. power plants, chemical plants, aero-engines and gas turbines. One important material behaviour at high temperature is creep, defined as the permanent time-dependent deformation of materials under a constant load or stress. Creep strains and creep damage may occur and lead to structural failure when the components operate at high temperature for an extended period of time. The primary region exhibits a large initial strain rate associated with elastic loading, which reduces throughout the primary region until a constant strain rate is reached. The region of constant strain rate is defined as the secondary region, which is associated with steady-state creep. Once the tertiary region is reached, the strain rate accelerates, leading to failure. It is crucial to assess the material strengths and estimate the remaining life to failure of the components subjected to creep damage. This information is usually captured by performing standard uniaxial creep tests. However, it requires producing standard test specimens which are not always possible. In some cases, it is desired to characterise the local material behaviours without greatly compromising the structural integrity of the components in service, e.g. coatings, welds and the heat affected zone (HAZ) of a weld. In some other cases, the amount of a material available may be limited, e.g. a new material in the process of development. Therefore, various small specimen testing techniques have been widely employed for material characterisation and component life assessment [106, 107]. Small punch creep (SPC) test is one of the most popular methods (e.g. [85-89]). The main advantage of SPC testing is that the full creep stages, including the primary, the secondary and the tertiary stage, are covered. Figure 2.11 shows the schematic of a typical displacement-time curve from a SPC test.

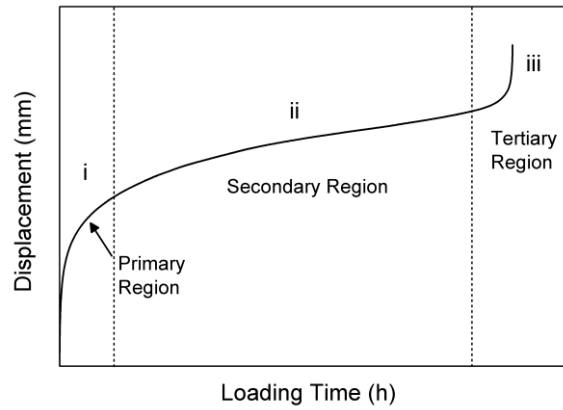


Figure 2.11. Schematic of a typical displacement-time curve obtained from SPC tests [93].

To improve the understanding of SPC test as a material characterisation method, many researchers have carried out experimental (e.g. [85, 86, 88, 89]) and numerical studies (e.g. [88, 108-110]). The most challenging part for the SPC testing technique is to relate the results (i.e. the displacement-time data) to those of a conventional uniaxial creep test (i.e. the strain-time data) and hence obtain the creep constitutive parameters. This is because of the complexity of its deformation and failure mechanisms, i.e. contact mechanism, material nonlinearity, relatively high deformation and localised plasticity effects [110]. This interpretation issue of SPC test has been addressed in many studies (e.g. [93, 94, 111]). A code of practice (CoP) was developed to convert the load and displacement data from SPC test to stress and strain data in 2006 [84]. Although the empirical CoP has been used by researchers and industrial organizations, a universally accepted physically-based interpretation method for SPC test is still missing.

A notable phenomenon which occurs in the early stage of SPC testing is the pre-straining, i.e. the localised plasticity. It should be noted that the effects of pre-straining on the creep behaviours have been neglected in most numerical simulations of SPC testing while they have only been taken into consideration for FE modelling by Cortellino et al [88] for the first time. It is suggested that the pre-existence of plasticity in the material has a significant influence on

the subsequent creep deformation and the time to failure for a SPC testing specimen, showing either creep resistance or creep enhancement and therefore cannot be neglected [88]. It is also shown that the accuracy of the predicted creep behaviour can be improved significantly by taking account of the pre-staining effects.

2.4. Applications of Inverse Methods for Material Characterisation

Inverse methods have been employed in many studies to characterise material behaviours using various testing techniques, such as indentation tests (e.g. [120, 121]), three-point bending (TPB) tests (e.g. [122]), and SPT tests (e.g. [112-117]). The general procedures of the inverse methods are: (i) set the experimental testing results as the target data; (ii) use a numerical or analytical model to generate the predicted testing results; (iii) evaluate and minimise the difference between the experimental and the predicted results; (iv) accept the optimised material properties within the allowed tolerance and (v) validate the acquired material properties by alternative experimental or numerical study (e.g. different testing techniques or FE models). One of the strengths of inverse methods is that they are relatively easy to implement, even for investigators with limited theoretical knowledge of the testing techniques. Also, most factors of influence are taken into considerations by inverse methods since they are entirely based on the experimentally measurable quantities. However, it also means that the validity of inverse methods greatly depends on the accuracy and consistency of the experimental data. In addition, it is convenient to use different material constitutive models and testing techniques with a well-developed inverse approach framework. It should be noted that inverse methods could incur high computation costs, especially when complex FE modelling is involved.

Regarding the difficulty in the interpretation of SPT and SPC tests, inverse approaches have the potential to extract the tensile and creep properties from

SP tests without converting the results to those of a uniaxial creep test. There are a few studies on material characterisation by SPT test using an inverse method, e.g. [112-117], in which the elastoplastic properties of the material are determined from the force-displacement data using a curve-fitting optimisation algorithm. The feasibility of extracting creep properties from SPC testing deformation data using an inverse method with FE analysis has been demonstrated by Li et al [118]. It has been proved that the inverse method with FE analysis delivers better predictions for the material properties than the commonly used method based on Chakrabarty's membrane theory for SPC test [119]. However, Li's model has only considered the simplified case of steady-state creep but not the full-stage creep damage evolution.

2.5. Conclusions of the Literature Review

It is revealed by the literature review that a good understanding of the mechanical behaviour of the coatings is crucial for the evaluation of the lifetime and performance of the serving coating systems and the development of new coating systems and their applications.

Compared to the comprehensive study on the characterisation of the conventional metal materials, the research work on the mechanical behaviour (e.g. elastic-plastic behaviour and creep behaviour) of coatings is relatively limited. The main reason is that many conventional mechanical testing techniques, e.g. the uniaxial tensile and creep tests, are not applicable to the coatings due to their relatively small thickness. Indentation and small punch tensile/creep tests have the potential to be the tool of mechanical characterisation of coatings because their applicable testing scales are close to the scales of coatings. Therefore, it is worth investigating their applicability for coatings further.

There have been some experimental and numerical studies on the mechanical characterisation of coatings. However, a universally accepted characterisation

method, being capable of acquiring the elastic-plastic and creep properties of the material from the testing of a miniaturised-size specimen, is still needed for coatings. Although there are studies aiming to extract the material properties from mechanical tests using miniature specimens geometrically similar to the conventional testing specimens (e.g. [123-126]). Generally, the agreements reported between the miniature and standard specimen tests are promising, though differences are observed despite that the sizes of some specimens used in the literature are relatively large.

The inverse method has been a widely used method for mechanical characterisation due to the good compatibility for different testing methods and material constitutive models. The application of the inverse method for material characterisation of coatings is limited by the insufficient experimental work.

Chapter 3 - Methodology

In this chapter, the research methods used during this study are presented. The numerical methods, including FE modelling and nonlinear optimisation, are introduced. The experimental techniques, including the indentation test, small punch tensile/creep test and a novel miniature specimen, are also described. Scanning electron microscopy (SEM) was used to study the fracture surfaces and material phases of the specimens. A general description is given for the inverse approach developed to determine the material properties of a single material and the material of interest in a two-material system.

3.1. Numerical Methods

3.1.1. Finite Element Modelling

The commercial finite element code ABAQUS 6.14-1 was used for the numerical simulations in this study. A series of FE models were built, including indentation tests, tensile tests, small punch tensile and creep tests. To build a FE model for a scenario of engineering applications using ABAQUS, the user is required to give some key definitions to generate a valid input file. Basically, developing a FE model is a process of translating the necessary information of the scenario into the code language. The FE models were built using the ABAQUS CAE user interface. The following are the main steps:

Parts

The first step is to define the geometries of the parts involved. For instance, the geometries of the indenter and the specimen are required for a FE model of an indentation test. Drawings of the parts with defined dimensions need to be produced. It should be noted that ABAQUS does not have a built-in unit system. Therefore, it is important to ensure the consistency of the unit used

in a model, e.g. mm, N and MPa were used for length (displacement and distance), force and stress, respectively.

Materials

The second step is to define the material behaviour. Various constitutive models can be applied to the model, such as elastic-plastic, creep and damage models etc.

Sections

The defined material properties need to be assigned to the corresponding sections of the parts, e.g. different material properties need to be assigned to a coating-substrate part. The boundaries of different sections need to be defined.

Assembly

All the parts created need to be assembled. The initial relative positions of the parts need to be defined in this step, e.g. the positions of the clamps in a small punch tests model.

Interaction

The interactions between the parts need to be defined. For example, the contact conditions between the surfaces of the clamps, the specimen and the punch in a small punch test model.

Steps

It needs to be specified how many stages are involved in the FE model. For example, an indentation test can be usually divided into three stages: loading, holding at the maximum load and unloading.

Boundary conditions and loadings

The boundary conditions and the load applied in each step of the model need to be defined separately. For a load-controlled model, load is defined as force, stress or moment. However, for a displacement-controlled model the loading process is usually defined as a variation of the displacement boundary conditions, e.g. the displacement of the clamps in a tensile test model.

Mesh

Finally, mesh geometries and element types need to be defined for the analysis. This step is important for the accuracy, efficiency and convergence of the FE model, e.g. a refined mesh should be used for some areas of a part to ensure good accuracy and convergence where high stress is expected while a coarse mesh can be used for the less critical areas to reduce computing time. A mesh sensitivity study is necessary for a complex time-consuming model to find the optimum point between the solution accuracy and computing time. Suitable element types need to be selected, e.g. a 2D plane-stress element could be used for a very thin specimen.

3.1.2. Inverse Method

An inverse method was developed and applied associated with different experimental techniques and it was adjusted to comply with different applications. The general framework can be described as follows: (i) supply the experimental data to the optimisation function; (ii) generate and execute the input file for the FE modelling or carry out analytical calculations and obtain the corresponding predicted results; (iii) pass the predicted results to the optimisation function to process; (iv) record the optimisation process until it is finished. The details are given in the later sections for the applications for different testing methods. This inverse method algorithm was constructed using the commercial code MATLAB. The optimisation work involved by the inverse method was carried out using the optimisation toolbox provided by this code. For the optimisation functions, a main script is required to

specify the control options, e.g. initial values, step size, limits and function tolerance, etc. An objective function is needed to specify the quantified optimisation values, which are decided by the available experimental data and the type of the optimisation function.

For the application of the inverse method in conjunction with FE software, ABAQUS in this study, a Python script is needed to read the output file of ABAQUS and to generate the files storing the required output data. For the applications involving a user-subroutine, e.g. FE modelling with creep behaviour, a Fortran script is needed to provide the definitions and options for the subroutine.

3.2. Experimental Methods

3.2.1. Small Punch Tensile Tests

Displacement controlled SPT tests were carried out on a Tinius Olsen H5KS single column materials testing machine. SPT tests were carried out at a displacement rate of 1 $\mu\text{m/s}$ at RT and between 500-700°C. The displacement of the specimen was recorded as the average of two LVDT's. The coating material used in the miniature tensile tests was produced by high velocity oxygen fuel (HVOF) thermal spraying using a commercially available powder known as Praxair CO-210-24 with the following nominal composition: Co-31.7Ni-20.8Cr-8.1Al-0.5Y (wt.%). The powder had a size range of $-45+20\ \mu\text{m}$ with a chemically analysed oxygen content of 0.037 wt.% O. The coating was deposited onto mild steel substrates of dimensions $60 \times 25 \times 1.8\ \text{mm}$ using a Met Jet III liquid fuel HVOF gun. A schematic of the rig is shown in Figure 3.1 with a punch head radius of 1.0 mm and a receiving hole diameter of 4.00 mm in accordance with the CEN workshop agreement [84]. The load is applied via a 2.5 kN load cell and transmitted to the sample through a hemispherical punch head (a) of 1 mm radius. The punch head is aligned through an alignment bush (not shown) which is removed after alignment is complete. The specimen is clamped by screwing the top nut (b)

onto the lower die (c), which presses a clamping disc (d) onto the specimen (e). Two pins (not shown) prevent the clamping disc from rotating and causing friction on the surface of the sample. The displacement was measured by two LVDT's which connect to the punch head at (f). The samples were heated to temperature prior to the application of the load and held at this temperature for 3 hours before the start of the test. The temperature was monitored by a thermocouple located at (g).

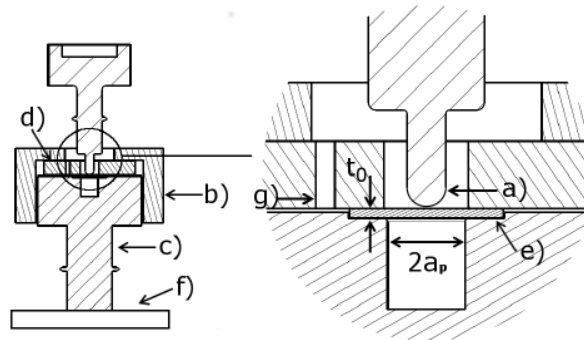


Figure 3.1. Schematic of custom built Tinius Olsen H5KS single column materials testing machine [127].

3.2.2. A Novel Miniature Specimen Tensile and Creep Test

The miniature specimen tensile and creep tests were performed using the same Tinius Olsen H5KS single column materials testing machine (described in 3.2.1) as the SPT tests. Special loading assembly parts (shown in Figure 3.2) were designed and manufactured for this novel miniature specimen testing method. More details of the miniature specimen testing technique are given in Chapter 5.

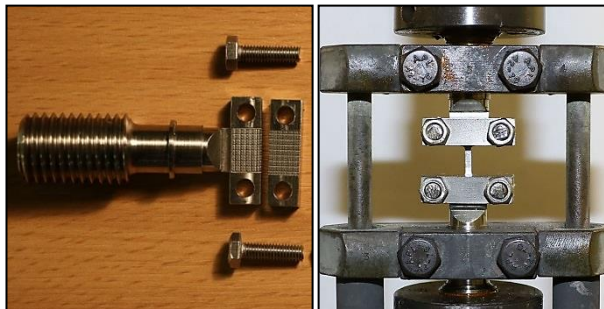


Figure 3.2. The loading assembly parts and the mounted miniature specimen.

3.2.3. Indentation Test

Indentation tests were performed using a Micro Materials Ltd. (MML) Nanotest NTX platform 2. Figure 3.3 shows the schematic of this indentation instrument.

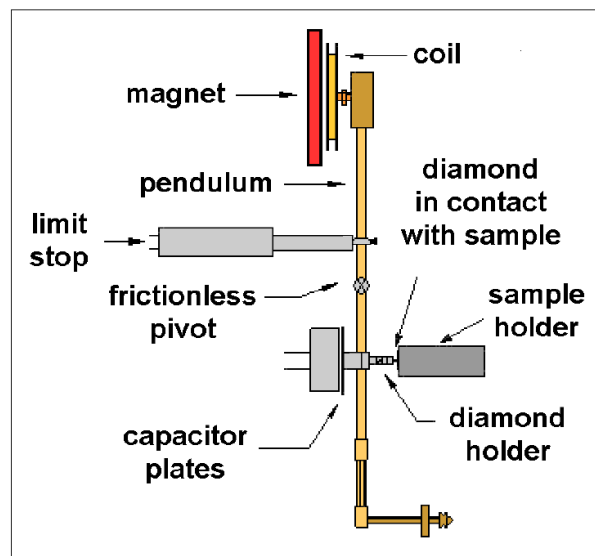


Figure 3.3. Schematic of Nanotest NTX platform 2 [128].

The indentation tests can be performed either as load-controlled (a constant load rate and a maximum load are applied) or displacement-controlled (a constant displacement rate and a maximum displacement are applied). For the NanoTest™, the pendulum assembly shown in Figure 3.3 is used to apply the load. Electromagnetic force actuation is used for this machine, an electromagnet is placed at the top of the pendulum next to a permanent magnet. The supply voltage to the electromagnet is increased during loading and the electromagnet is pulled towards to the permanent magnet. The assembly pivots around a frictionless hinge and the indenter is moved into the sample surface. The load is controlled by the applied voltage. The indenter displacement is recorded by monitoring the change in capacitance between

the capacitor plates. This is done by recording the output from a Wheatstone bridge circuit, which is sensitive to the change in the distance between the plates [128].

3.3. Microstructural Characterisation

Scanning electron microscopy (SEM) was used to study the fracture surface of the failed samples from the SPT and miniature specimen tensile tests. A tungsten filament FEI XL30 scanning electron microscope was used to carry out SEM. Secondary electrons (SE) were used to image the fracture surfaces and backscattered electrons (BSE) were used to form images of cross sections of mounted specimens [129]. A high energy electron beam is used by the electron microscopy to examine objects on a fine scale. Different signals such as characteristic X-rays, secondary electrons (SE) and back scattered electrons (BSE) are created when the primary electron beam interacts with matter in a thick sample, as shown in Figure 3.4 [130]. The scattered electrons are collected by detectors in the scanning electron microscope and the signals produced are used to form images. The contrast in SE images is due to sample topography and so are typically used to study surfaces. In BS images, the contrast is proportional to the mean atomic number of the phase, which allows compositional differences in the sample to be detected [131]. A phase with low mean atomic number will appear darker than a phase with high mean atomic number [129].

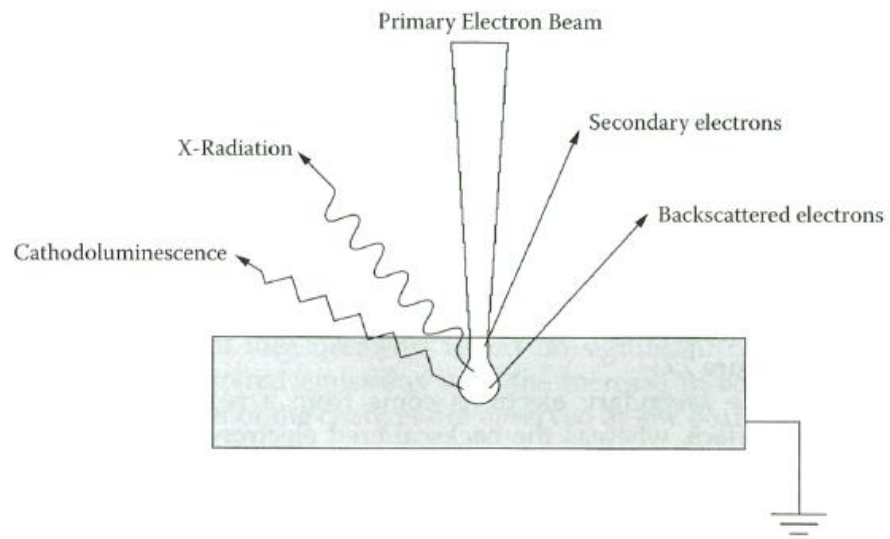


Figure 3.4. Schematic representation of the signals emitted during scanning electron microscopy [129].

Chapter 4 - Development and Applications of an Inverse Method

This chapter presents the development of an inverse method for determining the mechanical properties from the experimental results and the preliminary and more advanced applications of this inverse method. Some notable factors of influences are investigated.

4.1. Inverse Method Procedures

An inverse method framework was developed on the basis of nonlinear optimisation function provided by the commercial code MATLAB. Figure 4.1 shows the general procedures of the inverse method of determining the material properties from the experimental results: 1) an initial estimation x_0 is given for the material properties; 2) the current material properties x_i are supplied to a FE model or analytical model to obtain the predicted testing results y_i under the given conditions; 3) the experimental results y_{exp} are taken as the ‘target’ data; 4) the predicted and experimental testing results are subjected to nonlinear optimisation using MATLAB functions; 5) if the differences between the predicted and the experimental results are minimised within the acceptable tolerance, the current material properties are accepted as the optimised material properties x_{opt} , otherwise the optimisation continues to the next iteration.

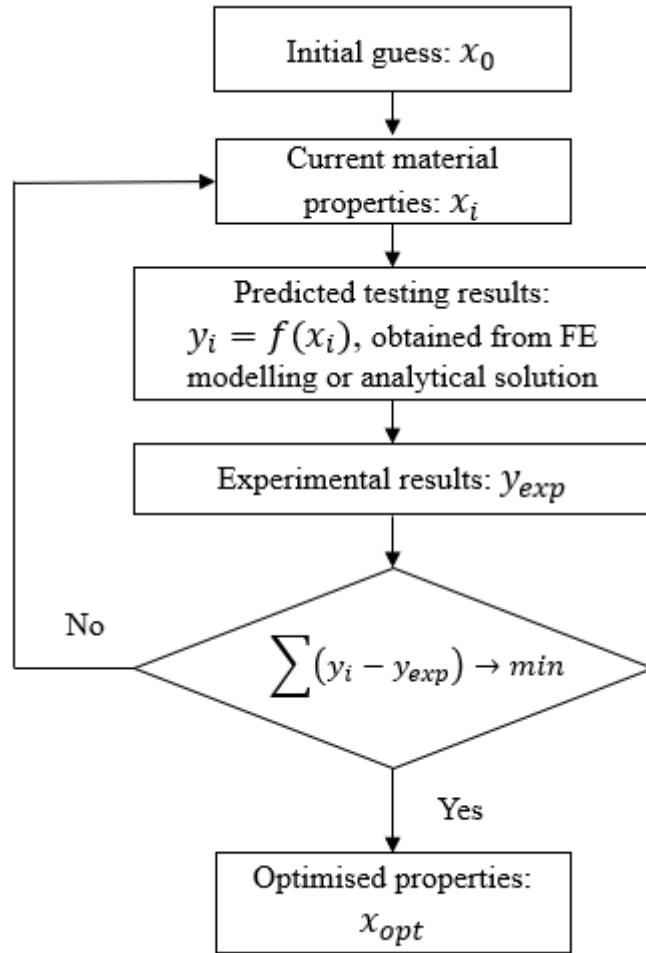


Figure 4.1. The general inverse method procedures.

4.2. Factors of Influence

There are a few important factors of influence regarding the FE modelling and the inverse method. One of the factors is the choice between the load-controlled condition and the displacement-controlled condition. The other factor is the scaling factors required by the nonlinear optimisation function. These factors are investigated here taking the case of indentation testing as an example.

Figure 4.2 shows the geometry and mesh design of a typical FE model of spherical indentation testing. The FE model is developed using the

commercial code ABAQUS 6.14. Axisymmetric model is used for simplicity and the analytical rigid element is used for the indenter, meaning that the deformation of the indenter is neglected. The mesh of the indented material near the indenter is refined (three-node elements with reduced integration, CAX3R, were used) due to the expected high stress and strain rate in that area. Coarse mesh (four-node elements with reduced integration, CAX4R, were used) is used for the area away from the centre to save the computing cost. Mesh sensitivity study was carried out to ensure the accuracy and efficiency of the FE model.

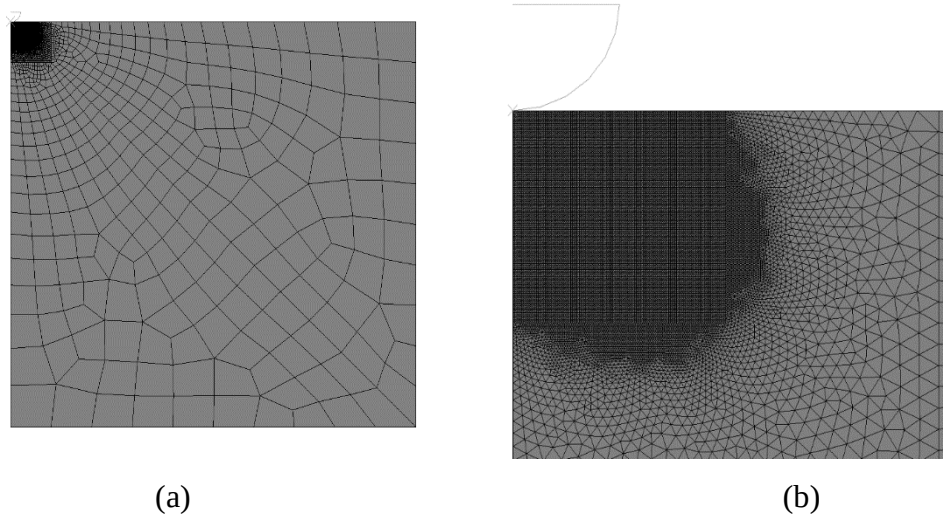


Figure 4.2. The spherical indenter FE model: (a) the overall view (b) the refined mesh near the indenter tip.

4.2.1. FE Modelling: Load-Controlled and Displacement-Controlled Models

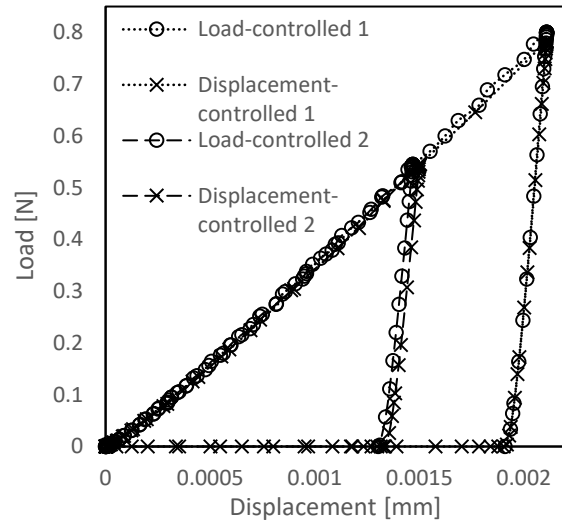
FE simulations are carried out to compare the two cases of load-controlled and displacement-controlled models. Generally, the same load levels, defined by the maximum force or indentation depth, are applied to simulate the indentation tests under the same conditions. Table 4.1 shows the applied loading cases. In case 1, 800mN is applied in the load-controlled model and the maximum indentation depth at the maximum load is found to be 2.1 μ m. Then the maximum depth is applied to the displacement-controlled model. In

case 2, the maximum indentation depth $1.5\mu\text{m}$ is first applied in the displacement-controlled model and the maximum force obtained, 544.62mN is then applied to the load-controlled model.

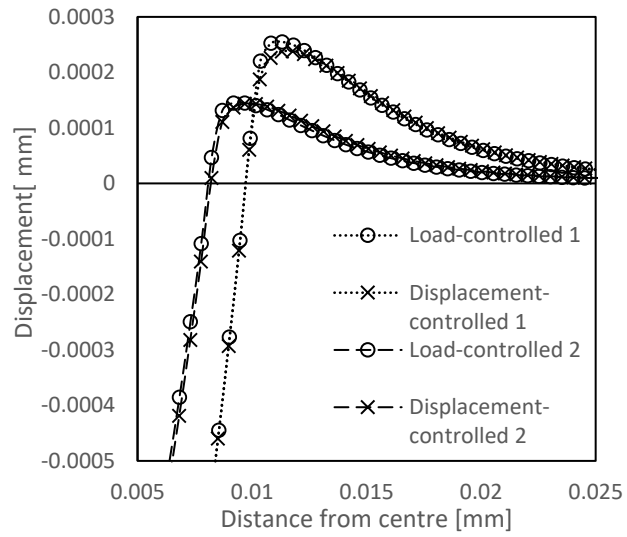
Both the load-displacement curves and the surface profiles from the two types of models are extracted and plotted in Figure 4.3. It can be found that there are differences between the results from the two different types of models. Regarding the load-displacement curves, as Figure 4.3 (a) shows, the load-controlled model predicts a smaller indentation depth than the displacement-controlled model during the unloading stage in case 1. As shown in Figure 4.3 (b), there are also differences between the surface profiles predicted by the two different models. Generally, the load-controlled model predicts a higher material pile-up than the displacement-controlled model. These results indicate that ABAQUS gives different predictions for the load-controlled and displacement-controlled models even for the same load conditions. This is due to the presence of very high plastic strains in the indentation zone which require a highly non-linear numerical iterations and load increments in the FE software. FE analysis of elastic-plastic behaviour is dependent on the load path.

Table 4.1. The applied loading cases.

Case	Maximum load (mN)	Maximum depth (μm)
1	800	2.1
2	544.62	1.5



(a)



(b)

Figure 4.3. Load-displacement curves for spherical indenters (a) and the surface profiles (b) obtained from displacement-controlled and load-controlled finite element models.

4.2.2. Inverse Method: Load-Controlled and Displacement-Controlled Models

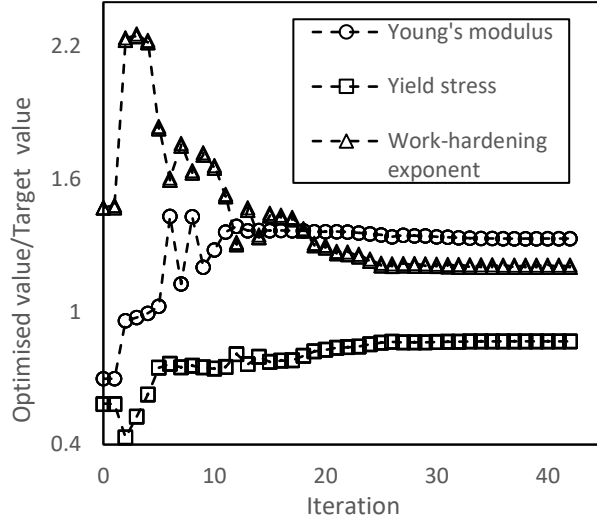
An inverse optimisation algorithm incorporating the load-displacement curve and the indented surface profile was applied to a load-controlled and a displacement-controlled indentation test FE model. Some typical results are presented in Figure 4.4, comparing the optimisation processes from the load-controlled model to the displacement-controlled model. The initial estimation used in the optimisations are shown in Table 4.2. The target material properties for P91 steel are: Young's modulus=215GPa, the yield stress=515MPa and the work-hardening exponent=0.136. Poisson's ratio is kept as a constant of 0.3. Generally, the predictions from the load-controlled model show poorer quality than the displacement-controlled model, e.g. in Figure 4.4 (a), the errors in the optimised predictions obtained from the load-controlled model are from 14% to 33% for the three material properties while they are within 1% for the displacement-controlled model as shown in Figure 4.2 (b). The FE model details and the initial guess with an average difference of 39% from the target values, are identical in these two cases.

The differences between the results from the two types of FE models are also shown by the load-displacement curves and the surface profiles in Figures 4.5 and 4.6. Generally, the load-displacement curve and the surface profile predicted by the displacement-controlled model in the final iteration agree well with the target data, as shown in Figure 4.6. However, there are clear deviations from the target curves in the case of load-controlled model, as shown in Figure 4.5.

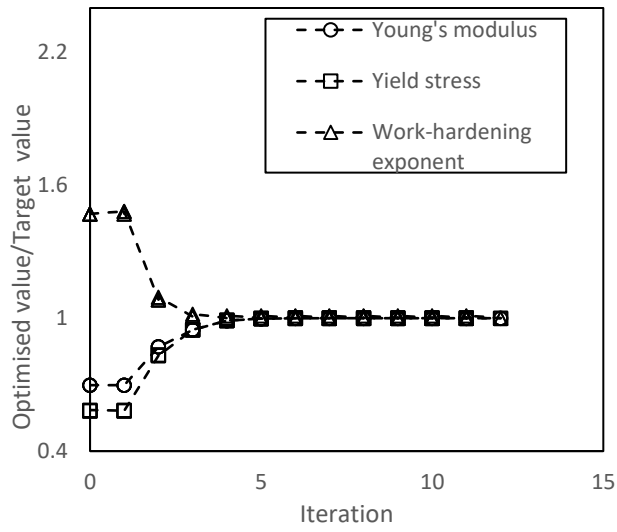
Table 4.2. The initial estimations used for the optimisations.

	Load-controlled model			Displacement-controlled model		
Initial guess	E (GPa]	σ_y (MPa]	n	E (GPa]	σ_y (MPa]	n
1	150	300	0.2	150	300	0.2

2	240	650	0.1	300	700	0.08
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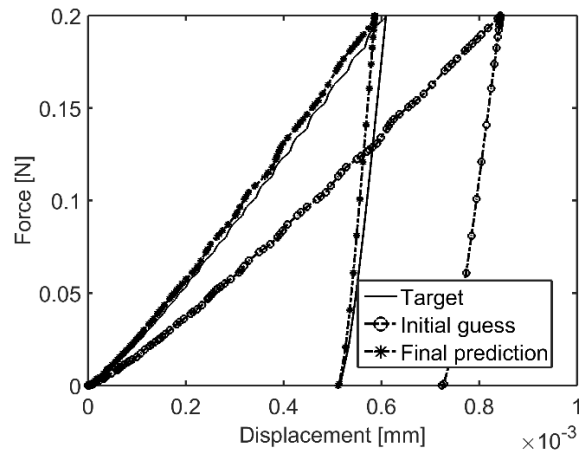


(a)

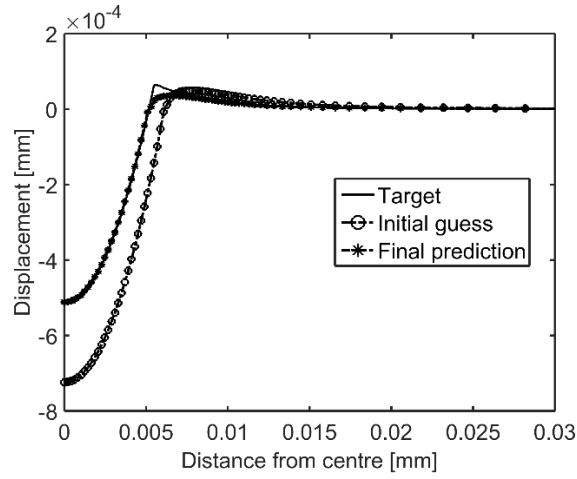


(b)

Figure 4.4. Evolutions of the predicted material properties for spherical indenters: displacement-controlled model, (a) load-controlled model, 200mN (b) displacement-controlled model, 2 μ m.

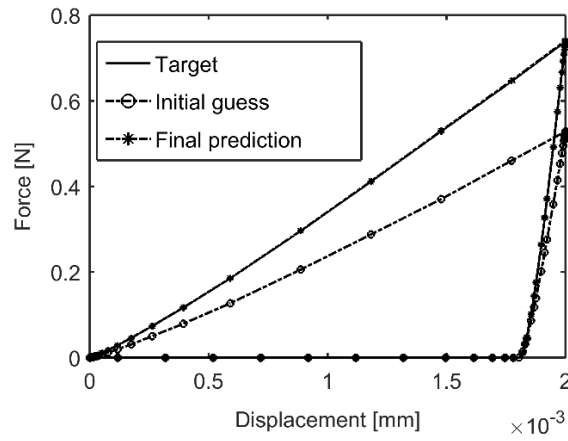


(a)

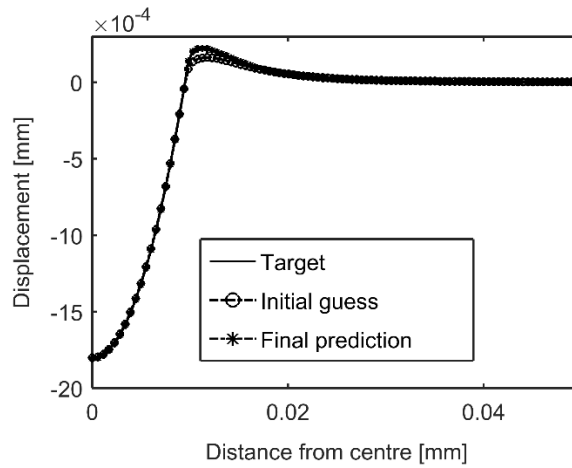


(b)

Figure 4.5. Comparison between the target data, the initial guess and the final prediction for the load-controlled model: (a) load-displacement curve and (b) the surface profile.



(a)

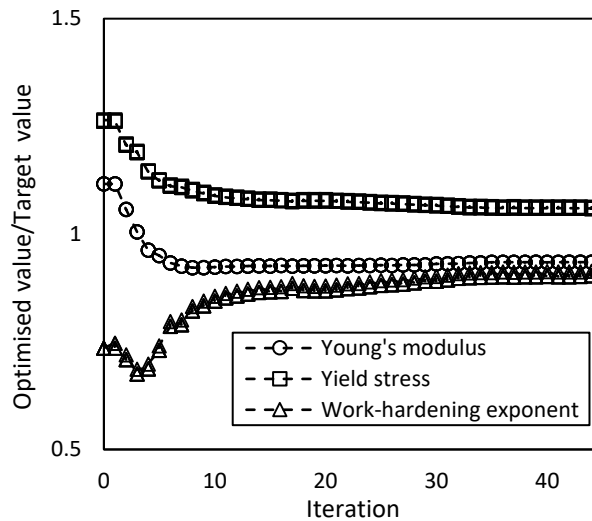


(b)

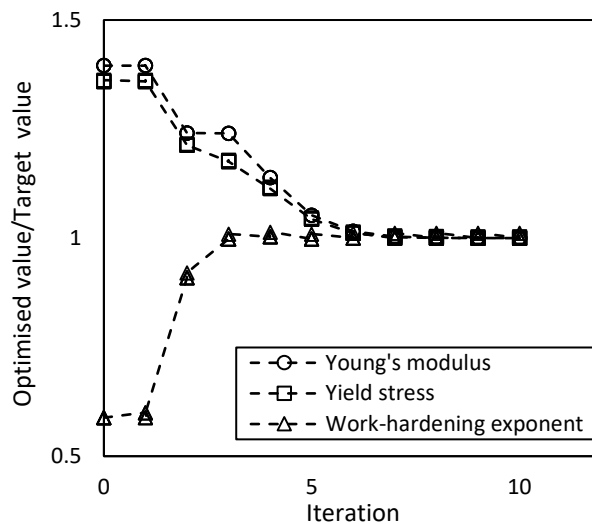
Figure 4.6. Comparison between the target data, the initial guess and the final prediction for the displacement-controlled model: (a) load-displacement curve and (b) the surface profile.

Different initial estimations with different maximum forces and maximum displacements are used to verify this significant difference between the two types of FE models. As shown in Figure 4.7(a), the initial guess is made closer to the target values for the load-controlled model, with an average difference of 21% from the target values. However, the errors from the load-controlled

model are still up to about 10%, while the displacement-controlled model shows a consistent level of high accuracy and faster convergence with the initial guess relatively further from the target values, with the error of 39%, as Figure 4.7(b) shows.



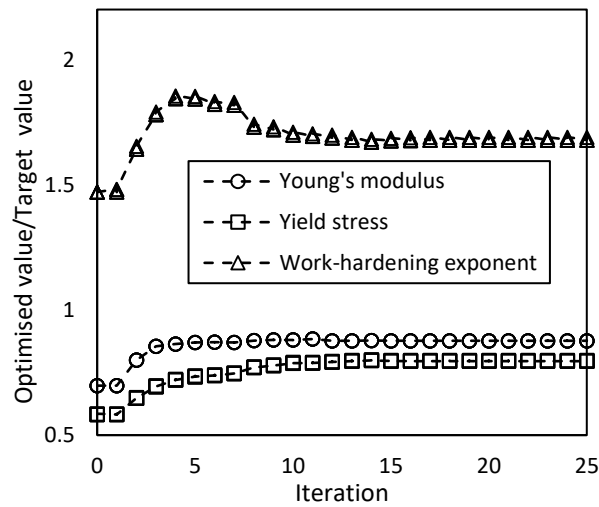
(a)



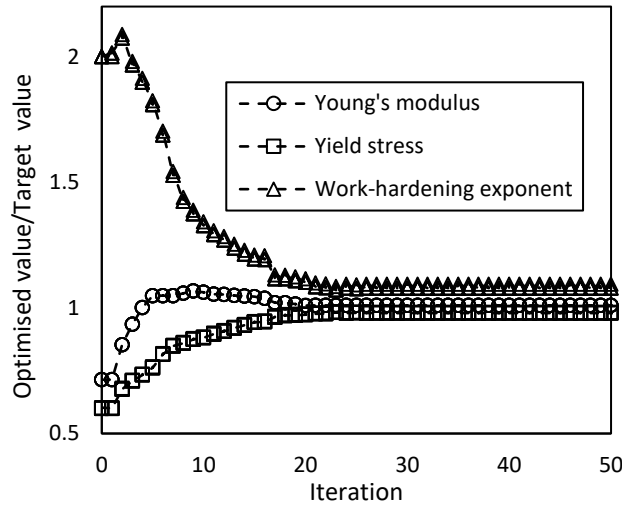
(b)

Figure 4.7. Evolutions of the predicted material properties for spherical indenters: (a) load-controlled model, 500mN (b) displacement-controlled model, 2 μ m.

To investigate the possibility of the indenter geometry effects, a load-controlled conical indenter model is used for the optimisation. As Figure 4.8 (a) shows, the load-controlled indenter conical model gives a poor quality prediction while the displacement-controlled conical indenter model shows a high accuracy in the predictions as Figure 4.8 (b) shows. These differences agree well with the results from the spherical models and it suggests that the indenter geometry is not a major factor of influence.



(a)



(b)

Figure 4.8. Evolutions of the predicted material properties for conical indenter: (a) load-controlled model, 800mN (b) displacement-controlled model, 50 μ m.

4.2.3. Effects of the Scaling Factors on the Inverse Method

As discussed above, the quality of the predictions made with a load-controlled FE model is not satisfactory. The average error percentage can be over 10%, generally. One possible reason is that the scaling factors of the two measured parameters, i.e. the load-displacement curve and the surface profile, used for the optimisation are not appropriate. They might require further investigation to be decided carefully. The necessity of introducing the scaling coefficients is due to the factor that the least square nonlinear optimisation function (*lsqnonlin*) used in MATLAB can only take one error vector from the objective function. Therefore, the errors in the two measured parameters have to be constructed in the same error vector to be passed to the optimisation process. Equ. 4.1-3 show the objective errors of the optimisation.

$$e_1 = c_1 \cdot (h1 - h0) \quad 4.1$$

$$e_2 = c_2 \cdot (U1 - U0) \quad 4.2$$

$$e = |e_1| + |e_2| \quad 4.3$$

The scaling factors c_1 and c_2 decide the ratio of the corresponding error e_1 and e_2 to the total objective error of the optimisation, e . h and U are the indentation depth and surface displacement, respectively.

Load-Controlled Model

Table 4.3 shows the tested scaling factors for a load-controlled spherical indentation FE model. The results of these tests are shown in Figure 4.9. All the optimisations start from the same initial guess of the material properties: Young's modulus=300000 MPa, yield stress=700 MPa and work-hardening exponent=0.08. The target material properties are: Young's modulus=215000 MPa, yield stress=515 MPa and work-hardening exponent=0.136. It can be found that the accuracy of the optimisations varies in a wide range with the use of different scaling factors. Test 9, with the scaling factors $c_1=0.4$ and $c_2=1$, gives the best results with an average error of 2.8% (5.43% for the Young's modulus, 0.05% for the yield stress and 2.92% for the work-hardening exponent). In this case, the errors in the predicted load-displacement curve are reduced by 60% artificially for the optimisation while the real values of the errors in the predicted surface profile are taken. Test 1 has the scaling coefficient $c_1=1$ and $c_2=0$, meaning that only the error in the prediction of the load-displacement curve is considered. The average error percentage of the optimisation is 33.35%. This indicates that considering only the load-displacement curve is not sufficient for this optimisation algorithm to provide accurate prediction on the material properties. Test 12 has both the coefficients of 1, meaning the two measured parameters are treated equally and the real values of the errors are used. The error percentage of the optimisation is 9.79% in this case. This suggests that treating the two measured parameters equally and taking the original values do not lead to the

best results for this optimisation, although the errors in the load-displacement curve and the surface profile are of the same order of magnitude. These results suggest that the scaling factors of the two parameters can be seen as another parameter in the optimisation and need to be investigated independently for every FE model used for this optimisation algorithm.

Table 4.3. The tested scaling factors for the load-controlled indentation FE model.

Test	1	2	3	4	5	6	7	8	9	10	11	12
c1	1	0.75	0.5	0.3	0.25	0.2	0.3	0.35	0.4	0.45	0.5	1
c2	0	0.25	0.5	0.7	0.75	0.8	1	1	1	1	1	1

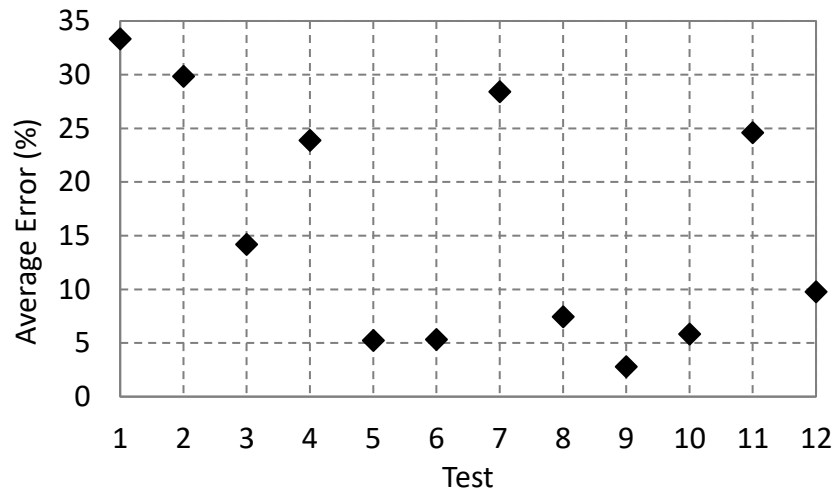


Figure 4.9. Effects of the scaling coefficients on the optimisation for a load-controlled indentation FE model.

Displacement-Controlled Model

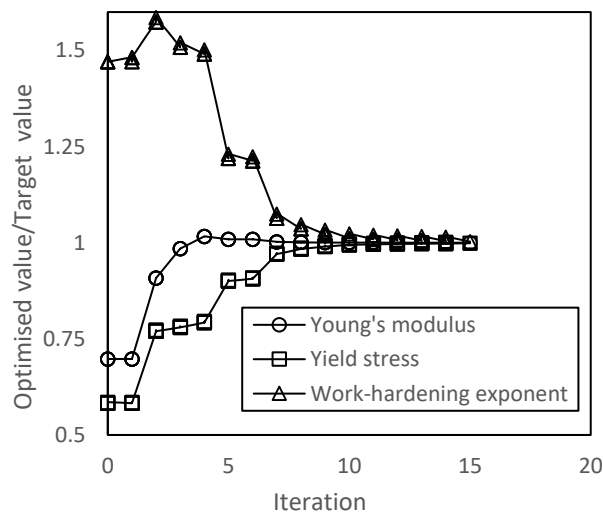
It has been shown previously that the inverse method is able to give accurate predictions for the mechanical properties with the displacement-controlled FE model. However, since the results obtained from the load-controlled model indicate that the scaling factors of the objective function parameters are able to make significant differences in the optimisation, it is worth investigating the sensitivity of the displacement-controlled model to the scaling factors as well. Similar to the previous section, a series of scaling factors are tested for the optimisation algorithm working with a displacement-controlled model.

Table 4.4 shows a series of scaling factors used for the displacement-controlled model and the corresponding average error percentage. Because the deviation in load-displacement and the surface profiles are of different orders, the scaling factors are selected to change the orders of the two parameters. The reason for this is that if the two parameters included in the objective function for the optimisation algorithm are of very different orders, then the total magnitude of the objective function is likely to be dominated by the higher order parameter. Although, it has been proved by the work done with the load-controlled model that scaling the two parameters to the same order does not necessarily lead to accurate predictions. As shown in Table 4, the average error percentages are generally within 0.5% for all three pairs of scaling factors, indicating excellent accuracy achieved by the inverse optimisation algorithm. This result differs from the load-controlled model, which shows very different levels of accuracy for different scaling factors as shown in Figure 4.10. It is worth noting that the optimisation gives very accurate prediction even in test 1 where the surface profile influence? is scaled to zero. The evolutions of the predicted material properties from these three tests are shown in Figure 4.10. Figure 4.10 (a) and (b) suggest that tests 1 and 2 are very similar, although the scaling factor c_2 is 0 and 1, respectively. The reason is that the deviation in surface profile for the displacement-controlled model is of a much lower order of magnitude than in the P-h curve. In this case, the difference is 10^4 , generally. Therefore, with the same scaling factor of 1, the objective function is dominated by the deviation in P-h curve

and the surface profile contributes very little to it. One possible explanation is that in this case it is easy to predict the material properties using a displacement-controlled model, even with little contribution from the information of the surface profile, so that the scaling factors are much less important. When the scaling factor for the surface profile is increased to 10^4 in test 3, the convergence of the optimisation is achieved more promptly as shown in Figure 4.10(c). This suggests that the extra information of surface profile still improves the efficiency of the optimisation algorithm, even in the case when the material properties can be predicted without taking it into consideration.

Table 4.4. The tested scaling factors for the displacement-controlled model.

Test	1	2	3
c1	1	1	1
c2	0	1	10000
Avg error %	0.146947	0.4567	0.000393



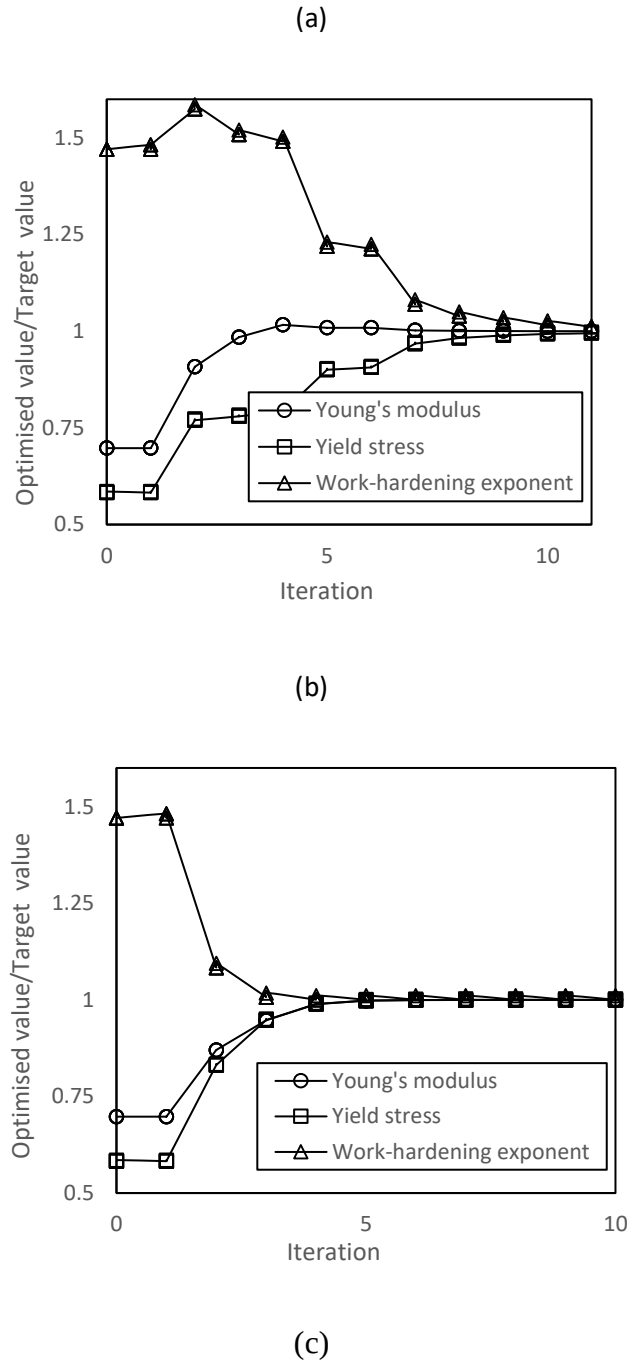


Figure 4.10. The evolutions of the predicted material properties for spherical indenters: displacement-controlled spherical indenter model, (a) test 1 (b) test 2 (c) test 3.

More different initial estimations are used to investigate the effects of the scaling factors on the optimisation with the displacement-controlled FE

model in the cases when the material properties are more difficult to predict. Table 4.5 shows the variation in the accuracy of the optimisation with different scaling factors for a different initial guess: Young's modulus=300000MPa, yield stress=700MPa and work-hardening exponent=0.08. The average error percentage varies from about 20% to below 0.0001% when the scaling factor of the surface profile increased from 0 to 10^4 . The variation is shown by the different evolution histories in Figures 4.11-4.12. It can be found that the optimisation process converges faster and more accurately when the scaling factor for the surface profile is increased, comparing Figure 4.12 (a)-(d). The best results are given by test 4, as shown in Figure 4.12 (d), when the two parameters are scaled to the same order of magnitude? generally. Tests 5 and 6 consider the situation when the errors in the surface profile are scaled to a higher order than the load-displacement curve, shown in Figure 4.12 (e) and (f), respectively. In test 5, the errors in the surface profile are generally scaled to be about 10 times in magnitude as the errors in the load-displacement curve. It can be found in Figure 4.12 (e) that a very accurate convergence to the target can be achieved, but it takes a much larger number of iterations to converge, i.e. about 180 iterations compared to fewer than 10 iterations in test 4. The significantly reduced efficiency of the optimisation may be due to fact that the contribution to the objective function from the load-displacement curve is limited, compared to the surface profile. In test 6, the errors in the surface profile are scaled to be about 100 times in magnitude as the errors in the load-displacement curve. It can be found in Figure 4.12 (f) that the optimisation is not able to converge close to the target, with an average error of about 26%. In this case, the objective function is overwhelmingly dominated by the surface profile, thus unable to make effective use of the information from the load-displacement curve. It is worth noticing that the predicted work-hardening exponent in test 6 is very accurate, with the error within 0.1%. This may be explained by the fact that the pile-up of the material, which is included in the surface profile, mainly depends on the work-hardening exponent. The effects of the scaling factors on the predictions of the optimisations are also shown by the load-

displacement curves and the surface profiles predicted by FEA, as shown in Figure 4.12 and 4.12 for tests 1 and 4, respectively. It can be found that in both tests the final predicted load-displacement curve agrees well with the target curve, as shown in Figure 13(a) and Figure 4.14 (a). However, when the scaling factor of the surface profile is 0, the predicted surface profile does not agree well with the target, as shown in Figure 4.13 (b). The predicted surface profile is almost identical to the target when the scaling factor of the surface profile is increased to 10^4 , as shown in Figure 4.14 (b). The results of this investigation indicate that using only the load-displacement curve could lead to difficulty in converging to the target material properties. The scaling factors are critical to the effectiveness of the inverse method and the optimum scaling factors should take account of both parameters to ensure effective contribution from them to the objective function.

Table 4.5. The tested scaling factors for the displacement-controlled model: an alternative initial guess.

Test	1	2	3	4	5	6
c1	1	1	1	1	1	1
c2	0	1	100	10000	100000	1000000
Avg error %	19.20	18.94	0.000541	4.09E-05	0.17	25.64

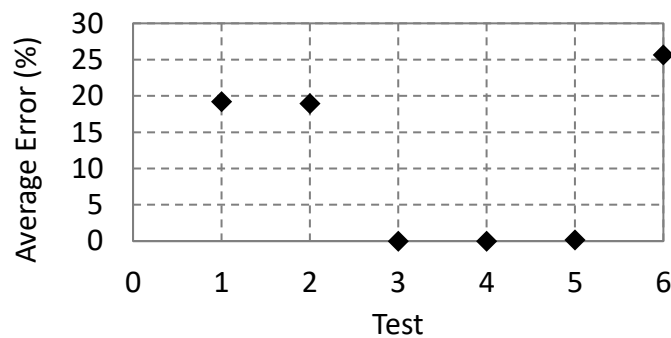
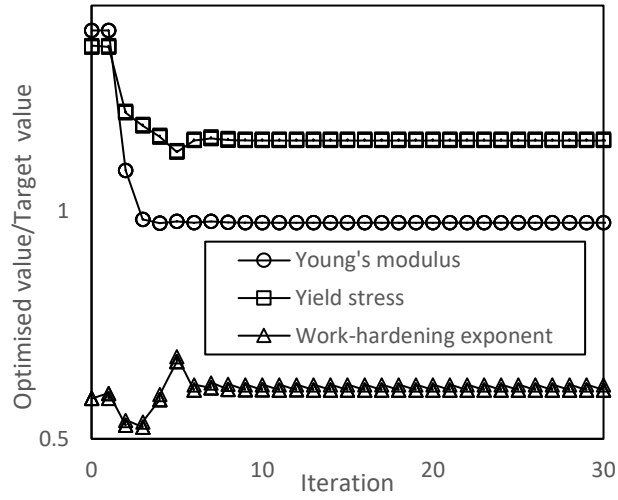
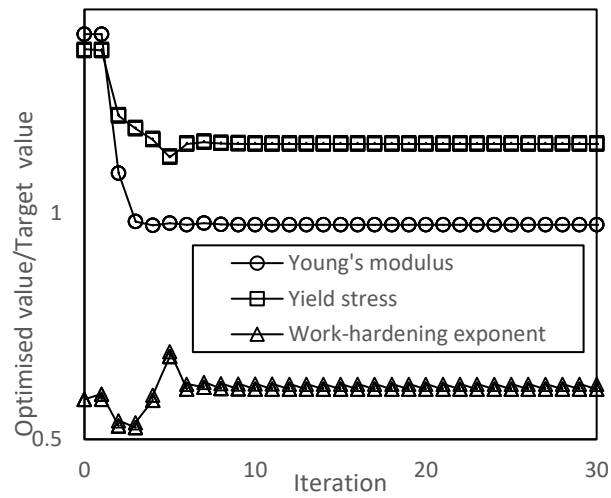


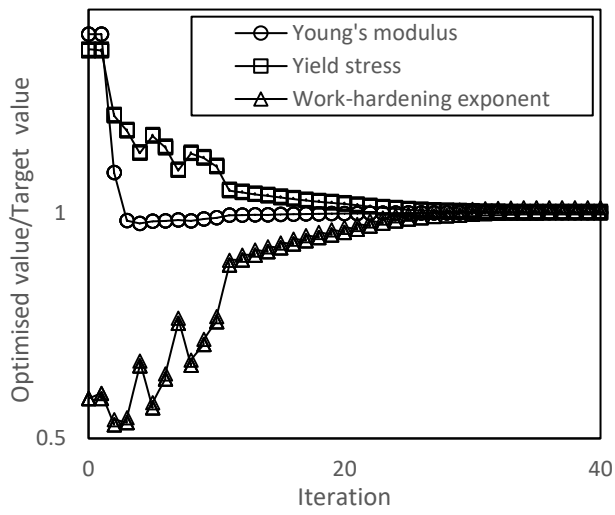
Figure 4.11. Effects of the scaling coefficients on the optimisation for a displacement-controlled indentation FE model.



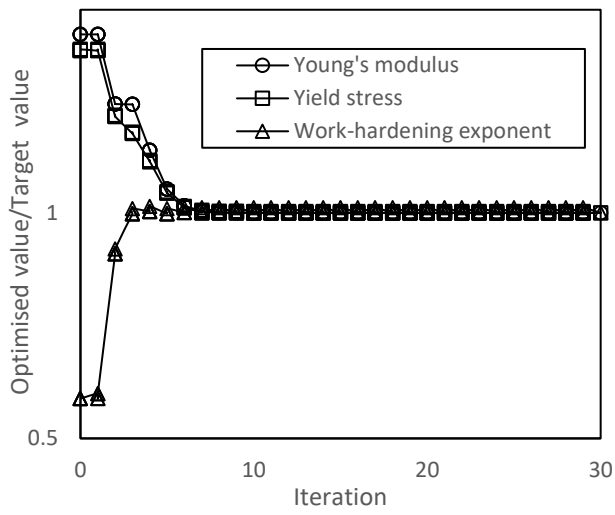
(a)



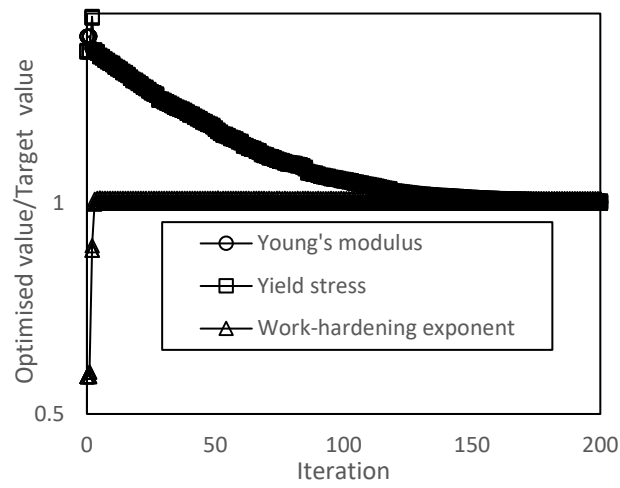
(b)



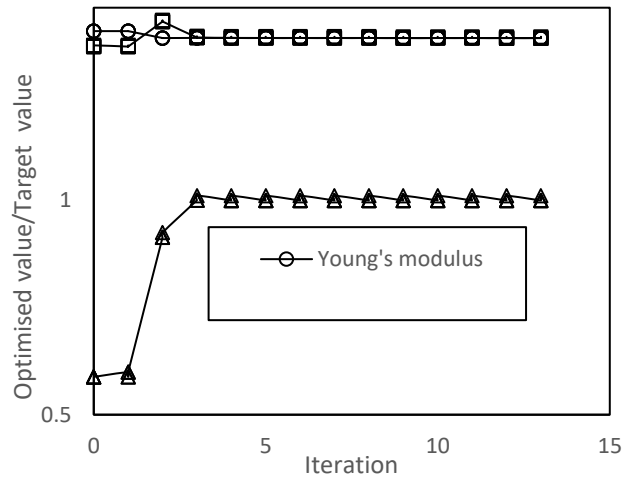
(c)



(d)

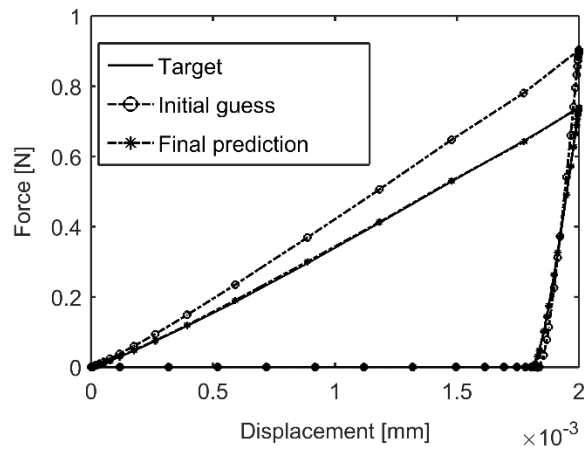


(e)

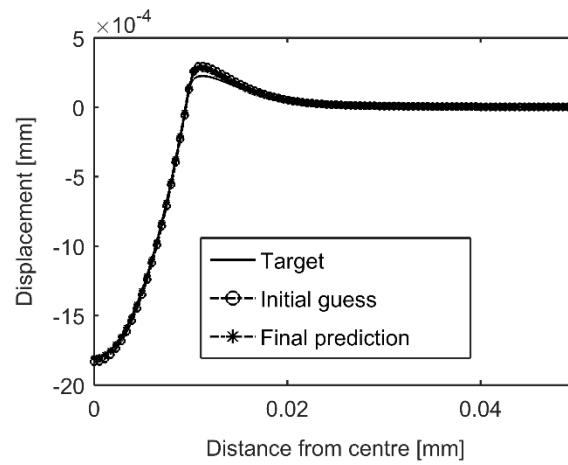


(f)

Figure 4.12. Evolutions of the predicted material properties for spherical indenters: displacement-controlled model, (a) test 1 (b) test 2 (c) test 3 (d) test 4 (e) test 5 (f) test 6.

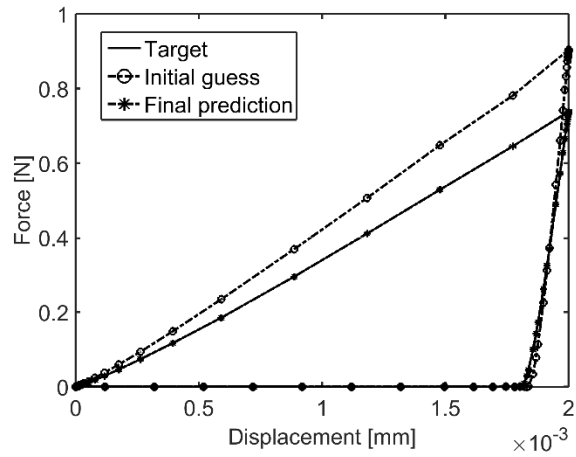


(a)

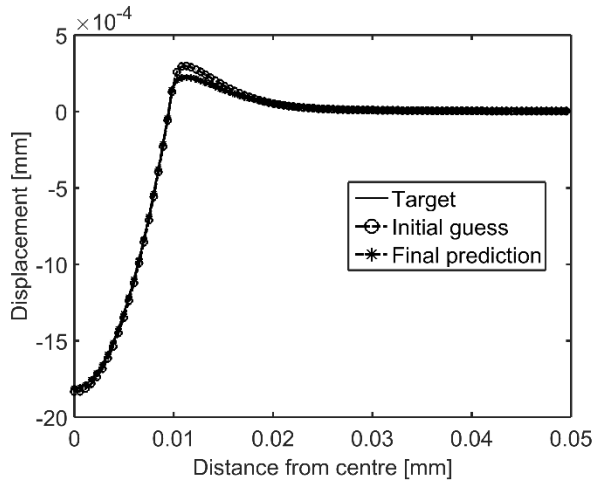


(b)

Figure 4.13. Comparison between the target data, the initial guess and the final prediction for the scaling factors investigation (spherical indentation): test 1.



(a)



(b)

Figure 4.14. Comparison between the target data, the initial guess and the final prediction for the scaling factors investigation test 4 (spherical indentation): (a) load-displacement curves (b) surface profiles.

4.2.4. Conclusions

The following conclusions can be arrived at based on the influence of the load and displacement controls on the optimisation method:

- 1) The load-controlled and displacement-controlled FE models produce different results when the indentation tests are simulated for the same loading level using the commercial FE code ABAQUS.
- 2) It is more difficult to determine the material properties accurately using the optimisation algorithm incorporating the indentation load-displacement curves and the surface profiles for the load-controlled FE model than the displacement-controlled one.
- 3) The scaling factors of the two optimisation parameters have significant effects on the accuracy and efficiency of the optimisation algorithm. A preliminary parametric study is necessary before applying the inverse method to an experimental method. The inverse method is more sensitive to the scaling coefficients when a load-controlled model is used than the case of a displacement-controlled model.

4.3. Preliminary Applications of the Inverse Method to Indentation Tests

4.3.1. Determination of the Mechanical Properties from the Numerical Indentation Curves and the Surface Profile Using an Inverse Method

The inverse method was applied to determine the elastic-plastic properties of materials with the numerical indentation curves as the target data obtained from FE modelling. The objective function for the multi-objective optimisation is shown as follows:

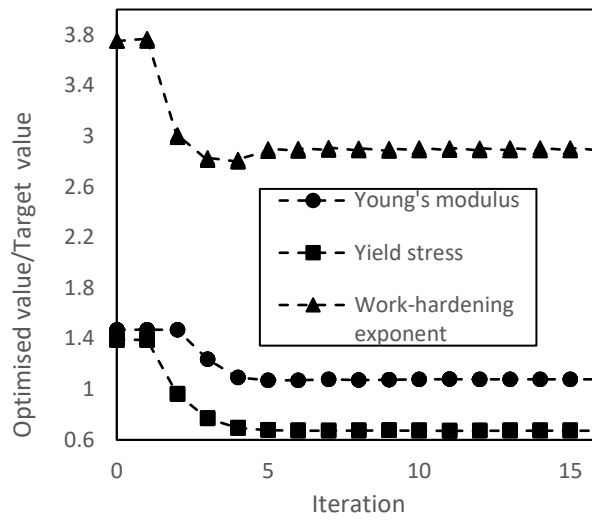
$$\text{error} = [P(h)_{FEA} - P(h)_{Exp}] + [U_2(x)_{FEA} - U_2(x)_{Exp}]$$

where P is the loading force, h is the displacement of the indenter, x is the distance from centre and U_2 is the displacement measured for the surface profile of the indented specimen. The error in U_2 is enlarged 10000 times. The target material properties and the initial estimations for Al 2024 T251

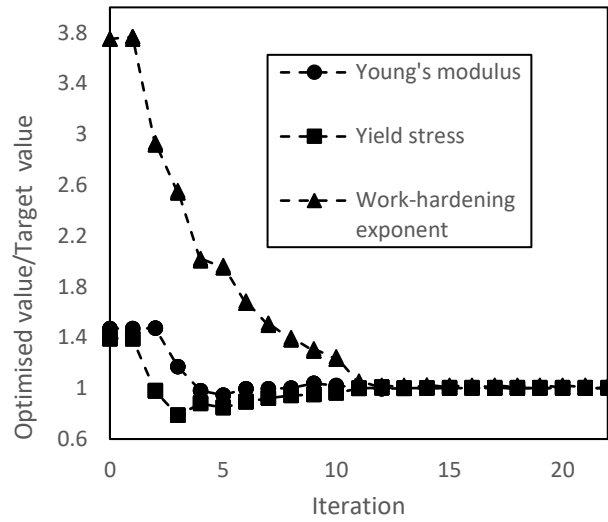
and a reference steel are shown in Table 4.6. The optimisation evolutions for the inverse methods with and without the surface profile as a part of the objective function are shown in Figure 4.15. It can be found that the optimisation converges much more accurately and faster with both the load-displacement curves and the surface profile as the objective function. The error percentages in the optimised properties are shown in Table 4.7. Figure 4.16 and 4.17 shows the optimised load-displacement curves and the optimised surface profiles compared to the target data and the initially predicted results. It can be found that both sets of curves are optimised to be very close to the target data, though the initial predictions are quite far away from the targets.

Table 4.6. Target parameters and the initial estimations (x_0) for Al 2024 T251 and a reference steel.

	E (GPa)	ν	σ_y (MPa)	n
Al 2024 T251	68	0.3	360	0.08
x_0	100	0.3	500	0.3
Steel	210	0.27	500	0.1
x_0	150	0.27	300	0.2



(a)



(b)

Figure. 4.15. Evolutions of the FEA predictions for Al 2024 T251: (a) optimisation without the surface profile (b) optimisation with the surface profile .

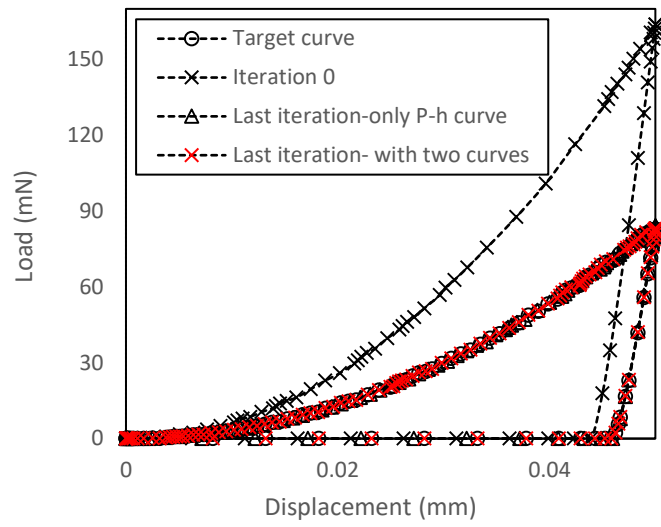
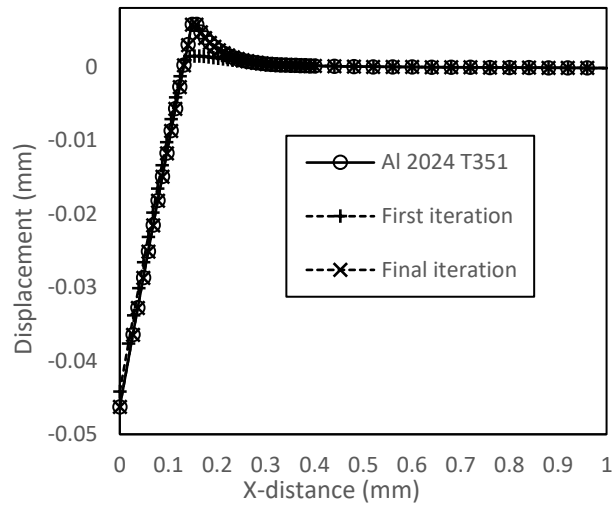
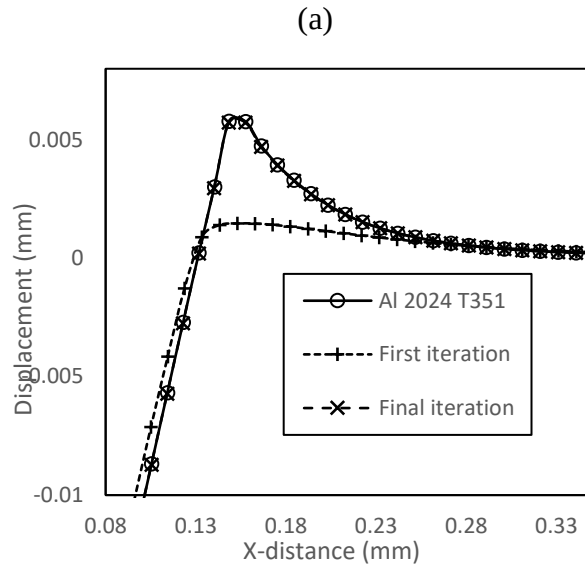


Figure. 4.16. Predictions for P-h curves: Al 2024 T251.





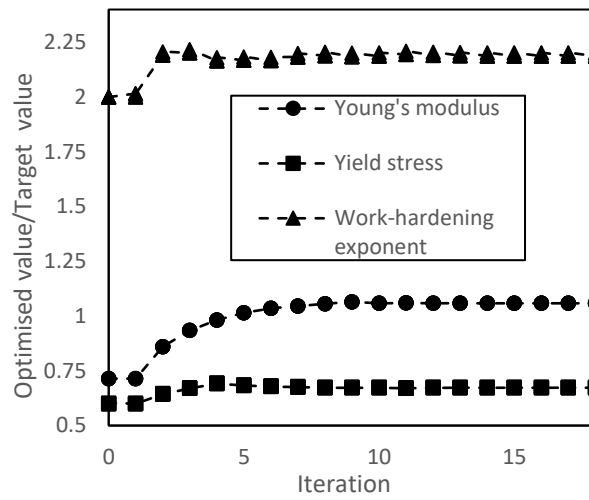
(b)

Figure. 4.17. Predictions of the surface profile for Al 2024 T251: (a) the complete profile (b) the zoom-in view.

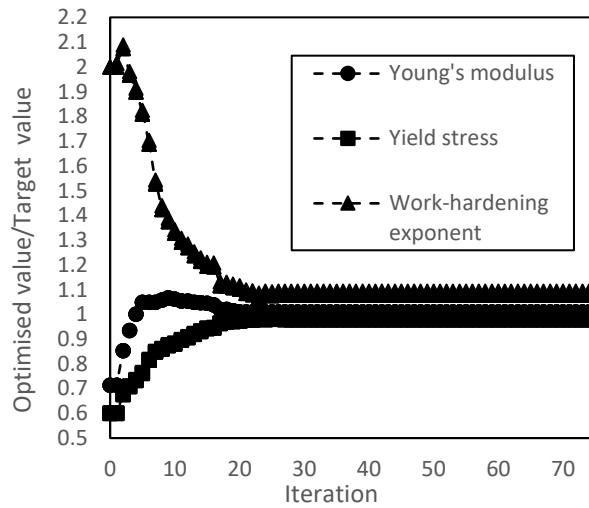
Table 4.7. Errors from the application of the inverse method for Al 2024 T251.

	E (%)	σ_y (%)	n (%)
Only P-h curve	7.83	-32.74	190.54
P-h curves and surface profile	0.14	0.13	2.32

Figure 4.18 shows the evolutions of the inverse analyses carried out for the reference steel. Similarly, the inverse method was less effective using only the load-displacement curves as the target data (average error over 50%) while it became much more accurate with the surface profile included as the additional target data (average error within 5%).



(a)



(b)

Figure 4.18. Evolutions of the optimisations for steel: (a) optimisation without the target area (b) optimisation with the target area.

4.3.2. A Study on the Plastic Zone Boundary as a Potential Target Quantity for the Inverse Method

The plastic zone boundary, defined as the outmost boundary of the area with positive plastic strain, was investigated as a potential additional target

quantity to be used for the inverse method. The investigations were carried out taking the indentation testing as an example.

Indenter Geometry

Conical indenter and spherical indenter were used for the same FE model to simulate the indentations with the identical material properties and indentation depth. Figure 4.19 shows the boundaries of the plastic zone with positive plastic strain value obtained from FE modelling for the conical and spherical indenters. It can be found that the geometry and size of the plastic zone are not insensitive to the indenter geometries.

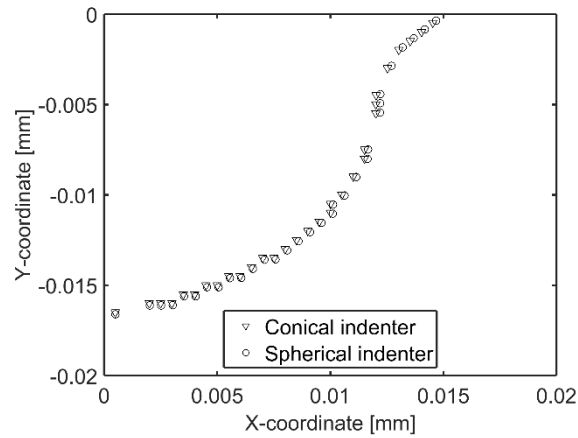


Figure 4.19. The plastic zone boundary from FEA with conical and spherical indenters.

The objective function used for the inverse method optimisation combining the load-displacement curve and the plastic zone is:

$$\begin{aligned} \text{error} = & [P(h)_{FEA} - P(h)_{Target}] + [X_{FEA} - X_{Target}] + [Y_{FEA} - Y_{Target}] \\ & + [\text{plastic strain}_{FEA} - \text{plastic strain}_{Target}] \end{aligned}$$

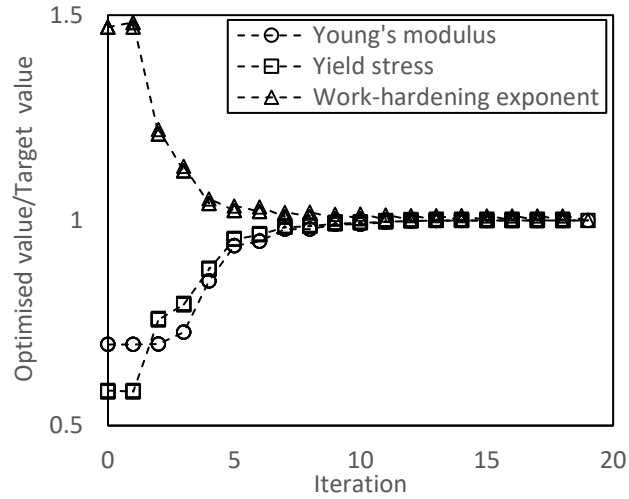
where X and Y are the distance of the nodes on the boundary of plastic zone.

Table 4.8 shows the target material properties and the initial estimations. The target material properties were applied for a conical indentation FE model to obtain the target plastic zone boundary. The evolutions of the inverse method optimisations for the mechanical properties are shown in Figure 4.20. As shown in Figure 20 (a) and (s), the inverse method was able to determine the mechanical properties accurately (within 1% of error) for both initial estimations.

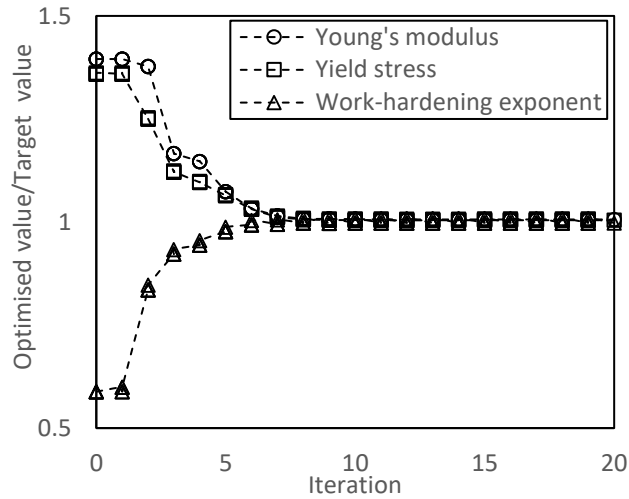
It is indicated by this numerical study that the plastic zone boundary is a potential target quantity for determining the mechanical properties using the inverse method, though experimental studies are needed for verification. However, unlike reading the FE modelling results, it is difficult to experimentally identify and measure the boundary of the plastic zone from a tested specimen.

Table 4.8. Material properties for the optimisations.

	E [MPa]	ν	Y [MPa]	n
P91 (target)	210000	0.3	515	0.136
Initial guess 1	150000	0.3	300	0.2
Initial guess 2	300000	0.3	700	0.08



(a)



(b)

Figure 4.20. The evolutions of the inverse method with a displacement-controlled conical indentation FE model: (a) initial guess 1 (b) initial guess 2.

4.3.3. Conclusions on the Preliminary Application of the Inverse Method

An inverse method has been developed to extract material properties from the experimental results. The application of the inverse method for the case of

indentation testing has shown its capability of determining the elastic-plastic properties to a high accuracy.

It has been found that it is difficult to obtain the correct material properties from the indentation testing results using only the load-displacement data. The accuracy of the inverse approach is greatly improved by including the additional measurable surface profile after the indentation as the experimental data.

The scaling of the multiple objective function values has also been investigated and found to be important for the effectiveness and efficiency of the inverse method.

4.4. Characterisation of a P91 Steel by Small Punch Creep Tests and Inverse Approach

4.4.1. Introduction

The inverse approach was applied to determine the complete creep damage properties from the creep deformation data of SPC testing. A FE model of SPC testing has been developed with the modified Liu-Murakami creep damage model [132] taking account of the pre-straining effects [88]. The SPC testing FE model has been applied cooperatively with the inverse optimisation algorithm to determine the creep damage properties of a P91 steel from multiple sets of stain-time data of SPC tests carried out at 600°C.

4.4.2. General Methodology

The objective of the current study is to develop a novel methodology to predict the creep behaviour and determine the creep properties from the SPC tests and apply the method for a P91 steel at 600°C. The investigation consists

of a series of experiments (uniaxial tensile tests, uniaxial creep tests and SPC tests), FE modelling and inverse optimisation work on the same material. Figure 4.21 shows a flowchart of the current investigation. SPC tests were carried out to provide the objective data set for the inverse algorithm. A FE model was built based on the SPC tests carried out. Uniaxial tensile and creep tests were carried out to obtain the reference material properties (for validation and initialization) for the FE modelling and the inverse optimisation. An inverse method was developed to take the data obtained from the experiments and FE modelling as the inputs to predict the SPC behaviour and to extract the creep properties of the material.

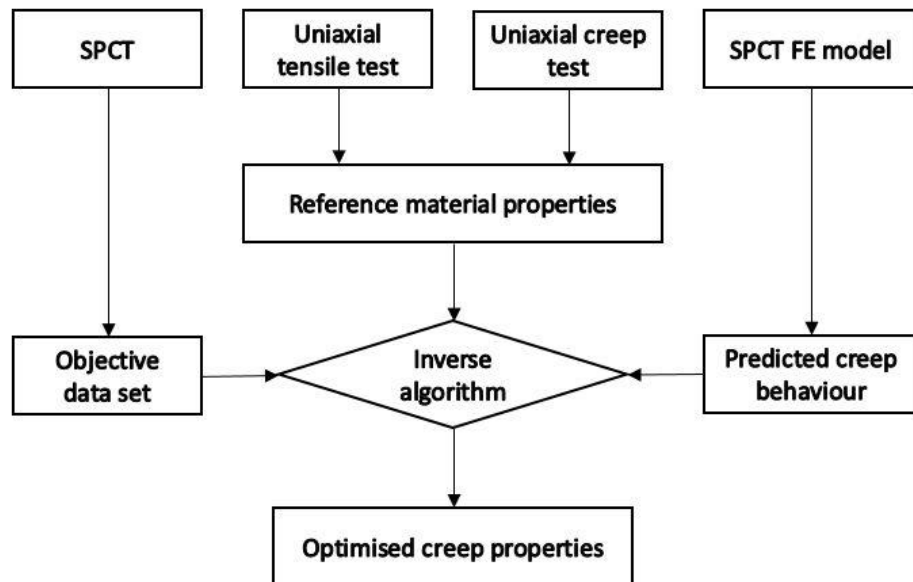


Figure 4.21. Flowchart of the current investigation

4.4.3. Experiments

Material Chemical Composition and Microstructure

A series of experiments, including uniaxial tensile tests, uniaxial creep tests and SPC tests, were carried out on a P91 steel at 600°C [88, 133]. The

chemical composition of the P91 steel used in the experimental work is presented in Table 4.9 in wt% [132]. The material microstructure of the virgin P91 steel obtained from the clamped, undamaged region is shown by Figure 4.22 [133]. The microstructure features homogeneous tempered martensite lathes with a number of finely distributed carbides, nitrides or carbo-nitrides along grain and sub-grain boundaries.

Table 4.9 Chemical composition (wt%) of the P91 steel used for the investigation [133]

Cr	Mo	C	Si	S	P	Al	V	Nb	N	W	Fe
8.60	1.02	0.12	0.34	<0.002	0.017	0.007	0.24	0.070	0.060	0.03	Bal

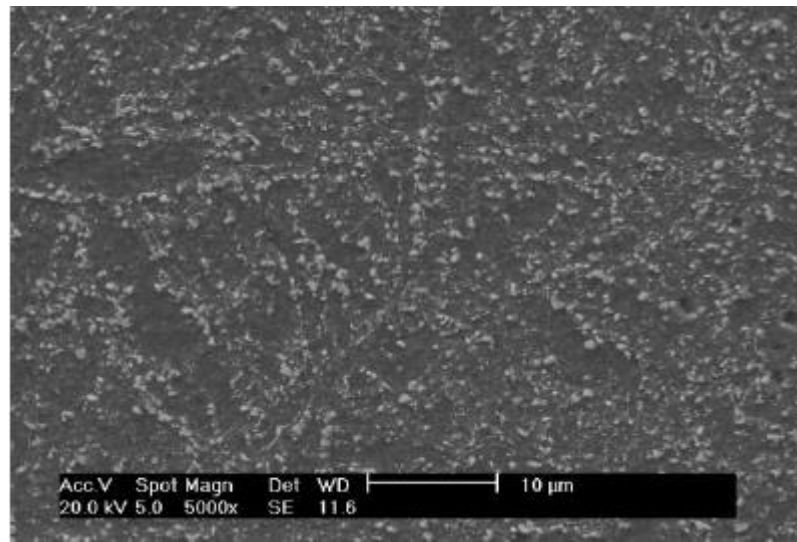


Figure 4.22. SEM image of the P91 steel used in this study [133]

Uniaxial Tensile and Creep Tests

The stress-strain curve obtained from the uniaxial tensile tests at 600°C on the P91 steel is shown in Figure 4.23 [133]. These data provide the reference elastic-plastic properties of the P91 steel in the current investigation. Uniaxial creep tests were carried out at various stress levels from 140MPa to 180MPa

[133]. Figure 4.24 shows the strain-time curves obtained from the uniaxial creep tests. Generally, the strain-time curves show the typical creep deformation/damage evolution: the primary stage with a decreasing strain rate, the secondary stage with the steady minimum strain rate and the tertiary stage with the increasing strain rate and the failure of the material. From the stress level of 140MPa to 180MPa, the minimum strain rate increases and the time to failure decreases. The empirical relationships from the CoP [84] are shown by equations 4.4 - 4.6, where P is the applied load, Δ is the total deformation, σ is the membrane stress, ε is the strain at the edge of contact, R_s is the punch radius ($R_s=1.25\text{mm}$), t_o is the original specimen thickness ($t_o=0.5\text{mm}$), a_p is the distance between the punch centre and the clamps ($a_p=2\text{mm}$) and K_{sp} is a non-dimensional correction factor, determined empirically for the material.

$$\frac{P}{\sigma} = 1.72476\Delta - 0.05638\Delta^2 - 0.17688\Delta^3 \quad 4.4$$

$$\varepsilon = 0.7959\Delta + 0.09357\Delta^2 - 0.0044\Delta^3 \quad 4.5$$

$$\sigma = \frac{0.3Pa_p^{0.2}}{K_{sp}R_s^{1.2}t_o} \quad 4.6$$

With the above equations, the uniaxial creep tests data can be used as a reference to estimate the required load level, i.e. the force to be applied to the punch, for the SPC tests to be carried out on the same material to obtain the same time to failure. Also, creep constitutive parameters can be extracted from the strain-time curves with a suitable creep damage model, e.g. Liu-Murakami's model [132], to provide the reference creep parameters for FE modelling of the SPC testing on the same material.

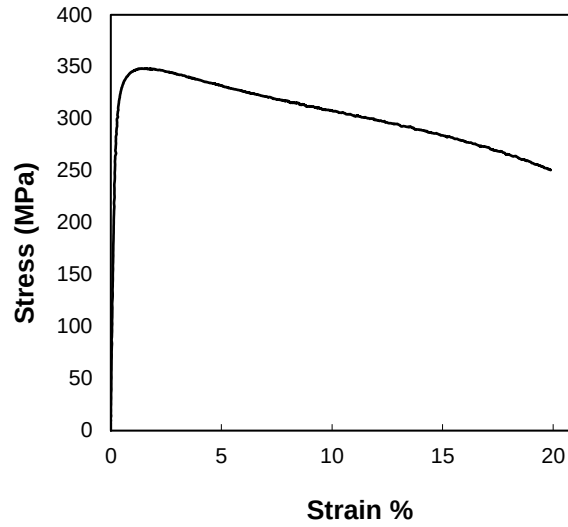


Figure 4.23. True stress-strain curve for a P91 steel at 600°C [133]

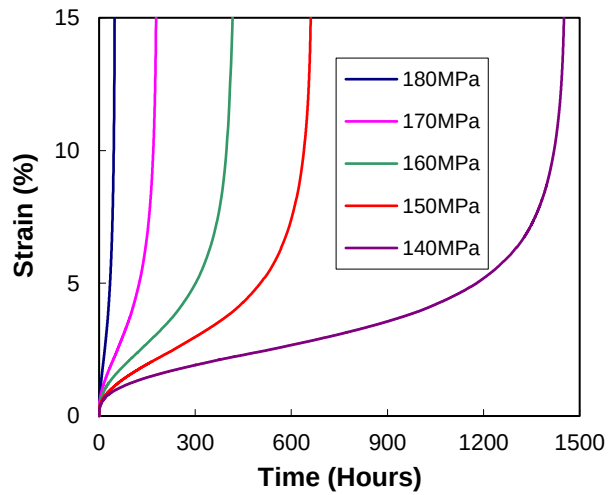


Figure 4.24. Strain-time curves of the uniaxial creep tests at various stress levels for a P91 steel at 600°C [133]

SPC Test

SPC tests were carried out with the same P91 steel used for the uniaxial tensile/creep tests at five different load levels: 25kg, 28kg, 30kg, 34kg and 40kg [88]. A schematic illustration of SPC test is shown in Figure 4.25, where

the punch radius $R_s=1.04\text{mm}$, the lower clamp radius $R_c=0.25\text{mm}$, the receiving hole radius $a_p=2\text{mm}$, the original diameter $D_0=8\text{mm}$ and the thickness of the disc specimen $t_0=0.5\text{mm}$. Figure 6 shows the displacement-time curves obtained from the SPCT. The fluctuations in displacement found in the primary stage of creep at the load of 25kg, shown in Figure 4.26, were due to the electronic noise resulted from the interference between two data acquisition units. Adjustments were made so that the interference was eliminated for the other tests. Similar to the uniaxial creep tests results shown in Figure 4.24, the three stages of the creep behaviours can be found in Figure 4.26. Figure 4.27 (a) and (b) show the variation of the minimum displacement rate (MDR) versus the load, P , and the variation of the time to failure, t_f , versus the load, respectively. Both the correlations between MDR (mm/h) and P (kg) and the correlations between t_f (h) and P can be expressed by power laws as expressed in equations 4.7 and 4.8, respectively. The fact that both of the correlations can be expressed in power law indicates that the results of the SPC tests are generally of high quality and consistency.

$$\text{MDR} = 6.457 \times 10^{15} P^{7.79} \quad 4.7$$

$$t_f = 1.995 \times 10^{17} P^{-10.24} \quad 4.8$$

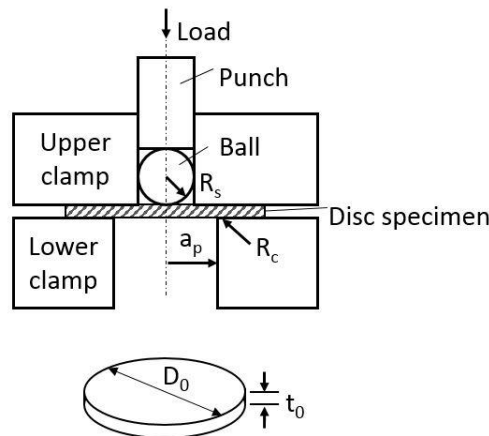


Figure 4.25. Schematics of small punch test

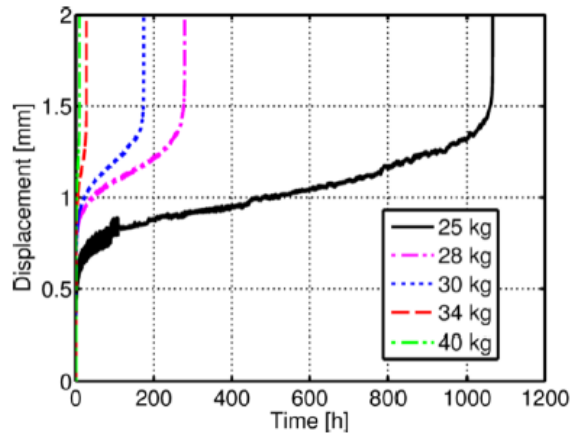
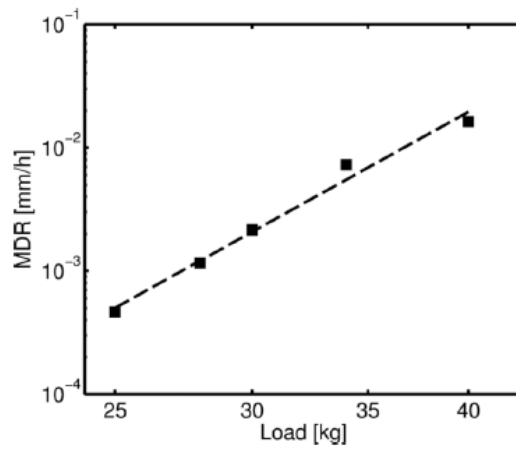
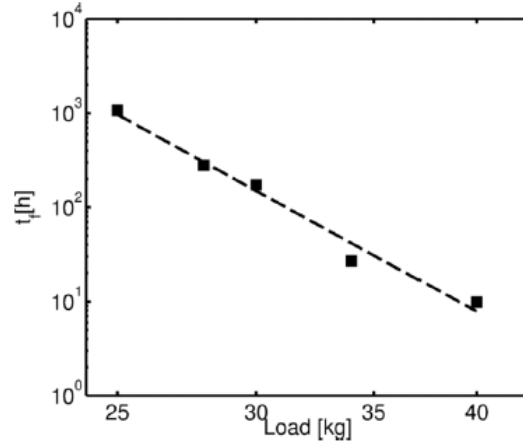


Figure 4.26. Punch displacement versus time for a P91 steel at 600°C with different load levels [6]



(a)



(b)

Figure 4.27. Logarithmic scale plots: (a) MDR versus load (b) time to failure versus load [88].

4.4.4. FE Modelling

4.4.4.1. Material Behaviour Models

A FE model of SPCT was developed with an elastic/plastic/creep material behaviour model using the commercial code ABAQUS. The stress-strain curve from a uniaxial tensile test on a P91 steel at 600°C [133], shown in Figure 3, was implemented in the material model in a tabular form. To simulate the creep behaviour of the material, Liu-Murakami creep constitutive damage model was applied to the FE model using a user subroutine [133]. The creep constitutive and damage evolution equations are shown as follows [132]:

$$\frac{d\varepsilon^c}{dt} = A\sigma_{EQ}^{N-1} \exp \left[\frac{2(N+1)}{\pi \sqrt{1 + \frac{3}{N}}} \left(\frac{\sigma_I}{\sigma_{EQ}} \right)^2 \omega^{3/2} \right] \quad 4.9$$

$$\frac{d\omega}{dt} = \frac{B[1 - \exp(-q_2)]}{q_2} (\sigma_D)^x \exp(q_2 \omega) \quad 4.10$$

$$\sigma_D = \alpha\sigma_I + (1 - \alpha)\sigma_{EQ} \quad 4.11$$

where ε^c is the creep strain, t is the time, σ_{EQ} , σ_I , σ_D are the equivalent stress, the maximum principal stress and the rupture stress respectively, ω is the creep damage value ($0 \leq \omega \leq 1$, there is no damage when $\omega = 0$ and the material is completely failed when $\omega = 1$) and A , B , χ , N , q_2 , α are the constitutive creep properties. A set of typical material properties are shown in Table 4.10 [34], in which the creep constitutive parameters were adopted from the uniaxial creep tests data shown in Figure 4, and they were taken as the reference material properties.

Table 4.10 Material constants for a P91 steel at 600°C adapted from ref. [133]

$E_0(MPa)$	ν	A	B	N	χ	q_2	α
1.5×10^5	0.3	1.51×10^{-30}	2.12×10^{-27}	11.795	10.953	5.3	0.3

4.4.4.2. FE model

The FE model was developed according to the same geometry and dimensions of the experiment setups as described previously. The boundary conditions of the FE model are shown in Figure 4.28. The punch and the clamps were modelled as rigid bodies, i.e. the deformation is neglected. The rotation and the horizontal displacement of the punch and the upper clamp were constrained. The radial and axial displacement and the rotation around the symmetry axis of the lower clamp were constrained. A clamp load $P_{\text{clamp}}=500$ kg was applied to the upper clamp and five levels of load (25 kg, 28 kg, 30 kg, 34 kg and 40 kg) were applied to the punch. The FE model of the specimen consists of 1186 bilinear axisymmetric 4-node elements with reduced integration (CAX4R in ABAQUS). The FE mesh used for the simulations is shown in Figure 4.29. Refined mesh was used in the region near the contact boundary between the punch and the specimen, where high stress and strain, large bending deformation and creep damage were expected. The contacts between the specimen, the punch and the clamps were defined

as surface-to-surface contact interactions. The friction coefficients between the surfaces were difficult to quantify, while a generally realistic value was used for the punch/specimen contact (0.3) and the clamp/specimen contact (0.8). The geometrical non-linearity formulation was used to account for the expected large deformation during SPC testing.

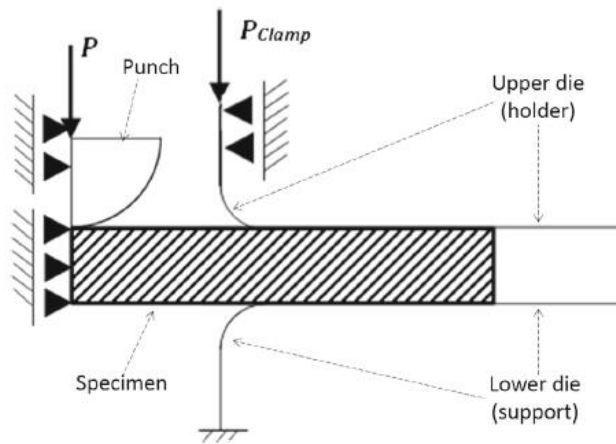


Figure 4.28. Boundary conditions of the SPCT FE model.

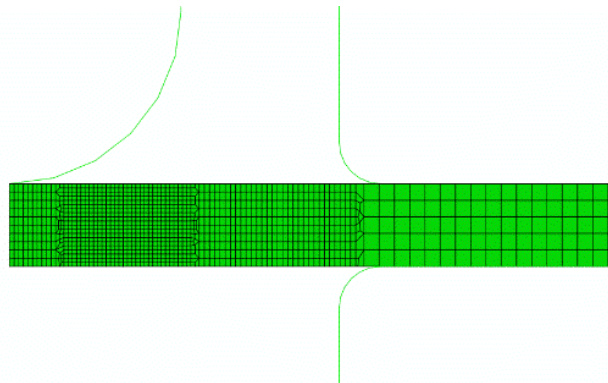


Figure 4.29. FE mesh used for the SPCT simulations.

4.4.5. Pre-Straining Effects

The initial equivalent plastic strain (PEEQ) predicted by FE modelling at the beginning of the creep step for the load of 25kg is shown in Figure 4.30. It is evident that there were plastic strains in the material, mainly in the contact region. Near the contact boundary, there is a region through the thickness with the pre-strain of about 16-22% (highlighted in red).

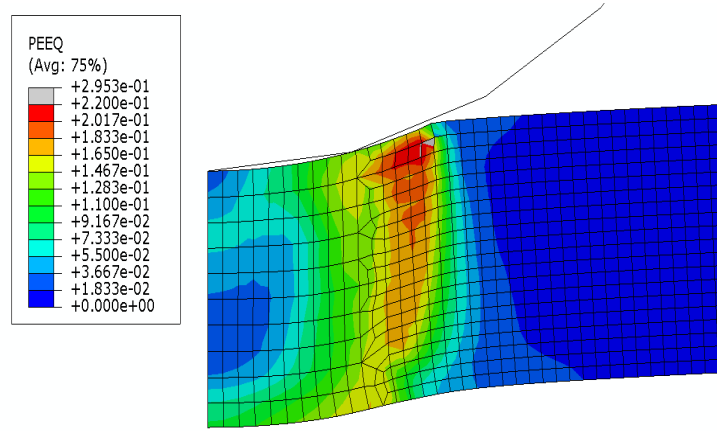


Figure 4.30. Initial equivalent plastic strain contour: 25kg.

The effects of pre-straining on the creep response of various materials have been investigated by other authors [134-138]. It has been revealed that the creep ductility, the minimum rate and the creep failure time can all be significantly affected by pre-straining [135, 136]. In order to take pre-straining effects into consideration, a modified Liu-Murakami's creep damage model has been proposed by Cortellino et al [88]. Two parameters, ϕ and ψ , are introduced into the creep constitutive equations (equations 4.9 and 4.10). Equations 4.12 and 4.13 show the definitions of ϕ and ψ , which account for the pre-straining effects on the minimum creep strain rate and the failure time, respectively. $\dot{\epsilon}_{min,0}^c$ and $t_{f,0}$ are the minimum strain rate and the failure time of material as received with no pre-straining, while the pre-

strained material is referred by $\dot{\varepsilon}_{min,p}^c$ and $t_{f,p}$. For example, when ϕ is greater than 1, the minimum strain rate of the pre-strained material increases compared to the material with no pre-strain, displaying a creep enhancement effect. On the contrary, when ϕ is less than 1, the minimum strain rate decreases when the material is pre-strained and it shows a creep resistance effect. Similarly, when ψ is greater than 1, the failure life is reduced by the pre-strain and it exhibits a creep enhancement effect, while it shows a creep resistance effect when ψ is less than 1. The modified Liu-Murakami's model is expressed as equations 4.14 and 4.15 [88].

$$\phi = \frac{\dot{\varepsilon}_{min,p}^c}{\dot{\varepsilon}_{min,0}^c} \quad 4.12$$

$$\psi = \frac{t_{f,0}}{t_{f,p}} \quad 4.13$$

$$\frac{d\varepsilon^c}{dt} = \phi A \sigma_{EQ}^{N-1} \exp \left[\frac{2(N+1)}{\pi \sqrt{1 + \frac{3}{N}}} \left(\frac{\sigma_I}{\sigma_{EQ}} \right)^2 \omega^{3/2} \right] \quad 4.14$$

$$\frac{d\omega}{dt} = \psi \frac{B[1 - \exp(-q_2)]}{q_2} (\sigma_D)^\chi \exp(q_2 \omega) \quad 4.15$$

The values of ϕ and ψ for a material can be obtained by carrying out uniaxial creep tests. The variation of the parameter ϕ and ψ with the plastic engineering pre-strain (ε_p) are fitted to the polynomial equations 4.16 and 4.17, respectively, at various stress levels. It is easy to show that ϕ and ψ are equal to 1 when there is no pre-strain, i.e. $\varepsilon_p = 0$. The fitting coefficients were obtained from the uniaxial creep test data for different stress levels and varying amount of pre-straining and then averaged to give a set of representative fitting coefficients for the material. Tables 4.11 and 4.12 show

the averaged fitting coefficients for ϕ and ψ obtained from the uniaxial creep tests on the P91 steel with different pre-strain percentages [88].

$$\phi = \exp[a_1 \varepsilon_p] + b_1 \varepsilon_p^2 + c_1 \varepsilon_p^3 + d_1 \varepsilon_p^4 \quad 4.16$$

$$\psi = \exp[a_2 \varepsilon_p] + b_2 \varepsilon_p^2 + c_2 \varepsilon_p^3 \quad 4.17$$

Table 4.11 Averaged fitting coefficients for ϕ [78].

a_1	b_1	c_1	d_1
-5.9534	6.69×10^{-2}	-8.8×10^{-3}	3.236×10^{-4}

Table 4.12. Averaged fitting coefficients for ψ [78]

a_2	b_2	c_2
-3.241	2.61×10^{-2}	4.763×10^{-4}

The contour plots of the ϕ and ψ parameters from the FE modelling for the punch load of 25kg with the modified creep model are shown in Figure 4.31 (a) and (b), respectively. The values of ϕ and ψ were calculated from equations 4.16 and 4.17 using the equivalent plastic strain at the beginning of the creep step shown in Figure 10. It can be found that the distribution and the variation of ϕ and ψ are consistent with the equivalent plastic strain. In the region where there is zero plastic strain (the unsupported region) ϕ and ψ are equal to 1, which means there is no pre-straining effect. However, in the contact area, ϕ and ψ are less than 1, which indicates there are creep resistance effects due to pre-straining. Global creep resistance effects are revealed by the punch displacement versus time curves in Figure 4.32. It shows the comparisons between the experiment results and the results from the FEA of the SPC tests on the P91 steel for the load of (a) 25kg and (b) 30kg at 600°C with and without pre-strain. For example, for the load of 25kg,

the MDR decreased by about 50% and the time to failure increased by about 200% when the pre-straining was included in the FE model, leading to a much more accurate prediction in the MDR and time to failure compared to the FE model without pre-straining, as Figure 4.32 (a) shows. Similarly, the FEA predictions of MDR and the failure time under the punch load of 30kg were both improved with the inclusion of the pre-straining effects in the FE model as shown in Figure 12(b).

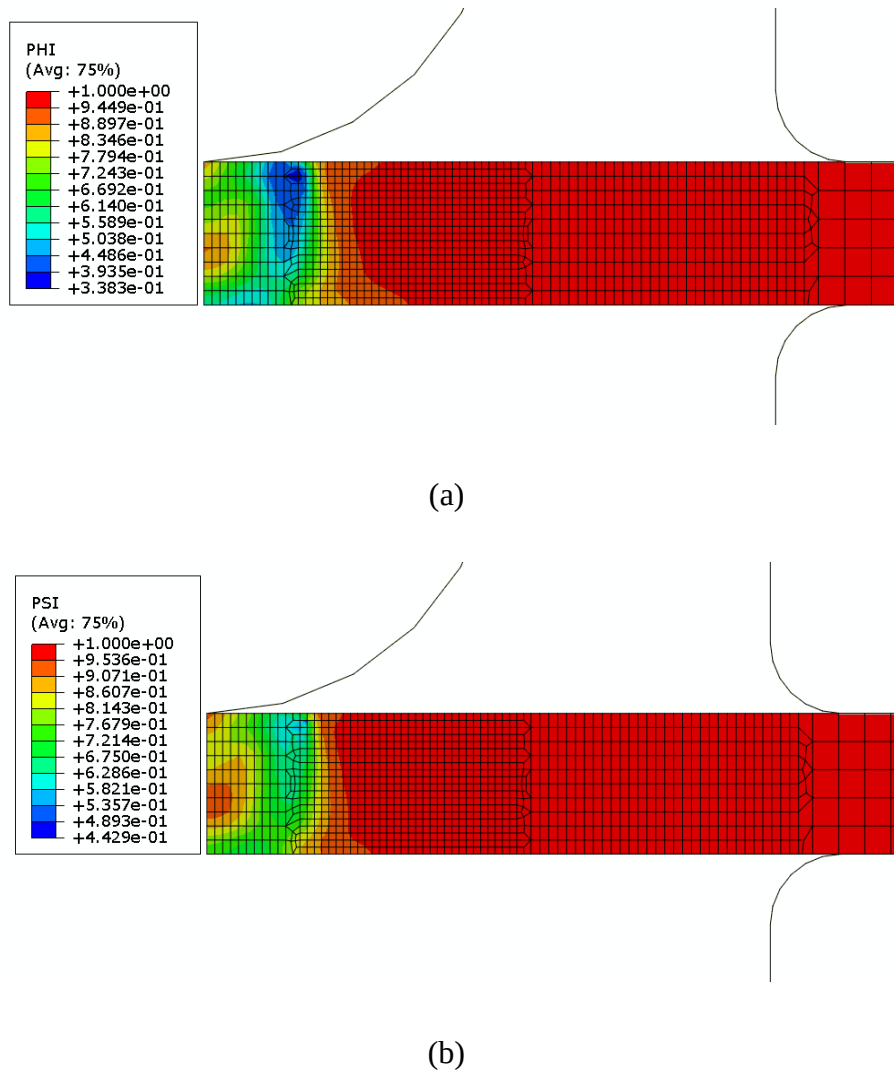
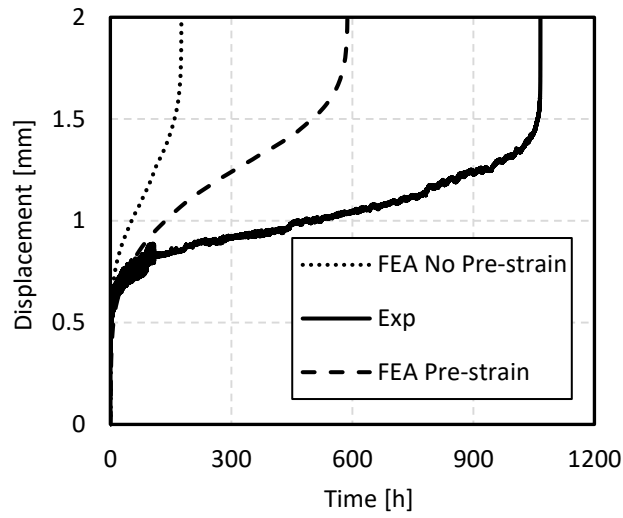
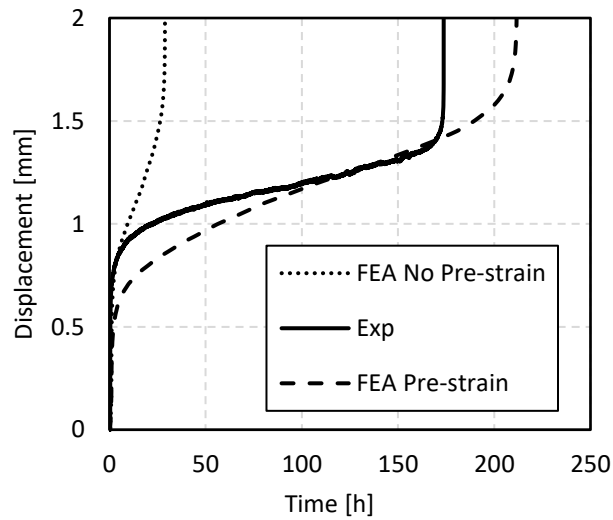


Figure 4.31. FEA contours for punch load of 25kg: (a) ϕ (b) ψ .



(a)



(b)

Figure 4.32. Punch displacement versus time: (a) 25kg (b) 30kg.

4.4.6. Development of the Inverse Method

An inverse method was developed to determine the creep properties from the SPCT data iteratively. As shown in the flowchart (Figure 4.33), initially, a reasonable estimation of the creep properties, e.g. based on the uniaxial creep tests, was inputted into the FE model to simulate SPCT at different load levels.

The pre-straining effects was included in the inverse method for the first time. The displacement-time data is extracted from the FE results and compared to the experimental data obtained from the experiment with the corresponding load level. The differences between the predicted and the experimental creep data (the output of the objective function) are fed back into a nonlinear least-square optimisation toolbox to generate the new inputs for the next iteration. This cycle is continued until the variation of the objective function is within the predefined tolerance, and the inputs of the last iteration will then be accepted as the optimised parameters (creep material properties).

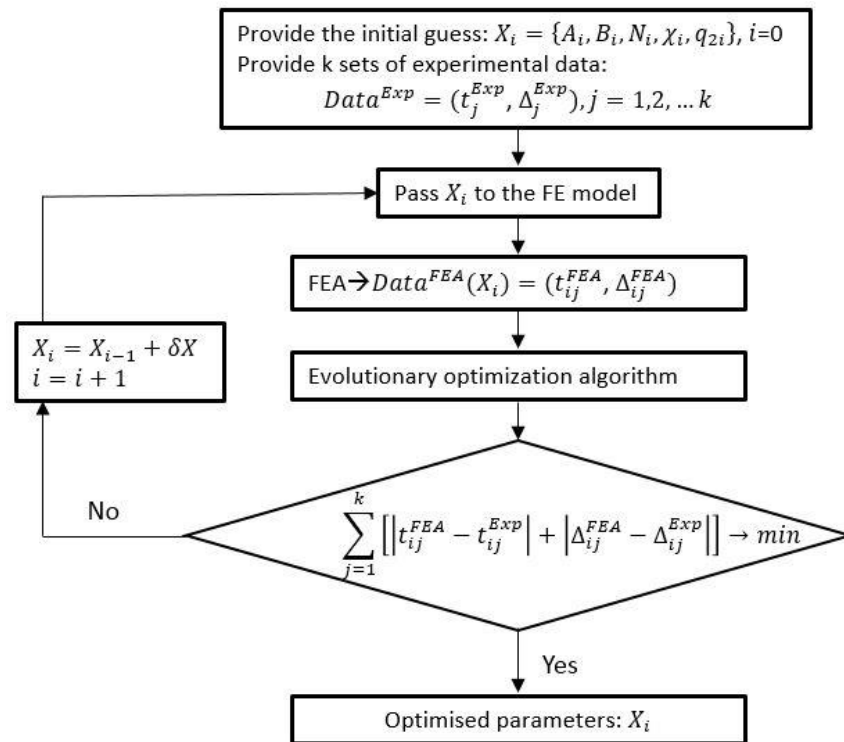


Figure 4.33. Flowchart of the inverse SPCT algorithm

The displacement-time data from the SPC tests carried out at load levels of 28kg, 30kg and 34k, were taken as the target curves for the optimisation

process. The reference material properties presented in Table 4.10 were supplied to the optimisation algorithm as the initial values of the creep properties (the elastic-plastic properties were set as constants).

It should be noted that the creep material constants have very different orders of magnitude which are not taken into account by the optimisation algorithm by default. Therefore, it is necessary to normalize the material properties to the same or similar orders of magnitudes. Also, the minimum step size and the parameter values should be kept in a reasonable ratio. If the step size is too small compared to the parameter values, the optimisation process may not effectively progress. On the other hand, if the changes made to the material properties are too large, then it might be difficult for the optimisation algorithm to converge. In addition, because the displacement rate is constant in the secondary stage, while it increases in the tertiary stage of SPC test, data points were taken in a higher frequency in the tertiary stage to better capture the shape of the curve which may result in a better quality of convergence. In addition, the values of the parameters N and χ should be constrained within reasonable boundaries since the strain and the damage evolution are highly sensitive to these two exponents, i.e. an extreme value could lead to convergence difficulty in the ABAQUS FE modelling and the inverse analysis could be interrupted.

The objective function of this optimisation algorithm consists of two parts: displacement and time. Because the magnitude of displacement (in unit of mm) and time (in unit of hour) were in different orders of magnitude, i.e. displacement was in the order of 10^0 while time was in the order of 10^2 - 10^3 , they were scaled to a similar order (10^0) so that the objective function value reflects the differences in displacement and time equally, instead of being dominated by the variable with the much larger magnitude (time in this case). All the creep constants were then scaled to the order of 10^{-6} for a similar level of sensitivity to the step change difference.

4.4.7. Results and Discussion

Application of the Inverse Method

Figure 4.34 shows the evolutions of the predicted creep properties, presented as the ratio of the predicted properties in every iteration to the final optimised properties, which are shown in Table 4.13. The comparison between the displacement-time curves obtained from the experiments, the initial FE modelling and the final optimised FE modelling is shown in Figure 4.35.

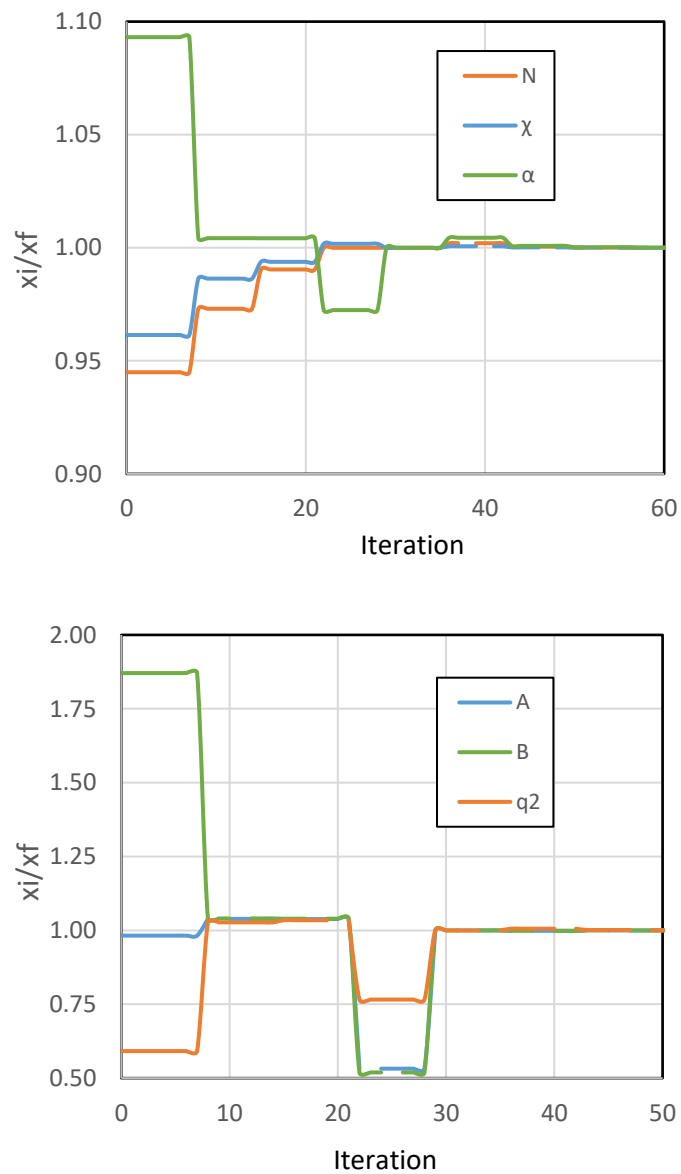


Figure 4.34. Evolutions of the predicted creep properties.

Table 4.13 Optimised creep properties for the P91 steel at 600°C.

A	B	N	χ	q_2	α
2.28E-31	1.41E-26	12.8790	10.8901	5.5778	0.2150

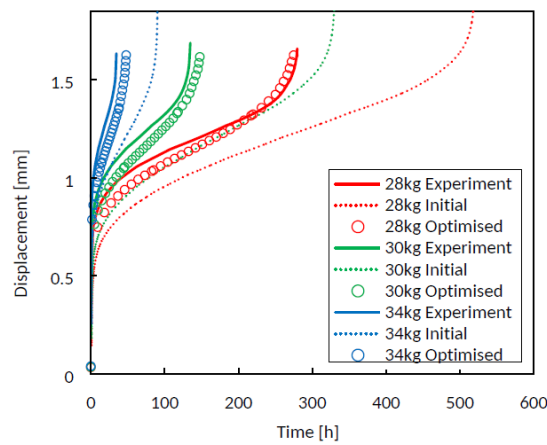


Figure 4.35. Displacement-time curves from the experiments and the inverse method

4.4.8. Discussion

As shown in Figure 4.35, the optimised predictions of the displacement-time curves are much closer to the experimental results compared to the initial predictions at all three load levels, regarding the time to failure especially. For example, the error percentage in time to failure for the load level of 28kg is about 70% for the initial prediction while it is about 10% for the optimised prediction. Similarly, at the load of 30kg the initial error percentage in time to failure is about 90% while it is less than 40% for the optimised prediction. However, the predicted MDR values are less accurate compared to the experimental results. This may be due to the constraining conditions being

inadequate. Figure 4.34 shows that all the predicted creep properties converged effectively to the optimised values within a relatively small number of iterations, e.g. 50 to 60 iterations.

Figure 4.36 shows a comparison between the predicted damage contours obtained from FE modelling with the optimised creep properties and the SEM images of the cross section of the specimen obtained from the SPCT with the punch load of 25kg interrupted at different stages. The evolution of the creep damage predicted by FEA agrees well with the SEM images, e.g. the critical creep damage is initiated on the bottom surface near the contact edge and then gradually spreads through the whole section area. Also, it can be found from Figure 4.36 (c) that the geometry of the crack predicted by FEA (shown as the region with the maximum damage value) is similar to the SEM observation. Figure 4.36 (c) also shows a necking phenomenon near the crack predicted by the FE modelling, similar to the SEM image. These agreements between the FEA predictions and the SEM observations provide good validation for both the FE model considering the pre-straining effects and the inverse method developed in the current investigation.

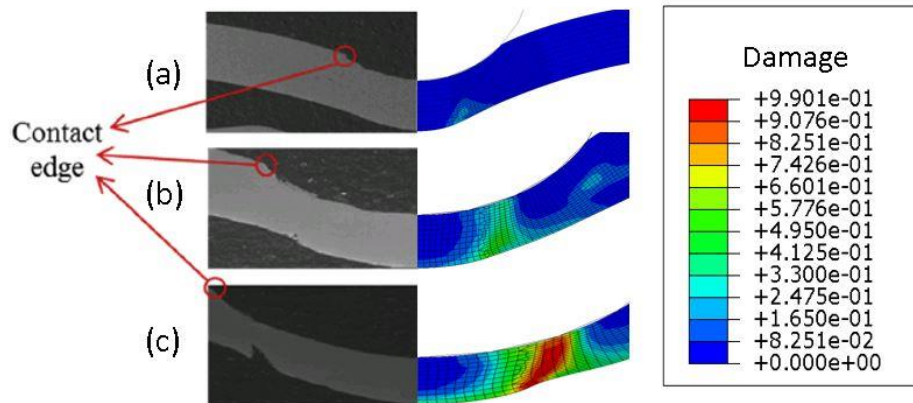


Figure 4.36 SEM images (left) from Ref. [6] for a small punch creep test on a P91 steel at 600°C with a load of 25 kg, interrupted after (a) 2h (b) 200h (c) 669h and the FE damage contours (right).

It should be noted that although the uniaxial creep properties were used as the reference properties for the application of the inverse method in this investigation, in many cases the uniaxial creep properties may not be available. In those cases, the initial guess for the inverse method should be estimated by using the CoP equations 4.4 – 4.6 to convert the SPC testing results to uniaxial creep test results.

4.4.9. Conclusions

The inverse method developed in the current investigation has been proven to be capable of extracting the material creep properties from the displacement-time data obtained from SPC tests on a P91 steel at 600°C. The effects of the pre-straining occurring in the early stage of SPC testing have been considered for an inverse method for the first time. The optimised predictions in the MDR and time to failure were within an acceptable range of error percentage compared to the experimental results. Generally, the optimisation processes converge within a relatively small number of iterations meaning that the optimisation algorithm is effective, and the computing time is acceptable. Also, the elastic-plastic-Liu-Murakami damage model has been incorporated, for the first time, in the inverse analysis for SPC testing.

One major concern over the inverse method with FE modelling is the cost of computing. It should be noted that it is possible to reduce the computing time for the current inverse method in the future since the FE modelling is controlled by a pre-defined step time (i.e. twice as the time to failure of the experiment at each load level) and hence there could be some unnecessary computing work as it progresses close to failure when the time increment is small and the displacement rate approaches infinity. Computing time could be reduced by monitoring the punch displacement constantly (an effective method is required to do so) and terminating the simulation when the punch displacement or displacement rate exceeds a certain threshold value. Also, it

should be noted that the fitting coefficients for ϕ and ψ were obtained from a relatively small number of tests [88]. Extended experimental work in pre-strained uniaxial creep tests is expected to lead to a better estimation of ϕ and ψ parameters for the FE modelling of SPC testing, hence to a further improved prediction in the creep behaviours from the inverse method developed in this investigation. Another noteworthy factor for the FE modelling of SPC testing is the initial plasticity damage. A more robust method is needed in future to deal with the initial plasticity damage and its effects on the subsequent creep deformation. In addition, the current optimisation algorithm can be improved by including extra constraint equations based on measurable quantities such as the minimum strain rate and the time to failure obtained from the SPC testing. The application of these constraints could lead to an improved prediction of the creep behaviours reflecting the realistic physics presented by the experimental results.

Chapter 5 - Development of a Two-Material Miniature Specimen Testing Technique and the Associated Inverse Method

5.1. Introduction

The objective of the current study is to develop a miniature specimen testing technique and an inverse method to determine the elastic-plastic and creep damage properties of an unknown material from a two-material system. The proposed testing method can be directly related to the conventional uniaxial test. Single material miniature specimen tensile tests and creep tests were carried out on an aluminum alloy and a P91 steel. Future tests on the miniature coating-substrate specimen are planned. The inverse method developed shows the potential to extract full stage creep damage properties of an unknown material from the compound creep testing results of a two-material system based on theoretical experiments of the two-material system.

5.2. Two-Material System

The general methodology of the current study is 1) carrying out single-material tests for a substrate material and a coating material to obtain the single material responses; 2) carrying out two-material system (e.g. coating on substrate) tests to obtain the compound responses; 3) applying the developed inverse method to determine the unknown material properties; 4) using the determined material properties for semi-analytical computation or FE modelling of the single-material tests and comparing the predicted material behavior to the experimental results to validate the reliability of the inverse method.

A two-material specimen in tension is shown in Figure 5.1. The two layers have equal length, L , equal width, d , and different thicknesses, h_1 and h_2 ,

therefore, different cross-sectional areas, S_1 and S_2 . It is assumed that the properties of one of the materials are known in this study (e.g. the substrate material properties are known for a coated system).

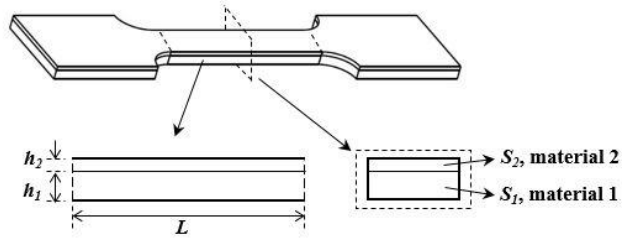


Figure 5.1. A two-material specimen in tension.

5.2.1. Tensile Behaviour

The equilibrium equation for the two-material system under tensile force, P , can be expressed as follows:

$$\sigma_1 S_1 + \sigma_2 S_2 = P \quad 5.1$$

where σ_1 , σ_2 are the stresses in materials 1 and 2, respectively. S_1 and S_2 are the cross-sectional areas of materials 1 and 2, respectively. The compatibility equation (the two layers have equal displacements) is:

$$u_1 = u_2 = u \quad 5.2$$

or

$$\varepsilon_1 = \varepsilon_2 = \varepsilon \quad 5.3$$

where u_1 , u_2 and ε_1 , ε_2 are the displacements and strains in materials 1 and 2, respectively. Assuming a power law hardening behaviour, the elastoplastic stress-strain relationships are expressed as follows:

$$\sigma = \begin{cases} E\varepsilon, & \varepsilon < \frac{\sigma_y}{E} \\ K\varepsilon^n, & \varepsilon \geq \frac{\sigma_y}{E} \end{cases} \quad 5.4$$

where $K = E^n \sigma_y^{1-n}$. E , σ_y and n are the Young's modulus, yield stress and hardening exponent, respectively. The stress in the known material 1 can be determined as follows:

$$\sigma_1 = \begin{cases} E_1 \varepsilon, & \varepsilon < \frac{\sigma_{y1}}{E_1} \\ E_1^{n_1} \sigma_{y1}^{1-n_1} \varepsilon^{n_1}, & \varepsilon \geq \frac{\sigma_{y1}}{E_1} \end{cases} \quad 5.5$$

where E_1 , σ_{y1} and n_1 are the Young's modulus, yield stress and hardening exponent of material 1, respectively. The stress in the unknown material 2 can be determined from Equ (1) and (3):

$$\sigma_2 = \frac{P - \sigma_1 S_1}{S_2} \quad 5.6$$

The constitutive equations for material 2 are as follows:

$$\sigma_2 = \begin{cases} E_2 \varepsilon, & \varepsilon < \frac{\sigma_{y2}}{E_2} \\ E_2^{n_2} \sigma_{y2}^{1-n_2} \varepsilon^{n_2}, & \varepsilon \geq \frac{\sigma_{y2}}{E_2} \end{cases} \quad 5.7$$

where E_2 , σ_{y2} and n_2 are the Young's modulus, yield stress and hardening exponent of material 2, respectively. They can be determined numerically.

5.2.2. Creep Behaviour

Steady-State Creep

For steady-state creep, the creep strain rate, $\dot{\varepsilon}^c$, can be expressed as follows:

$$\dot{\varepsilon}^c = A\sigma^n \quad 5.8$$

where σ is the stress and A and n are material constants. Assuming that the properties of material 1, i.e. A_1 and n_1 , are known, the stress in material 1 can be determined with the measured strain rate $\dot{\epsilon}^c$ using Eq (8) as follows:

$$\sigma_1 = \left(\frac{\dot{\epsilon}^c}{A_1} \right)^{\frac{1}{n_1}} \quad 5.9$$

with the equilibrium and compatibility equations (1)-(3), the stress in material 2 can be determined as follows:

$$\sigma_2 = \frac{P - \left(\frac{\dot{\epsilon}^c}{A_1} \right)^{\frac{1}{n_1}} S_1}{S_2} \quad 5.10$$

The creep properties of material 2, A_2 and n_2 , can be determined numerically from the following constitutive equation:

$$\dot{\epsilon}^c = A_2 \sigma_2^{n_2} = A_2 \left(\frac{P - \left(\frac{\dot{\epsilon}^c}{A_1} \right)^{\frac{1}{n_1}} S_1}{S_2} \right)^{n_2} \quad 5.11$$

Creep Damage

A Kachanov type creep damage model can be used to represent the creep behavior (e.g. [33]). The formulations for the case of uniaxial stress state can be expressed as follows:

$$\dot{\epsilon}^c = A \left(\frac{\sigma_{eq}}{1-\omega} \right)^n \quad 5.12$$

$$\dot{\omega} = B \frac{\sigma_r^\chi}{(1-\omega)^\phi} \quad 5.13$$

where σ_{eq} and σ_r are the equivalent and rupture stresses, respectively ($\sigma_{eq} = \sigma_r$ in the case of uniaxial stress); ω ($0 \leq \omega \leq 1$) is a damage variable; A , n , B , χ , and ϕ are material constants. For material 1 shown in Figure 5.1, the constitutive equations are:

$$\dot{\epsilon}^c = A_1 \left(\frac{\sigma_1}{1-\omega_1} \right)^{n_1} \quad 5.14 (a)$$

$$\dot{\omega}_1 = B_1 \frac{\sigma_1^{\chi_1}}{(1-\omega_1)^{\phi_1}} \quad 5.14(b)$$

Assuming the creep properties of material 1 (A_1 , n_1 , B_1 , χ_1 , and ϕ_1) are known, the stress σ_1 can be determined from Eq 5.14 (b) by applying the experimentally determined minimum strain rate (MSR), $\dot{\epsilon}_{min}^c$, for the steady-state creep, during which there is no creep damage, i.e. $\omega_1 = 0$:

$$\sigma_1 = \left(\frac{\dot{\epsilon}_{min}^c}{A_1} \right)^{\frac{1}{n_1}} \quad 5.15$$

With the equilibrium and compatibility equations 5.1-5.3 and Eq 5.15, the stress in material 2 can be determined as follows:

$$\sigma_2 = \frac{P - \sigma_1 S_1}{S_2} = \frac{P - \left(\frac{\dot{\epsilon}_{min}^c}{A_1} \right)^{\frac{1}{n_1}} S_1}{S_2} \quad 5.16$$

The creep constitutive equations of material 2 are as follows:

$$\dot{\epsilon}^c = A_2 \left(\frac{P - \left(\frac{\dot{\epsilon}_{min}^c}{A_1} \right)^{\frac{1}{n_1}} S_1}{S_2 (1 - \omega_2)} \right)^{n_2} \quad 5.17a$$

$$\dot{\omega}_2 = \frac{B_2}{(1 - \omega_2)^{\phi_2}} \left(\frac{P - \left(\frac{\dot{\epsilon}_{min}^c}{A_1} \right)^{\frac{1}{n_1}} S_1}{S_2} \right)^{\chi_2} \quad 5.17b$$

where A_2 , n_2 , B_2 , χ_2 , and ϕ_2 are the unknown creep constants of material 2, which can be determined numerically using Eq 5.16, 5.17a and 5.17b.

5.2.3. Forward Analysis of the Two-Material System

Forward analysis can be performed for any two-material system with the known material properties based on the analysis presented in section 5.2.2. A demonstration of the forward analysis of a two-material creeping system with fictional material properties is presented in this section. The elastic-plastic behaviours were neglected for simplification since the elastic-plastic

deformation was relatively small in these cases. Table 5.1 shows the creep damage properties of two fictional materials M1 and M2. The forward analysis was implemented through a semi-analytical computing script developed based on the Kachanov creep damage model as presented in section 5.2.2. Since the creep properties of the two materials are known, the compatibility condition (equal creep strain rate), the equilibrium condition and the Kachanov creep damage equations can be used for semi-analytical solutions. To verify the accuracy of the analytical solutions, FE analysis was carried under the same conditions and with the same material properties shown in Table 5.1, using the commercial code ABAQUS 6.14-1. A three-dimensional eight-node brick element with reduced integration (C3D8R in ABAQUS) was used for the FE model. Figure 5.2 shows the stress contour obtained from the FE analysis (a quarter section of the specimen shown in Figure 2). The stresses in materials 1 and 2 in most regions of the uniform section are close to be in a uniaxial state, which is consistent with the assumption in the analytical solutions.

The comparison between the semi-analytical solutions and the FE results is shown in Figure 5.3. Figure 5.3 (a) shows that the compound creep strains (the creep strains in the two material layers M1 and M2 are equal in both cases) obtained from semi-analytical solutions and FE analysis agree very well. Figure 5.3 (b) shows the creep damage evolutions of the two materials. The damage history of the two material layers are different; the MSR in material M1 is much higher than that in material M2. The analytical solutions and the FE analysis show very close results again. For the semi-analytical solutions, the instantaneous stresses in the two materials were obtained semi-analytically using the instantaneous damage values and creep strain rates obtained from the Kachanov creep damage model. The slight differences found between the results from the semi-analytical solutions and FE analysis might be mainly due to that a uniaxial stress case was assumed for the analytical solutions while that was not exactly the case for the FE analysis. As a result, the equivalent stresses obtained from the FE analysis were slightly lower than the analytically obtained stresses. This explains the fact that the

creep strains and creep damage values from the FE analysis are slightly lower than the corresponding analytical results, as shown in Figures 5.3 (a) and (b).

Table 5.1. The material properties of the fictional materials M1 and M2.

Material	E	ν	A	n	B	χ	ϕ
M1	150GPa	0.3	5×10^{-24}	11	2×10^{-22}	11	5
M2	180GPa	0.3	5×10^{-25}	10	2×10^{-24}	10	4.5

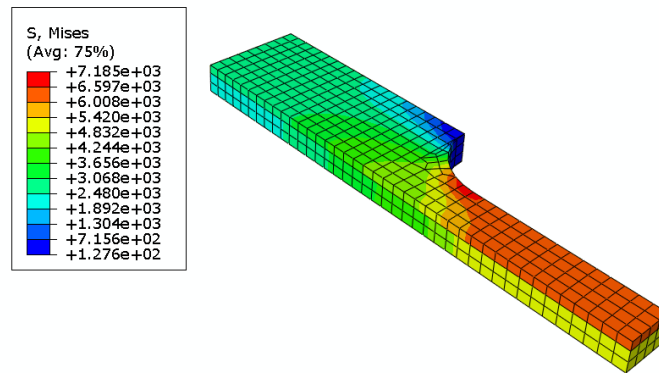
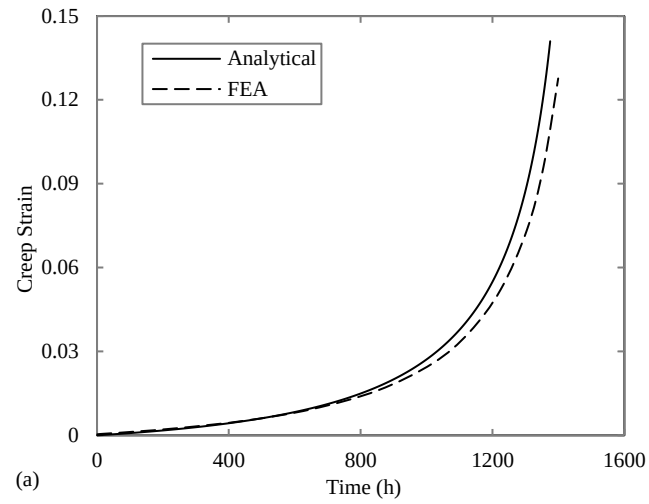
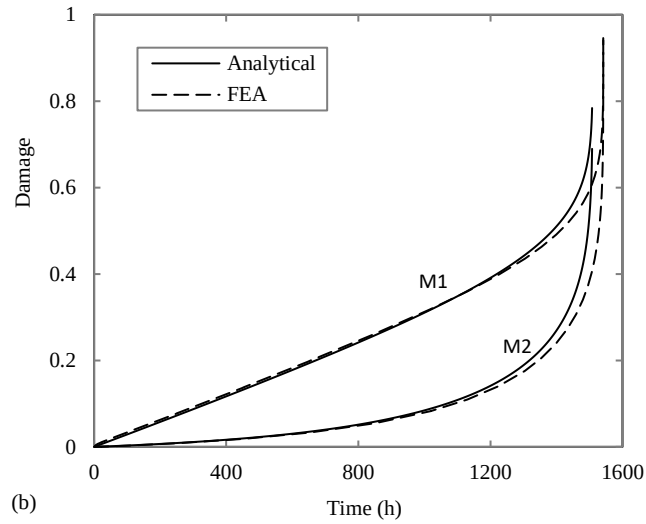


Figure 5.2. von Mises stress contour of the two-material system with the fictional materials M1 and M2 obtained from FE analysis.



(a)



(b)

Figure 5.3. Forward creep analysis for the two-material system (M1 and M2) under the load of 42N: (a) creep strain (b) creep damage.

5.3. Inverse Method and Application

5.3.1. The Inverse Method

An inverse method was developed based on the forward analysis method presented in section 5.2.3. FE modelling can be avoided to save computing

time since the results of the semi-analytical computations have been proven to be very close to the FE modelling. Figure 5.4 shows a flowchart of the inverse algorithm. The experimental compound creep test results are supplied as the target values. The known material creep properties and an initial guess of the unknown material creep properties are supplied to the forward analysis method developed based on the Kachanov creep damage model (section 5.2.2). A nonlinear optimization algorithm is used to minimize the differences between the predicted compound creep responses and the experimental results until a set of optimized creep damage properties are obtained for the unknown material.

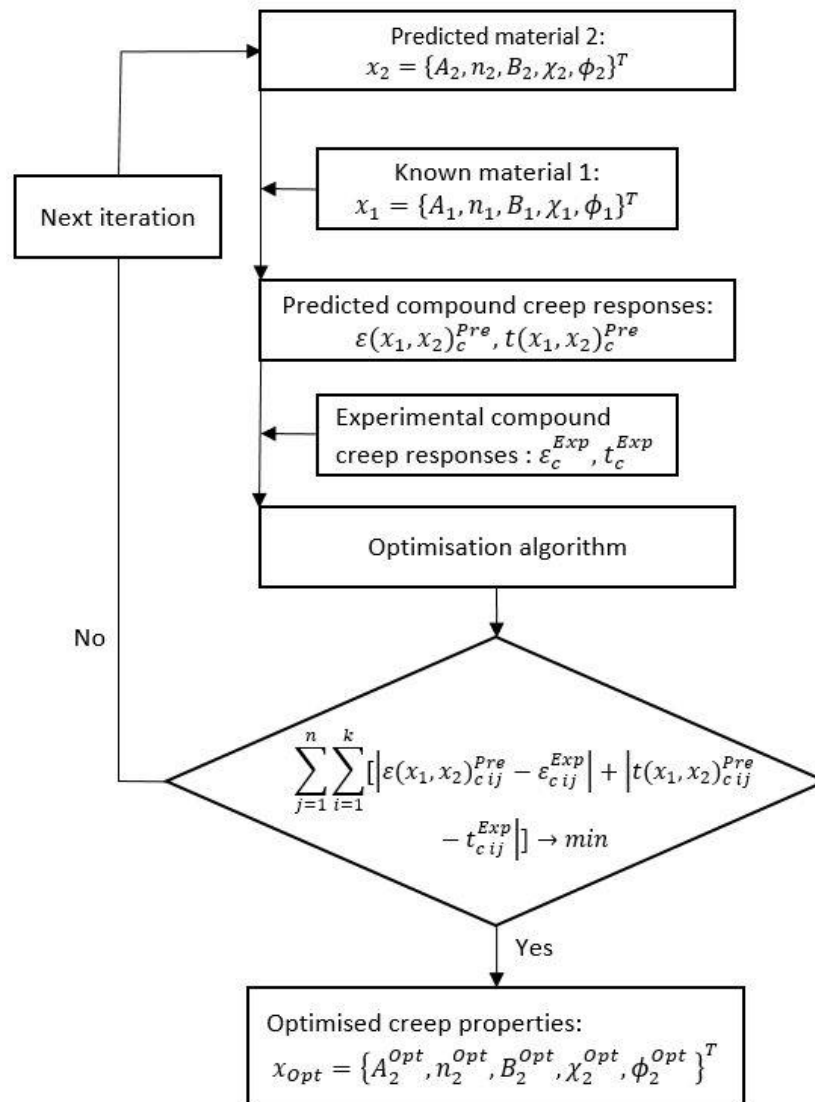


Figure 5.4. Flowchart of the inverse method.

5.3.2. Application of the Inverse Method

To demonstrate the application of the proposed inverse method, it was applied to the two-material system used for the forward analysis presented in section 2.3, assuming that the properties for material M1 are known while the properties for material M2 are unknown. The objective was to determine the creep damage properties of M2 from the compound creep responses. Three different loads, 44N, 46N and 48N, were applied to the two-material system with the creep properties for materials M1 and M2 shown in Table 5.1 to produce three “theoretical” compound creep stress-strain curves (equivalent to the experimental compound creep responses shown in Figure 5) as the targets of the optimization. Figure 5.5 shows the strain-time curves obtained from the optimized creep damage properties compared to the theoretical experiment. It can be seen that the optimized results agree very well with the target curves for all three different loading forces regarding the creep strain rate and the time to failure. To evaluate the quality of the optimization, the creep strain-time curves were also computed numerically using the creep properties of M2 and the optimized creep properties for a single-material model with three different loading forces: 75N, 80N and 85N. The comparison is shown in Figure 5.6. It can be seen that the agreement is still acceptable, i.e. the strains during the steady-state creep stage are very close for the material M2 (Table 1) and the optimized material at all three loading forces. Although the deviation increases as the creep develops near to failure. These results reveal that the optimized creep properties were close to the actual properties of the target material M2. It should be noted that the current inverse optimization algorithm is still sensitive to the initial values of the unknown creep damage properties (e.g. some initial values could lead to difficulty in convergence to the desired tolerance or extended computing time). This sensitivity to the initial conditions should be minimized in further development.

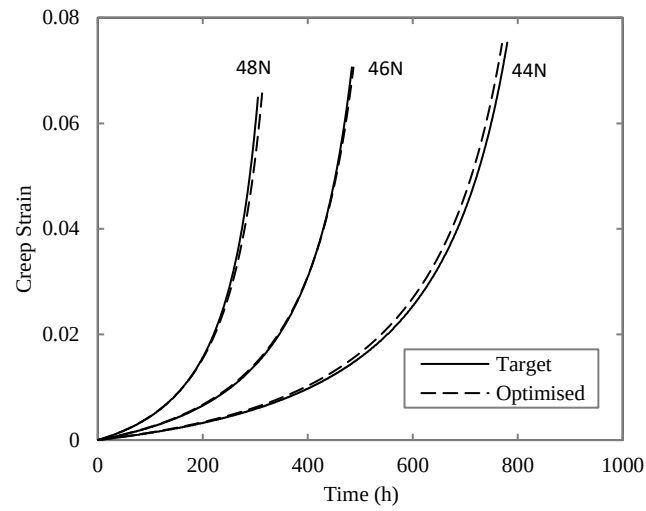


Figure 5.5. The inverse optimized curves and the target curves.

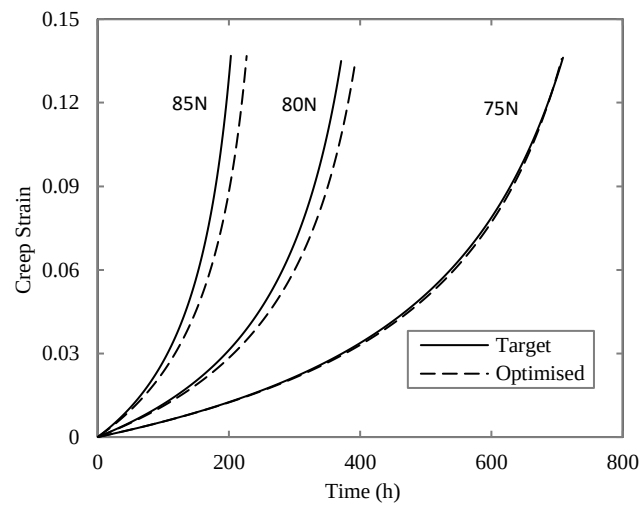


Figure 5.6. The single-material model computations for material M2 (Table 1) and the optimized creep properties.

5.4. Development of a Novel Miniature Specimen Testing Technique

5.4.1. Testing Method

A miniature specimen tensile/creep testing technique has been developed. Tensile tests have been carried out for an aluminum alloy with both miniature specimens and large specimens. Creep tests have been performed at 650°C on a P91 steel at different stress levels: 100MPa, 93MPa and 87MPa for both the miniature and large specimens. Figure 5.7 shows the geometry and dimensions of the miniature specimen. The testing rig is shown in Figure 3.2. The loading assembly was made of a high temperature resistant nickel-based superalloy, i.e. Nimonic 115. The specimen was clamped at both ends by specially manufactured clamps with machined surface features to strengthen the grip. The displacement was measured by two linear variable displacement transducer (LVDT) connected to both sides of the loading part of the machine. The mounted specimen was kept inside a furnace for the tests requiring heating.

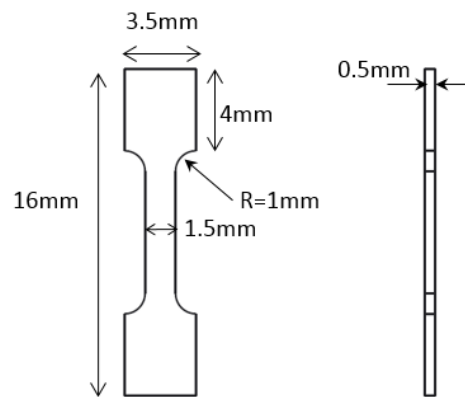


Figure 5.7. Geometry and dimensions of the miniature specimen.

5.4.2. Miniature Tensile Tests on an Al Alloy

Tensile tests were carried out on an aluminum alloy to be compared to the tensile testing results obtained from the large specimen to validate the developed miniature testing method. Figure 5.8 shows the comparison between the tensile testing results obtained from the miniature specimens and the large specimens at different temperatures. The results from the two types of tests are generally comparable. The differences in the strain measurement might be due to the random error in the clamping location, which is manually fixed. Also, the sensitivity of the detectors could be another factor of influence. The miniature specimen could also be more sensitive to the imperfection of the manufacturing process, e.g. inconsistent geometry and dimensions. The possible size effects, e.g. on the elastic modulus, yield stress and ultimate tensile stress, will be investigated in future studies.

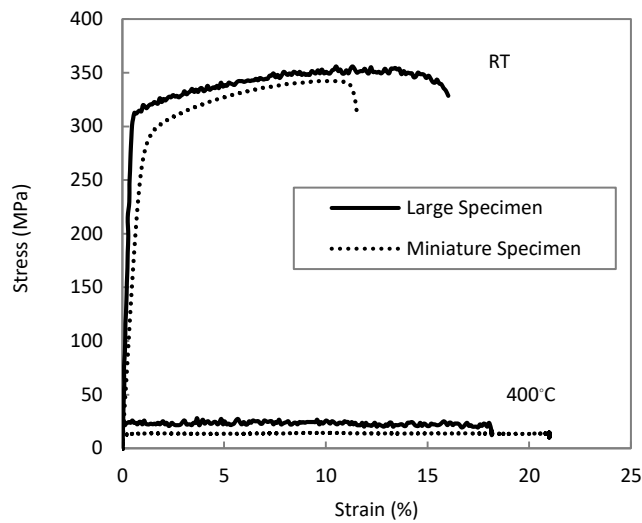


Figure 5.8. Miniature tensile test results on an aluminium alloy compared to the large specimen tensile test at different temperatures.

5.4.3. Miniature Creep Tests on a P91 Steel

5.4.3.1. Data Conversion

It should be noted that the measurements were taken outside the gauge length region (at the clamped ends) for the miniature specimen tests because it is not feasible to fit a gauge into the very limited space. Therefore, the measured displacement was slightly different from the actual displacement of the gauge length area. In order to validate and interpret the data obtained from the miniature specimen creep test, it is important to ensure that the data can be accurately converted to the corresponding uniaxial creep test results. A reference stress method was applied to determine the conversion relationships for stress and strain. The case of steady-state creep was applied for the demonstration.

Two conversion factors, η and β , were introduced to define the conversion relationships as follows:

$$\sigma_{ref} = \eta \sigma_{nom} \quad 5.18$$

$$\dot{\epsilon}_{ref} = \frac{\dot{\Delta}}{\beta L_0} \quad 5.19$$

where σ_{ref} , $\dot{\epsilon}_{ref}$, σ_{nom} , L_0 and $\dot{\Delta}$ are the reference stress, reference strain rate, nominal stress, the uniform section length, and the measured displacement rate, respectively. βL_0 is defined as the equivalent gauge length (EGL). The conversion factors are determined by carrying out a series of FE models of the miniature specimen creep test. A Norton power law is assumed to simulate the creep behaviours of the material, i.e. the steady state creep strain rate $\dot{\epsilon}^c$ is expressed as follows:

$$\dot{\epsilon}^c = A \sigma^n \quad 5.20$$

where A and n are material constants. The conversion factor β can be expressed as follows:

$$\beta = \frac{\dot{\Delta}}{A(\eta\sigma_{nom})^n L_0} \quad 5.21$$

The conversion factors were assumed to depend solely on the geometry, so they should be material-independent. A series of FE models of the miniature specimen creep tests were carried out with different material constants A and n . The parameter n , with the range of 4 to 12, was used for FE modelling. The corresponding values of A were calculated assuming a constant creep displacement/strain rate, i.e. $A_0\sigma^{n_0} = A_i\sigma^{n_i}$. The steady-state creep displacement-time curves obtained from the FE models are shown in Figure 5.9. The displacement rates, or strain rates, are very close to each other for different cases, indicating that the assumption of an approximately constant strain rate is valid. The minimum displacement rate, $\dot{\Delta}_{ss}$, was calculated for each curve. The objective was to find the value of η with which the value of $\alpha = \frac{\dot{\Delta}}{A(\eta\sigma_{nom})^n L_0}$ becomes independent of A and n . Following the FE modelling, $\log(\alpha)$ was calculated for a range of values of η (0.5 to 1.2) and the results are shown in Figure 5.10. It can be seen that $\log(\alpha)$ is nearly constant for $\eta = 0.984$, independent of n and A . Therefore, the conversion factor β can be calculated, i.e. $\beta = 1.205$.

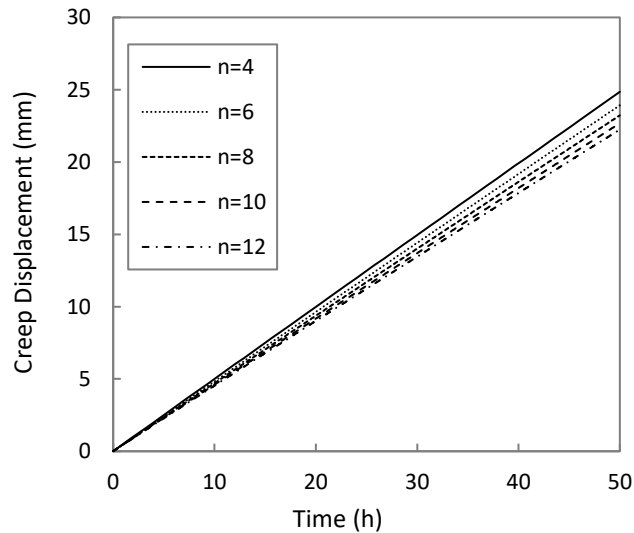


Figure 5.9. Steady-state creep displacement with variation of n and A .

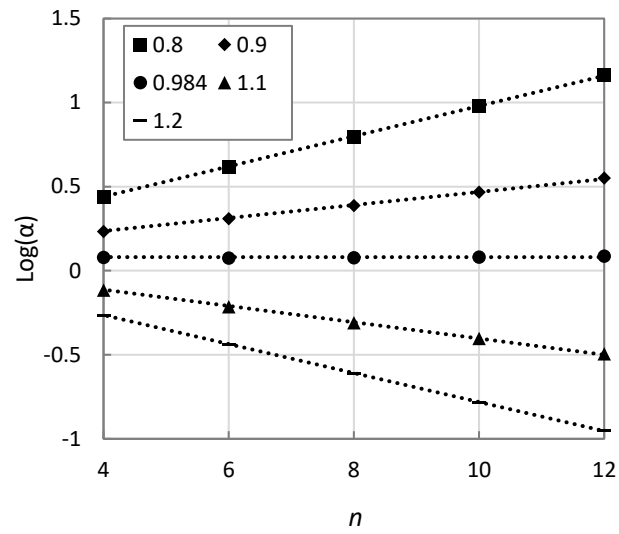


Figure 5.10. Variation of $\log(\alpha)$ with n (and the corresponding A).

5.4.3.2. Miniature Specimen Creep Testing Results of P91 Steel

Preliminary creep tests were carried out on P91 steel at 650°C. The specimen geometry and the experimental set-up were the same as that as shown in Figure 5.5. Constant forces were applied to the specimen to achieve the stress levels of 100MPa, 93MPa and 87MPa. Figure 5.11 shows the creep testing results obtained from the miniature specimen and the uniaxial creep test. The creep strain rates during the steady state agree well for the miniature specimen test and the uniaxial test at the stress levels of 87MPa. The miniature specimen tests show a higher strain rate and a shorter time to failure compared to the uniaxial tests at higher stress level such as 93MPa and 100MPa. These differences might be due to the local stress concentration resulting from machining, e.g. near the clamp at the gauge length ends. Also, the testing results of the miniature specimen could be affected by the amount of clamping force applied to it, e.g. the pre-existing stress in the material before the creep test. In addition, the differences might be due to surface oxidation (observed from the specimens after the tests) occurred during the tests. The influences of surface oxidation might be amplified by the small thickness of the specimen. Figure 5.12 shows the variation of the MSR and the time to failure versus stress level, plotted on logarithmic scales. The approximate linear trends indicate the general consistency of the miniature specimen creep tests at different loading levels.

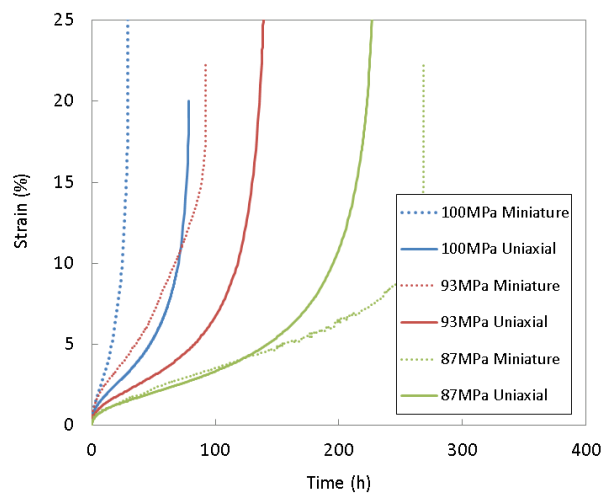
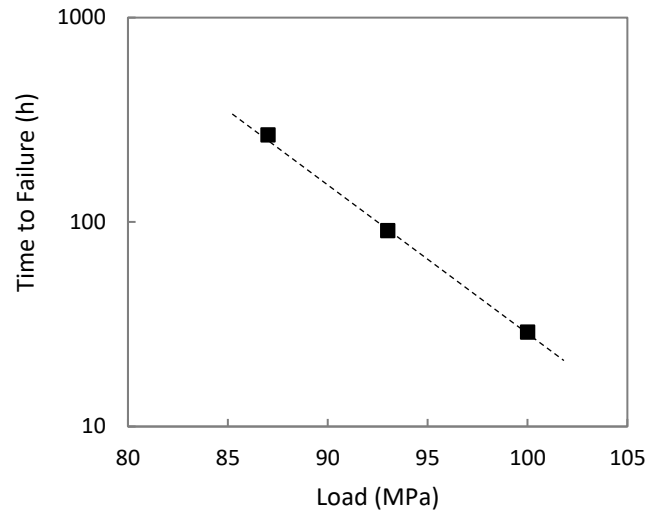
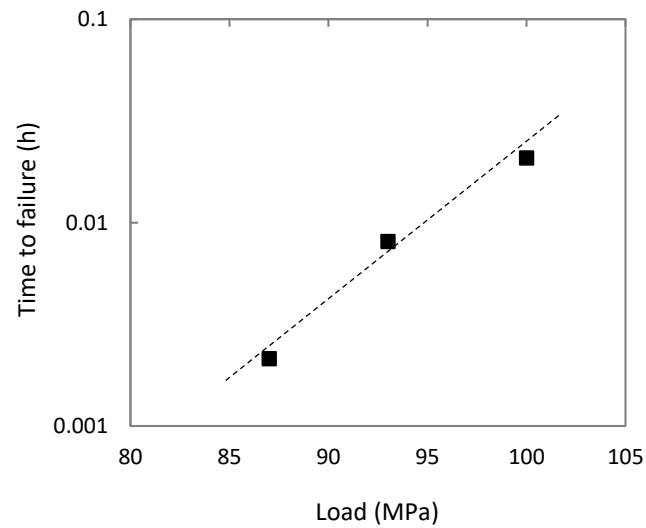


Figure 5.11. Miniature specimen and uniaxial creep tests on P91 steel at 650°C.



(a)



(b)

Figure 5.12. Variation of (a) the time to failure and (b) the MSR versus stress level, plotted on logarithmic scales.

5.5. Discussion

The methodology development of a miniature specimen testing technique and an inverse method to determine the creep properties of an unknown material from the compound creep behaviors of a two-material system has been presented. The forward analysis and the inverse method were demonstrated through a case study of a theoretical experiment. The semi-analytical computation and the FE modelling showed very good agreement. The optimized compound creep behaviors were close to the target results. It was verified by the single-material model computations that the optimized creep properties were close to the target material. The differences might be due to that the actual stress distribution in the two material layers was not evaluated by the inverse optimization approach, since the stress was not a measurable quantity in experiment. As a result, the stresses in the two material layers acted as the extra ‘variables’ during the optimization process, leading to some random deviations.

Single-material miniature tensile tests and creep tests were carried out on an aluminium alloy and P91 steel, respectively. The experimental results were compared to the results from the large specimen uniaxial tests on the same materials. The results of the miniature specimen tests were generally comparable to the uniaxial tests while some differences were found. Those differences could be a result of the possible local stress concentration due to machining, the random errors in the manual handling procedures (e.g. the clamping locations), the pre-existing strains and the surface oxidation. The testing technique might be improved by optimized specimen design for lower stress concentration factor, enhanced manufacturing quality and minimized influences from surface oxidation (e.g. carrying out the tests in a protective atmosphere or vacuum).

Chapter 6 - Characterisation of a Heat-Treated P91 Steel and a CoNiCrAlY Coating

As clarified by the two-material system study (Chapter 5), it is required to characterise the individual materials separately for the characterisation of a two-material/multi-material coating system. In this study, the two-material system consists of a P91 steel as the substrate and a CoNiCrAlY coating. This chapter presents the characterisation of a heat-treated P91 steel and a CoNiCrAlY coating using the miniature specimen testing techniques. The same heat-treatment as typically received by the thermal barrier coatings was carried out for all specimens. This is the first part of the preparational experimental work for the characterisation of the two-material coated system.

6.1. Miniature Specimen Tensile Tests on a Heat-Treated P91 Steel

6.1.1. Testing Method

Specimen and testing rig

P91 steel specimens were made in the same geometry as shown in 5.4. The tests were performed on the same Tinius Olsen H5KS single column materials testing machine (detailed description in 5.4) at the temperature of 500 and 600°C.

Heat treatment

Same heat treatment as initially received by the thermal barrier coatings was applied to the P91 specimens to replicate the typical effects on the substrate material. Vacuum heat treatment was carried out on the P91 specimens at 1100°C for two hours in an Elite Thermal Systems TVH12 vacuum tube furnace held at approximately 10⁻⁹ bar followed by furnace cooling to room temperature over a period of 6 hours.

6.1.2. Testing Results

The results of the miniature tensile tests on the heat-treated P91 steel at 500 and 600°C were shown in Figure 6.1 with the comparison to a uniaxial tensile test at 600°C on the P91 steel without heat treatment. At the same temperature of 600°C, the miniature heat-treated specimens show much lower yield and ultimate stress and higher ductility compared to the uniaxial specimen without heat treatment. Generally, it indicates that the heat treatment had significant effects on the material behaviour. On the other hand, it was shown that the tensile behaviours of the heat-treated specimens were highly dependent on the temperature: the material shows a much higher tensile and ultimate stress and much lower ductility at 500°C than at 600°C.

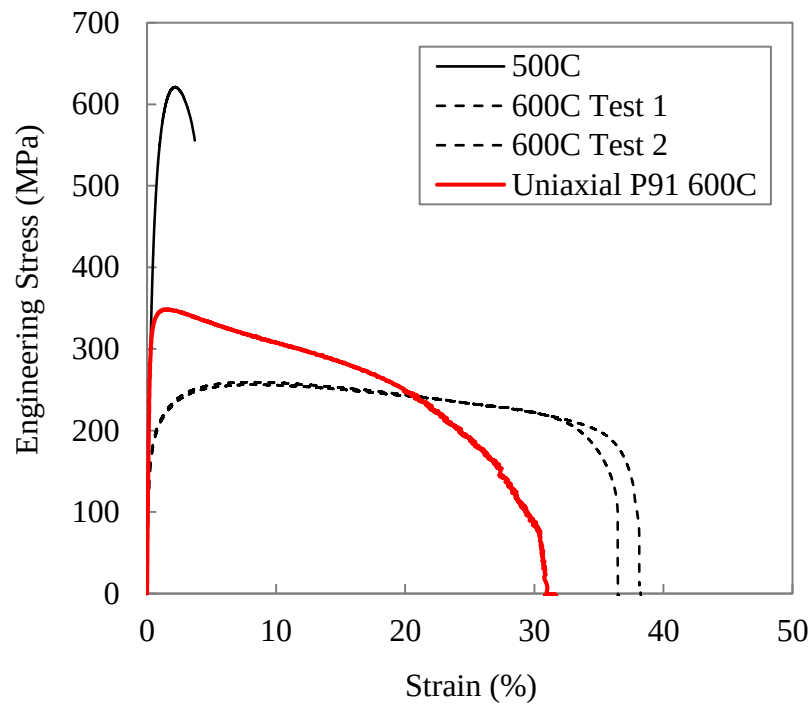


Figure 6.1. Miniature tensile tests on the heat-treated P91 specimens at 500 and 600°C compared to the uniaxial tensile test on a P91 specimen without heat treatment.

6.2. Miniature Specimen Creep Tests on a Heat-Treated P91 Steel

Miniature creep tests were carried out at 650°C for the heat-treated P91 steel specimens at the tensile stress levels of 65, 70, 85 and 100 MPa.

6.2.1. Testing Results

The miniature creep tests results for the heat-treated P91 steel at 650°C are shown in Figure 6.2. The stress-strain curves were converted from the load-displacement curves obtained from the tests (the data conversion is introduced in 5.4.3.1).

Figure 6.3 shows the logarithmic scale plots of (a) time to failure and (b) minimum strain rate (MSR) in the function of stress level for the miniature creep tests. It can be found that the time to failure and the MSR are generally in linear functions of stress level, indicating the good consistency and reliability of the testing results.

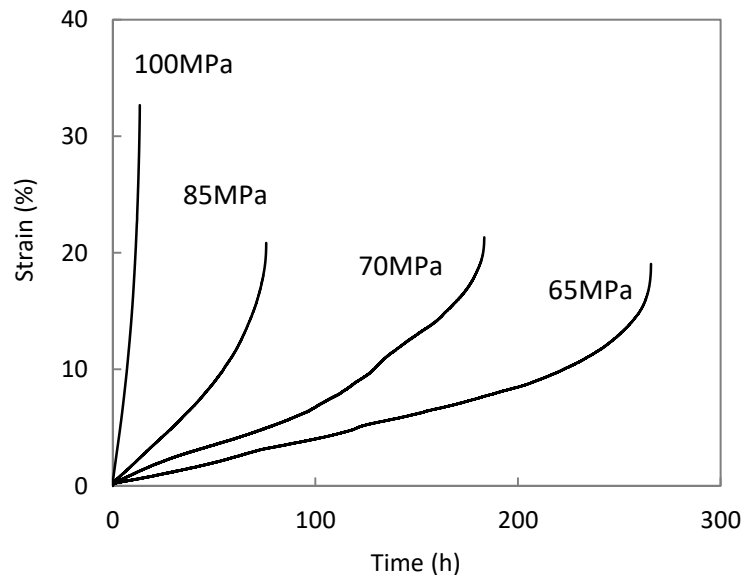
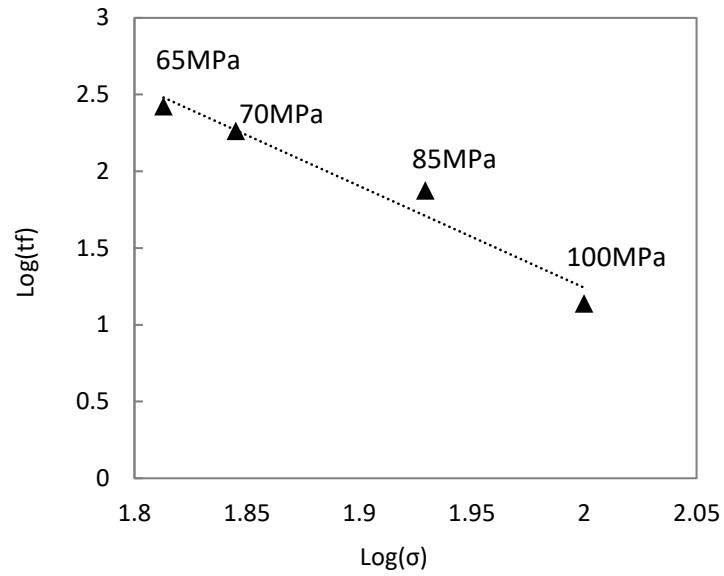
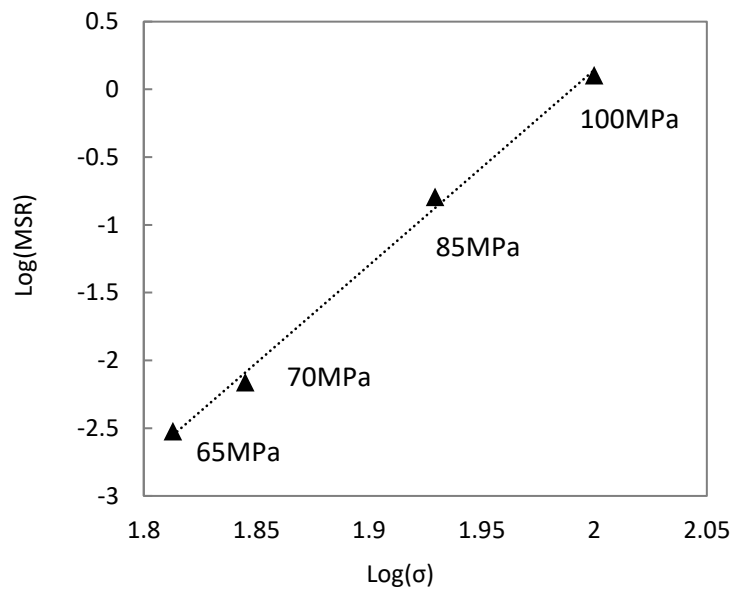


Figure 6.2. Miniature creep tests on the heat-treated P91 steel at the stress levels of 65, 70, 85 and 100 MPa at 650°C.



(a)



(b)

Figure 6.3. Logarithmic scale plots of (a) time to failure and (b) minimum strain rate in the function of stress level for the miniature creep tests on the heat-treated P91 steel at 650°C.

6.3. Discussion

Due to the limited amount of available material, only a few miniature tensile tests were carried out for the heat-treated P91 steel. The repeated tests at 600°C show very similar results, indicating a high reliability. This series of experiments could benefit from more repeated tests at different temperatures. For example, it is worth investigating the variation of the tensile behaviour of this material from RT to 750°C.

Although the results of the miniature creep tests showed generally good consistency, it should be noted that repeated tests were only carried out for the stress level of 100 MPa. Only one test was performed for the other stress levels, meaning it is possible that the results were subjected to some random errors. For example, it can be found in Figure 6.2 that the strain rate of 70 MPa test experienced some unexpected variation between the steady-state creep and the failure stage, indicating possible experimental errors. Therefore, more repeated tests could make this series of testing results more convincing. For building a more comprehensive database for this material, miniature creep tests could be performed at other stress levels and temperatures of interest.

Because the main purpose of carrying out the miniature tensile and creep tests for this heat-treated P91 steel was to characterise the single material behaviour before investigating the two-material system, it is important to compare the tensile and creep behaviours of the heat-treated P91 steel and the CoNiCrAlY coating at different temperatures and stress levels and then choose appropriate testing conditions for the future tests on the two-material coated specimens.

6.4 - Characterisation of a CoNiCrAlY Coating

It is required to characterise the CoNiCrAlY coating as a single material before the characterization of the two-material coated system can be carried out. This chapter presents the investigations carried out for the CoNiCrAlY coating using small punch tensile, miniature tensile and indentation testing methods. This is the second part of the preparational experimental work for the characterisation of the two-material coated system.

6.4.1. Materials, HVOF Thermal Spraying and Heat Treatment

The HVOF spraying procedure is detailed in the reference [129]. The mild steel substrate was not grit blasted to aid debonding after spraying. The coating was sprayed to a thickness of approximately 600 μm and then debonded from the mild steel by bending around a mandrel to produce free standing coatings. Vacuum heat treatment was carried out on free-standing samples at 1100°C for two hours in an Elite Thermal Systems TVH12 vacuum tube furnace held at approximately 10⁻⁹ bar followed by furnace cooling to room temperature over a period of 6 hours. This treatment was applied to approximately replicate the initial heat treatment given to bond coats during the manufacture of thermal barrier coatings. This type of heat treatment has been shown to reduce the porosity commonly present within sprayed coatings and allow the precipitation of secondary phases [129, 130]. Specimens were cut from the heat-treated coating by electro-discharge machining. They were ground down from the as-deposited thickness to a final thickness (0.4 mm for SPT tests and 0.5 mm for miniature tensile and indentation tests) on 1200 grade silicon carbide paper. The final thickness was controlled to within ± 5 μm as measured by a digital micrometer and both surfaces had the same finely ground surface finish.

6.4.2. Small Punch Tensile Tests on a CoNiCrAlY Coating

6.4.2.1. Small Punch Tensile Testing Method

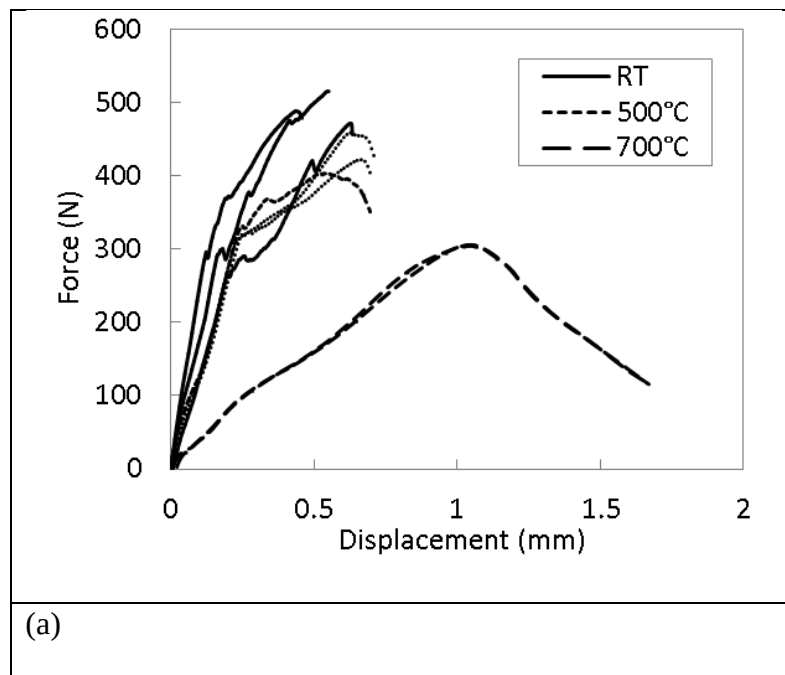
The SPT tests were performed on a Tinius Olsen H5KS single column materials testing machine as displacement-controlled at the displacement rate of $1\mu\text{m/s}$ at RT, 500°C and 700°C . This testing rig is introduced in Chapter 3.

6.4.2.2. Results of Small Punch Tensile Tests

SPT tests were carried out on a CoNiCrAlY coating material. The force-displacement curves obtained at RT, 500°C and 700°C are shown in Figure 6.4 (a). The consistency of the repeated tests at different temperatures is dissimilar. At RT, the scattering of the results of the repeated tests is quite significant, even for the initial part of the curve. At 500°C , the initial parts of the curves are very similar, indicating the similar elastic bending deformation while the scattering presents after the early load drop, which indicates the crack initiation. At 700°C , the repeated tests show almost identical results through the whole test until fracture. It should also be noted that there is no early load drop at 700°C , indicating that there is no early crack initiation. These differences between the results at 700°C and the lower temperatures suggest that the material behaviour varies significantly with temperature. Figure 6.4 (b) shows the total external work done during the SPT tests (calculated as the average integrations of the load-displacement curves) as a function of temperature. This quantity can be used as an indicator of the toughness of the material, conceptually similar to the tensile toughness obtained from a conventional uniaxial tensile test. The increase in the total external work is relatively small from RT to 500°C while it goes up dramatically from 500 to 700°C . This phenomenon indicates the ductile to brittle transition of the material between 500 and 700°C . Similar transition is reported by Jackson et al [2], where the ductile-to-brittle transition temperature (DBTT) is found to be between $500\text{--}700^\circ\text{C}$. Due to the tendency

of showing scattering results at lower temperatures (e.g. lower than the DBTT), SPT testing might be a less effective mechanical testing technique at those temperatures for this coating material.

The fracture surfaces of the failed SPT test specimens were examined using SEM. The fracture at 500°C was characterised by a star-like cracking pattern in the centre of the specimen known to be associated with brittle failure (shown in Figure 6.5 (a)). At 700°C, fracture was characterised by circumferential cracking, as seen in Figure 6.5 (b), which is known to be associated with ductile failure. These findings indicated that the DBTT of HVOF coatings manufactured from Praxair CO-210-24 was between 500-700°C.



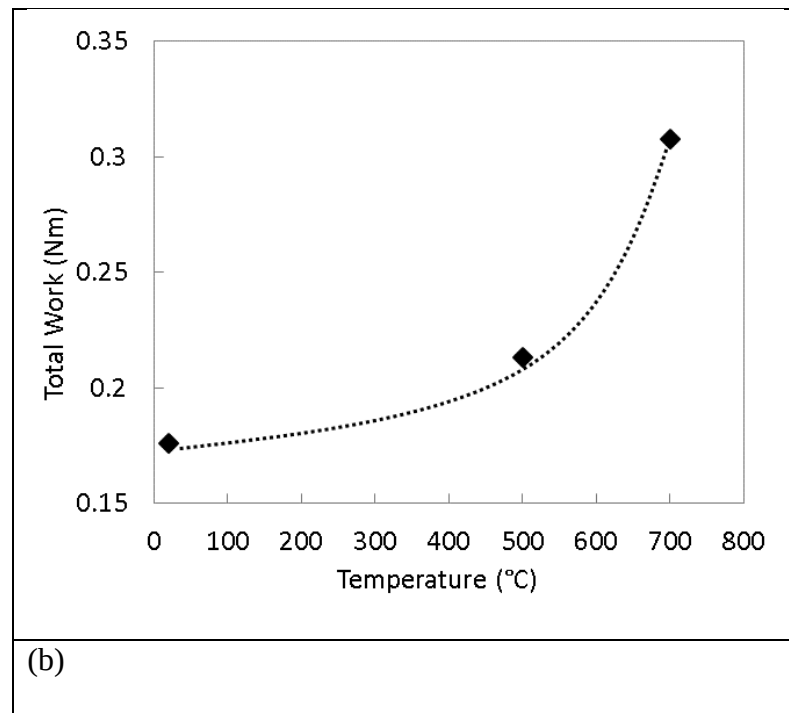
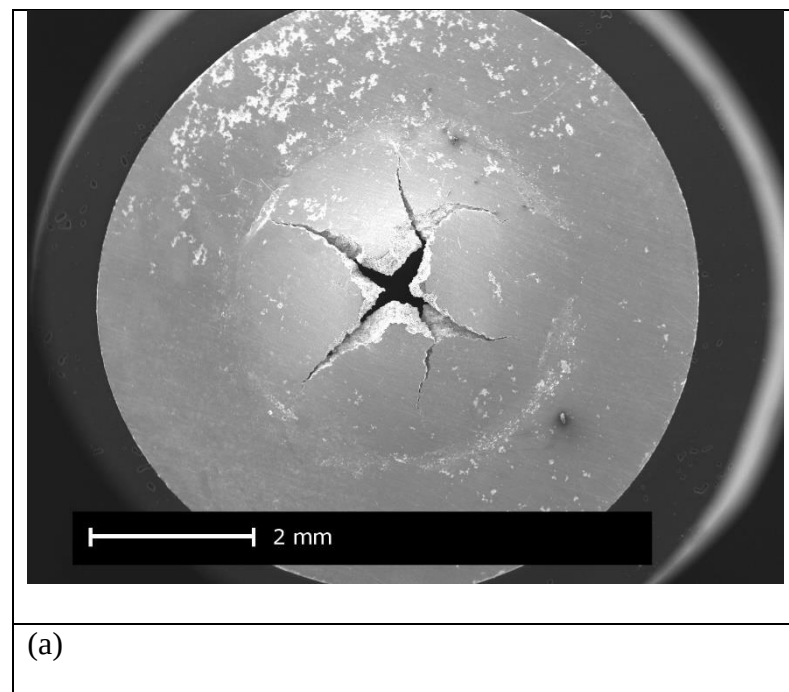


Figure 6.4. (a) Load-displacement curves and (b) total external work as a function of temperature (b) for SPT tests between RT and 700°C.



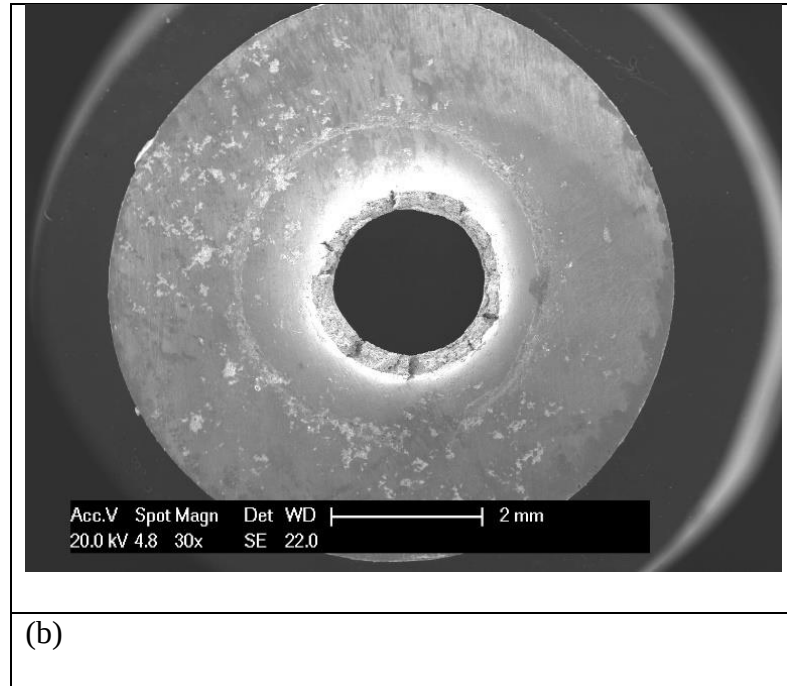


Figure 6.5. SEM images of freestanding CO-210-24 HVOF coatings following SPT testing at (a) 500°C and (b) 700°C.

6.4.3. Miniature Tensile Testing on a CoNiCrAlY Coating

Miniature tensile tests were carried out on the same CoNiCrAlY coating to provide alternative source of data for comparison and verification as well as the preparation for the future work of two-material coated system characterisation.

6.4.3.1. Miniature Tensile Testing Method

The miniature tensile tests were carried out using the same Tinius Olsen H5KS single column materials testing machine. The geometry and dimensions of the miniature testing specimen are Figure 5.7. Figure 6.6 (b) shows the loading assembly and the miniature specimen clamped and mounted to the machine. The loading assembly parts were made of a high temperature nickel-based superalloy, Nimonic 115. The specimen was

clamped at both ends by the specially manufactured clamps with the machined surface features to strengthen the grip. The displacement was measured by two LDVT's connected to both sides of the loading parts of the machine. The mounted specimen was kept inside a furnace for heating, except for the RT test. Tensile tests were carried out at RT, 500°C, 600°C and 700°C. Multiple tests were carried out at each temperature to verify the consistency of the testing method (some testing results were excluded due to the sign of sliding or cracks induced by clamping). The loading process was controlled at the constant displacement rate of 0.006mm/s (to achieve a strain rate of 0.001/s, approximately). It had been verified by preliminary tests that the results of the tensile tests had very low sensitivity to the loading rate between 0.0006mm/s and 0.06mm/s. The torque applied to the clamps was adjusted to 0.5Nm to avoid inducing any crack in the specimen while providing sufficient clamping force to avoid slipping.

6.4.3.2. Results of the Miniature Tensile Tests on a CoNiCrAlY Coating

6.4.3.2.1. Mechanical Behaviour

The true stress-strain curves, estimated from the loading force, cross-section area and loading direction displacement, obtained from the miniature tensile tests are shown in Figure 6.6. It should be noted that the displacement measurements were taken at the clamped ends (outside the gauge length region) because it is very difficult to fit a gauge into the very limited space. Therefore, the measured displacement was different from the actual displacement of the gauge length area. An equivalent gauge length (EGL), $L_{EGL} = \beta L_o$ (β is a constant decided by the geometry of the specimen and the loading conditions, L_o is the original gauge length), was estimated based on FE analyses, where $\beta \approx 1.2$. The repeatability of the multiple tests carried out at each temperature, RT, 500°C, 600°C and 700°C, was very good, indicating the good consistency of this testing technique. The ultimate tensile stress (UTS) decreases as temperature increasing from RT to 700°C. At and

below 600°C, there were only elastic deformation phase and strain-hardening phase, but no clear sign of material softening before failure while there was a clear phase of material softening before reaching failure at 700°C. The observed material softening at 700°C might due to the recrystallisation of the material at elevated temperature, i.e. higher than the recrystallisation temperature of this coating material, which was suggested to be between 600 and 700°C. A notable phenomenon found in the SPT is the sudden load drop in early stage, which indicates the formation of early cracking, at and below 500°C (shown in Figure 6.4 (a)). The formation of early cracking in the SPT tests of the coating material at low temperatures is mainly due to the deformation mechanisms involving bending and coning and the low ductility of the material. No sign of early cracking was found in the miniature tensile tests, indicating that it is an effective mechanical testing technique for this coating material at a wide range of temperature, despite the ductile-to-brittle transition of the material.

The estimated mechanical properties of this coating material are presented in Table 6.1. Generally, the elastic modulus and the yield stress decrease with increasing temperature. Regarding the failure strain, the increase from RT to 600°C is relatively small while it increases significantly from 600°C to 700°C. These transitions are shown more clearly in Figure 6.7. The intersection of the UTS and failure strain curves, shown in Figure 6.7 (a), indicates that the DBTT to be near 650°C. Figure 6.7 (b) shows the tensile toughness, calculated as the area under the stress-strain curves, and the estimated elastic modulus as function of temperature. It is clear that the tensile toughness of this coating material remained relatively constant between RT and 600°C while it increased by nearly 300% when temperature is increased to 700°C. Again, this is another indication of ductile-to-brittle transition. Similarly, the temperature effect on the elastic modulus was not significant at temperature between RT and 500°C while the elastic modulus dropped by over 60% when temperature was increased from 500°C to 700°C. Those findings were in very good agreements with the results from the corresponding SPT tests presents.

Regarding the deformation of the failed specimens, there was no visible necking, which makes the engineering stress-strain deviate significantly from the true stress-strain, in all the miniature tensile tests even until the failures of the specimens at all temperatures. These facts indicate that the true stress-strain curves obtained from the miniature tensile tests are relatively complete and reliable. Compared to small punch testing technique, this miniature specimen testing method has the advantage that the interpretation of the experimental results is relatively straightforward, i.e. the stress-strain relationships can be easily obtained from the force-displacement data.

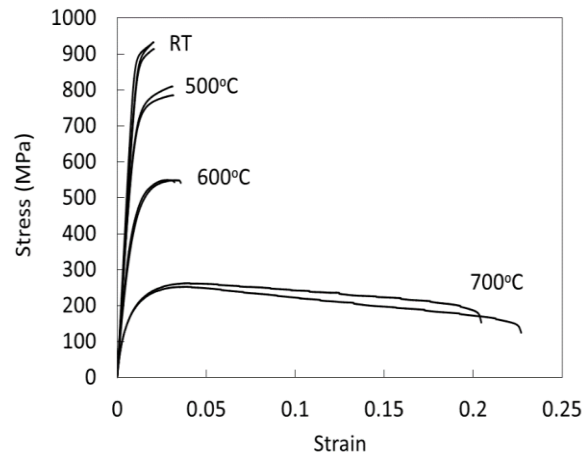


Figure 6.6. Stress-strain curves obtained from the miniature tensile tests on the CoNiCrAlY coating at temperatures from RT to 700°C.

Table 6.1 Mechanical properties of the CoNiCrAlY coating estimated from the stress-strain curves.

	RT	500°C	600°C	700°C
E (GPa)	83.6	75.6	47.5	40.0
σ_y (MPa)	850	750	450	100

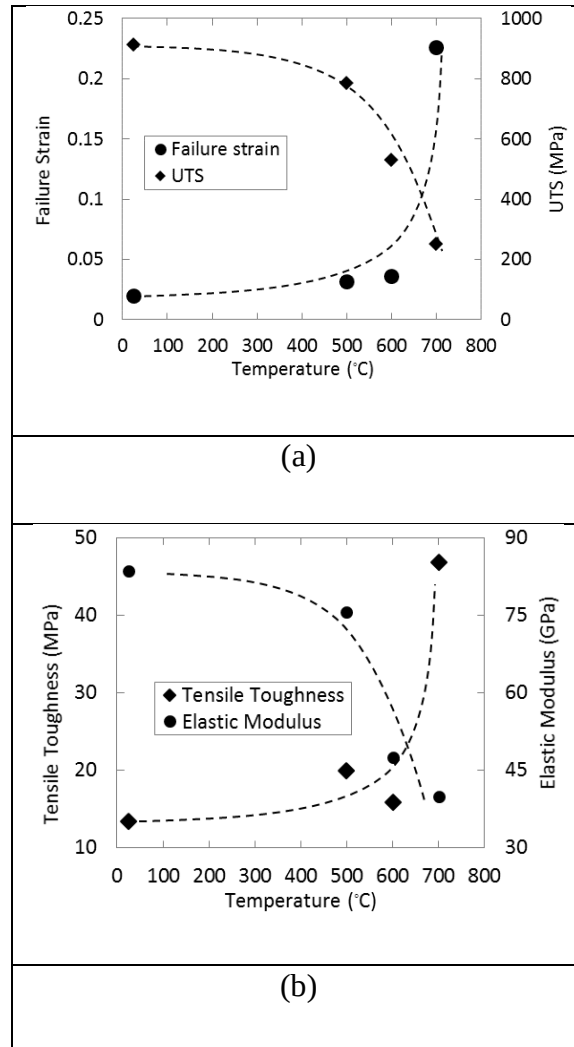


Figure 6.7. (a) The failure strain and UTS and (b) the tensile toughness and elastic modulus as functions of temperature for miniature specimen tensile tests between RT and 700°C.

6.4.3.2.2. Microscopic Fracture Behaviour

The failed miniature tensile testing specimens were mounted, ground and polished to a 1 μm diamond finish. Scanning electron microscopy (SEM) was carried out using a FEI XL30 scanning electron microscope equipped with an Oxford Instrument Link ISIS-3000 energy dispersive X-ray analysis (EDX)

detector. Micrographs of the tested miniature tensile test specimens were obtained using backscattered electrons (BSE).

Secondary electron (SE) microscopy images of the fracture surfaces of the miniature tensile tests specimens tested at 500°C and 700°C are shown in Figure 6.8. At 500°C (Figure 6.8 (a-b)) there are noticeable concave or convex features, which are relatively smooth and approximately up to 30 to 40 μm in size. These features are identified as powder particles which indicates brittle failure occurred along the powder particle boundaries. In contrast, no such features are identified on the fracture surface of the specimen tested at 700°C, as shown in Figure 6.8 (c-d). The 700°C fracture surface consists of small, rounded surface features that indicate ductile failure.

The fracture surfaces of the miniature tensile testing specimens for 500°C and 700°C demonstrate a clear change in fracture behaviour between 500-700°C. This verifies the ductile to brittle transition indicated by the stress-strain curves (Figure 6.4). Generally, brittle fracture of metallic alloys includes both cleavage and intergranular fracture while ductile failure is developed from cavitation or plastic instability [141]. The results from both the SPT and miniature tensile tests indicate that the energy required for crack propagation in the β -phase is lower than the threshold energy required for plastic flow in the γ -phase at low temperatures, such as RT and 500°C, while the energy required for plastic flow decreases as temperature increases (reflected by the decreasing yield stress) and it becomes lower than the energy required for brittle cleavage and intergranular fracture when the temperature is higher than the DBTT [127].

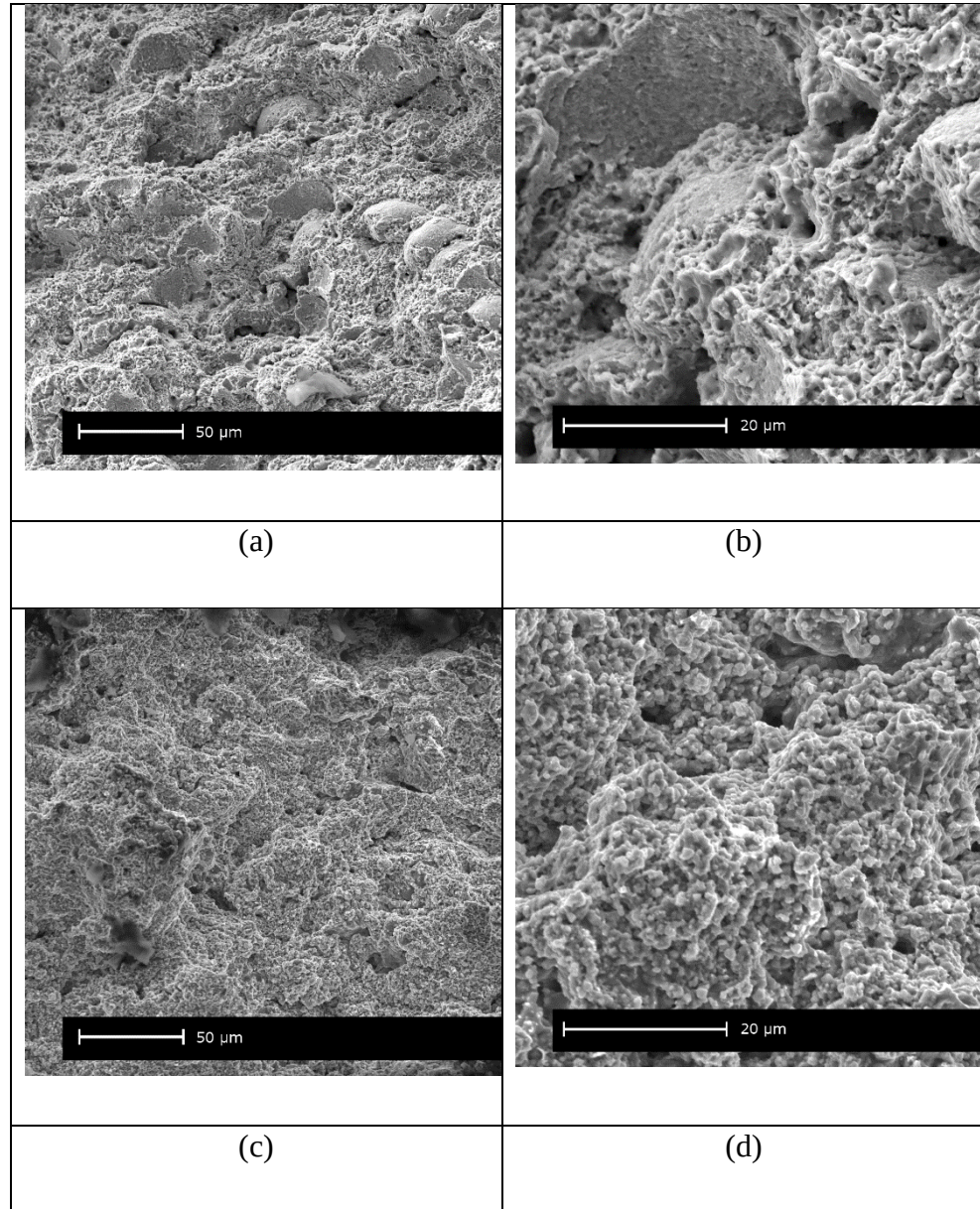


Figure 6.8. Back-scatter electron images of the coating fracture surface after miniature specimen tensile tests at (a-b) 500°C and (c-d) 700°C.

6.4.3.3. Inverse Approach

6.4.3.3.1. Material Constitutive Model

To characterise the mechanical behaviours of the coating material, an elastic-plastic material model was employed under the uniaxial conditions. A simple Hooke's law was applied for the elastic deformation, i.e. $\sigma = E\varepsilon$, where σ is

the stress, ε is the strain and E is the elastic modulus. For the plastic deformation, the Johnson-Cook constitutive model [142] was used to describe the strain hardening behaviour, considering of the effects of temperature. The original Johnson-Cook model is expressed as follows:

$$\sigma = (A + B\varepsilon^n)(1 + C \ln \dot{\varepsilon}^*)(1 - T^{*m}) \quad 6.1$$

$$\dot{\varepsilon}^* = \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \quad 6.2$$

$$T^* = \frac{T - T_r}{T_m - T_r} \quad 6.3$$

where A is the yield stress at the selected reference temperature, T_r , B is the strain hardening coefficient, n is the strain hardening exponent, C is the material constant for strain rate hardening, m is the material constant for thermal softening exponent, $\dot{\varepsilon}$ is the applied strain rate, $\dot{\varepsilon}_0$ is the reference strain rate, T is the applied temperature and T_m is the melting temperature of the material (temperatures are in unit of K). The melting temperature of the CoNiCrAlY coating material is about 1350°C or 1623K (the powder used to prepare the coating was obtained from Praxair, powder code CO-210-24).

In this study, the strain rate hardening was neglected because the loading rate was kept constant for all tests. The Johnson-Cook constitutive equation, Eq (1), was simplified as follows:

$$\sigma = (A + B\varepsilon^n)(1 - T^{*m}) \quad 6.4$$

It should be noted that the temperature effects on elastic properties are not included by the Johnson-Cook constitutive model.

6.4.3.3.2. Inverse Method Procedures

An inverse method was developed to extract the Johnson-Cook constitutive properties from the miniature tensile tests on the CoNiCrAlY coating. Figure

6.9 shows the flowchart of the inverse method. The experimental data obtained from the miniature tensile tests are supplied as the target values in the form of load (P)-displacement (Δ) curves. An initial guess of the Johnson-Cook constitutive properties is provided to start the iterative algorithm. This initial guess can be obtained by performing a numerical function fitting based on the Johnson-Cook constitutive equations and the stress-strain data obtained from a similar material. A nonlinear optimisation function based on the optimisation toolbox in the commercial code MATLAB R2015a is employed, cooperated with a FE model of the miniature specimen, to obtain FE predictions for the corresponding miniature tensile tests and calculate the total differences compared to the experimental data. The FE results were extracted using a Python script constructed for the FE model built using the commercial code ABAQUS 6.14-1. An objective function was constructed to obtain the minimum value of the total differences between FE predictions and experimental results. The inverse algorithm stops automatically once the objective function value is minimised to be smaller than a user-defined threshold value. It should be noted that the constitutive constants (A , B , n and m) are in very different orders. These constants need to be scaled similar orders, based on a selected minimum step size, to ensure that the optimisation algorithm works efficiently on all constitutive constants. The ratio of the minimum step size to the magnitude of the scaled constant is crucial, e.g. if the step size is too small then the optimisation might not progress efficiently or not able to converge close to the target conversely. A selected number of data points were taken from each part of the load-displacement curve, i.e. more data points are taken from the curved part than the linear part (the Johnson-Cook constitutive model describes the plastic behaviours). Other constitutive material models, e.g. creep models and damage models and so on, can also be conveniently adapted to this inverse approach with suitable adjustments.

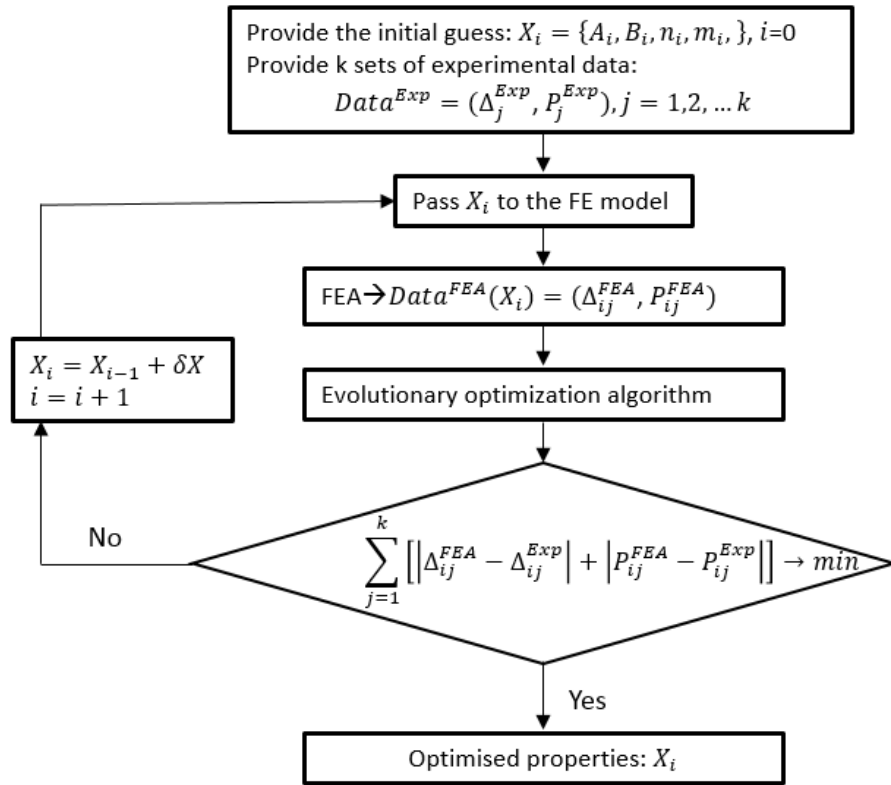


Figure 6.9. The flowchart of the inverse method.

6.4.3.3.3. Application of the Inverse Approach

The inverse approach was applied for the experimental load-displacement curves obtained from the miniature tensile tests. It should be noted that the shape of the experimental curves from 700°C were very dissimilar to those from RT, 500°C and 600°C, e.g. the ductility was significantly higher. The Johnson-Cook constitutive model is more suitable for relatively small strain. Also, damage behaviour was not included by this constitutive model. Therefore, the Johnson-Cook constitutive model was applied to the inverse method for the experimental data obtained at RT and 500°C. According to Equ 6.3-6.4, it is clear that the constitutive properties are dependent on the choice of reference temperature. In this case, RT (293K) was selected as the reference temperature, i.e. the constitutive constant A was the yield stress at RT.

The evolution of the optimisation function is shown in Figure 6.10. The function value is the measure of the total differences between the predicted load-displacement curves extracted from FE modelling and the experimental curves. It should be noted that the Johnson-Cook constitutive model does not take account of the temperature effects on the elastic modulus. The elastic modulus was taken as a constant for the optimisation since it is relatively easy to extract the elastic modulus from the experimental results, but certain amount of error might still exist. The comparison between the target curves, initial guess and the optimised curves is shown in Figure 6.11. The optimised curves are much closer to the experimental curves than the initial guess. The initial guess and the optimised Johnson-Cook constitutive properties are summarised in Table 6.2. Generally, the inverse method was proved to be capable of extracting the Johnson-Cook constitutive properties of the CoNiCrAlY coating using the miniature tensile testing results obtained at different temperatures with very good agreement to the experimental data.

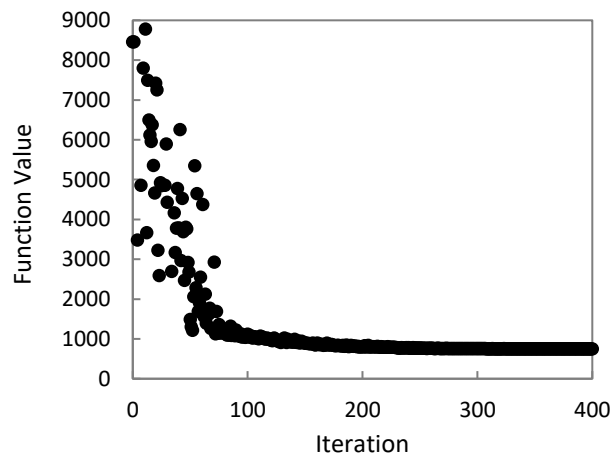


Figure 6.10. Function evolution of the inverse approach.

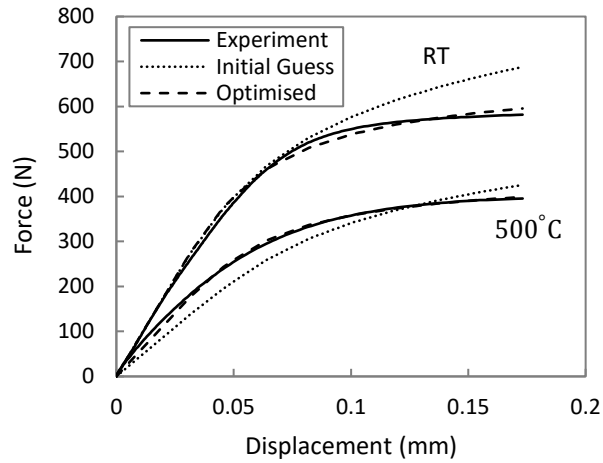


Figure 6.11. Load-displacement curves: experiment, initial guess and optimised.

Table 6.2. The initial guess and the optimised Johnson-Cook constitutive properties (RT and 500°C).

	A	B	n	m
Initial guess	700MPa	1000	0.5	0.5
Optimised	765.16MPa	607.18	0.3	1.715

6.4.3.4. Finite Element Modelling of SPT Test

FE modelling of SPT tests of the CoNiCrAlY coating was carried out using the commercial code ABAQUS with the stress-strain behaviour acquired from the miniature specimen tensile tests. The geometry of the FE model was shown in Figure 6.12 with the identical dimensions for the SPT tests described previously. The punch head, the holder, and the die were modelled as axisymmetric analytical rigid bodies. The specimen was modelled as axisymmetric deformable body and meshed using the 4-node bilinear

axisymmetric stress quadrilateral with reduced integration elements (CAX4R) and the 3-node linear axisymmetric stress triangle elements (CAX3) for the transition zones. Refined mesh with the elements size 0.02 mm was used for the un-supported region of the specimen due to the expected high stress and large deformation.

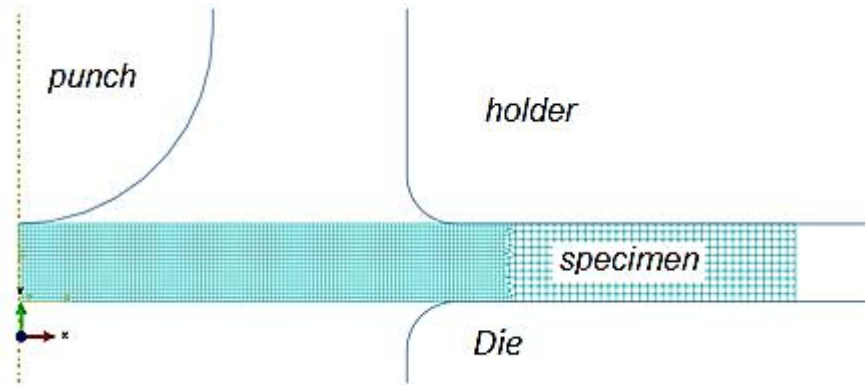


Figure 6.12. The FE model of SPT Testing.

A failure mechanism was required to characterise the damage evolution of the SPT testing specimen. Figure 6.13 shows a typical uniaxial stress-strain curve of a material with (a-b-c-d) and without (a-b-c-d') damage evolution. The fracture energy, i.e. the tensile toughness shown in Figure 6.7 (b), obtained by integrating the stress with respect to the strain from the stress-strain curves shown in Figure 6.6, was used for the damage evolution simulation with ABAQUS/Explicit. When the energy dissipated in an element reaches the fracture energy value, the element is regarded as failed and removed from the model. As the tensile test proceeds, the global damage evolution is reflected by the increasing number of failed elements removed from the FE model.

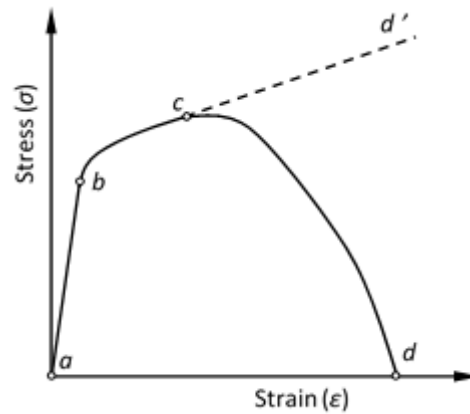


Figure 6.13. A typical uniaxial stress-strain curve of a material with and without damage evolution.

The results of the FE modelling of SPT tests with and without damage evolution compared to the average experimental results are shown in Figure 6.14. It should be noted that the Johnson-Cook properties presented in Table 6.2 were used for the FE modelling for RT and 500°C while the estimated stress-strain data was used for 700°C because the material became ductile at 700°C and could not be characterised using the Johnson-Cook model together with RT and 500°C. Generally, at RT and 500°C the maximum load tends to be overestimated when damage evolution is not included while it is predicted closer with damage evolution. More importantly, including damage evolution enables this FE model to predict the early load drops due to cracking found in the SPT tests at lower temperatures as the RT and 500°C curves show.

The damage sites on the SPT specimen are depicted by the removal of the failed elements from the FE model. The comparison between the experimentally observed failure site for a 700°C SPT testing specimen and the corresponding FE modelling contour is shown in Figure 6.15. The FE model was able to predict the failure site to be near the contact boundary of the specimen and the punch head and also predict the circular geometry of the fracture pattern. It should be noted that the FE model was not able to predict

the star-shaped brittle failure pattern observed at lower temperatures (e.g. Figure 6.5 (a)) because the anisotropy of the material was not considered.

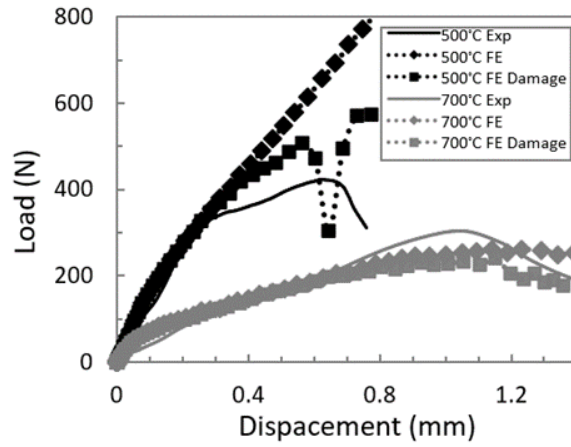


Figure 6.14. The Load-Displacement curves of experimental data and FE model results on Small Punch Tensile Testing processing

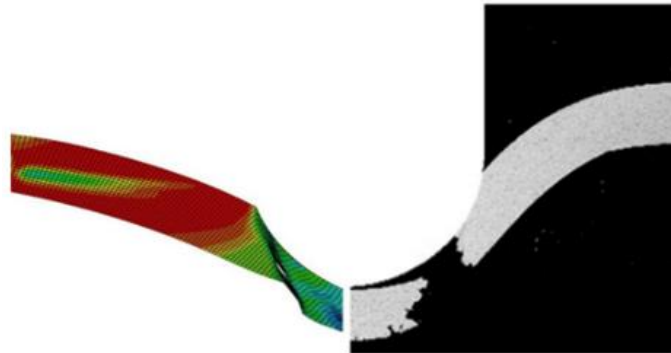


Figure 6.15. Comparison between the damage sites observed for a 700°C SPT test and the corresponding FE modelling

6.4.3.5. Discussions and Conclusions

SPT tests were performed on a CoNiCrAlY coating at RT and high temperature (up to 700°C). Good agreement was achieved regarding the DBTT of the material compared to a similar study [127]. The repeatability of

the SPT tests was very good at 700°C while clear scattering of the results was found at lower temperatures due to the early cracking. The consistency and repeatability of the SPT testing at lower temperatures might also be compromised by the local heterogeneity of the specimen, e.g. the random presence of porosity, local surface roughness variation and specimen thickness variation, which might have more significant effects on the testing results under the multiaxial stress condition than under the uniaxial stress condition. It was indicated by the results that SPT testing is more reliable as a mechanical testing technique for ductile materials, but less effective for brittle materials.

Miniature tensile tests were carried out at RT and up to 700°C on the same CoNiCrAlY coating used for the SPT tests. Compared to the SPT test, this miniature tensile testing method is easier to interpret, i.e. no complex data conversion method is required since the deformation mechanisms in the uniform section are similar to those of the conventional uniaxial testing specimens. It also eliminates the early cracking found in small punch tensile tests at temperatures lower than the DBTT, which was studied via the load-displacement curves and the SEM images of the fracture surfaces, found to be very close to the one found during the SPT tests. The transition of the material failure mechanism from brittle cleavage and intergranular fracture to ductile plastic flow is mainly governed by the threshold energy required by the different fracture mechanisms at different temperatures. The repeatability of the tests was excellent at all temperatures. It can be concluded that this novel miniature specimen testing method showed great potential to be a universally effective mechanical testing technique for both brittle and ductile materials in a wide range of temperatures.

An inverse approach was developed based on the Johnson-Cook constitutive model and applied to characterise the temperature-dependent mechanical behaviour of the coating material using the miniature tensile testing results. The inverse method was proved to be capable of accurately determining the constitutive material properties from the experimental data.

FE modelling of SPT test was carried out using the constitutive properties obtained by the inverse method and comparable results were obtained compared to the corresponding experimental results of SPT tests. Damage evolution was modelled based on the fracture energy obtained from the miniature specimen tensile tests. The model was able to predict the early cracking found in the low temperature SPT tests (i.e. RT and 500°C) as well as the circular geometry of the fracture surface observed at high temperature, i.e. 700°C. The star-shaped fracture pattern observed at low temperatures could not be predicted by the FE model since the material was assumed to be isotropic.

6.4.4. Indentation Testing on a CoNiCrAlY Coating

Nano-indentation tests were carried out on the same CoNiCrAlY coating to provide additional source of data for mechanical characterisation of this material as well as for verification.

6.4.4.1. The Critical Relative Indentation Depth of a Two-Material Coated System- A Numerical Study

6.4.4.1.1. The Critical Relative Indentation Depth

The critical relative indentation depth (CRID) is the ratio of the indentation depth to the coating thickness, beyond which the influence from the substrate material is not negligible. If the indentation depth is limited within the CRID, the plastic deformation will be confined within the coating and the substrate effects can be neglected for data interpretation with confidence. A rule-of-thumb suggests that the CRID is about 10% [143], but other studies [144-147] indicate that this value is not generally appropriate for different material combinations. Numerical and experimental work concerning the CRID has been carried out by other researchers, e.g. [145, 148-150]. A method has been

proposed to estimate the CRID of a coated system consisting of carbon-based film and silicon substrate [149]. It has been pointed out by early studies that the CRID is dependent on the indenter geometry, yield strength and elastic modulus ratios between the substrate and the coating, e.g. [150, 151]. A qualitative relationship between the CRID and the coating/substrate yield strength ratio and the coating/substrate Young's modulus ratio was developed for a spherical indentation on a coated system [152]. In another study, the CRID is formulated quantitatively in terms of the ratios of Young's modulus and yield stresses between the coating and the substrate for a range of ratios with a conical indenter [153].

In the present study, a series of numerical modelling of indentation tests on a coated system are carried out to reveal a general picture of how the CRID is influenced by the ratios of Young's modulus and yield stresses of the coating and the substrate using a conical indenter with a half angle of 70.3° . Maps of the CRID vs. the Young's modulus and the yield stress ratios have been generated. The major objective of this study is to provide a general guidance for the future experimental work on a coated system to decide on an appropriate indentation depth for the given materials and coating thickness. The present results are compared and critically discussed with the relevant experimental findings and the quantitative formulations proposed in a predecessor study [153] for a similar case.

6.4.4.1.2. Indentation Testing and The FE Model

Indentation testing

An illustration of an indentation test on a coated system with a conical indenter is shown in Figure 6.16. A loading force P is applied to the indenter to press it into the coating material. θ is the half indenter angle. h and h_0 are the indentation depth and the coating thickness, respectively. Figure 6.17 shows a typical indentation load-displacement curve obtained from a ductile

material, in which the loading part can be characterised by Kick's law; $P=Ch^2$, where C is the loading curve curvature. The initial unloading part can be interpreted to relate the unloading contact stiffness, $S=dP/dh$, to the Young's modulus, E , using Olive and Pharr's theory [11]. The surface profile of an indented material, e.g. obtained from atomic force microscope, is another type of indentation data that can be related to the mechanical properties of the material (e.g. [154]).

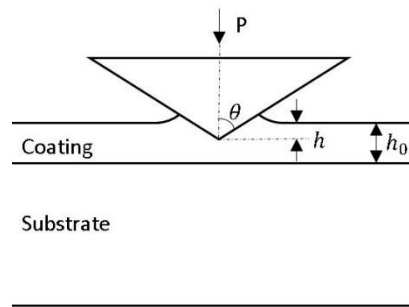


Figure 6.16. Illustration of an indentation test on a coated system.

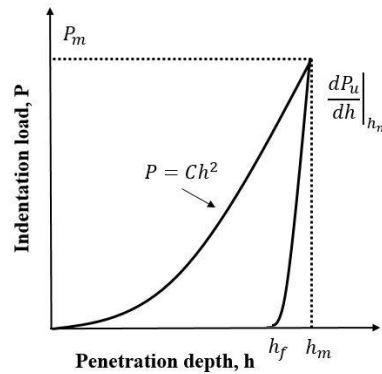
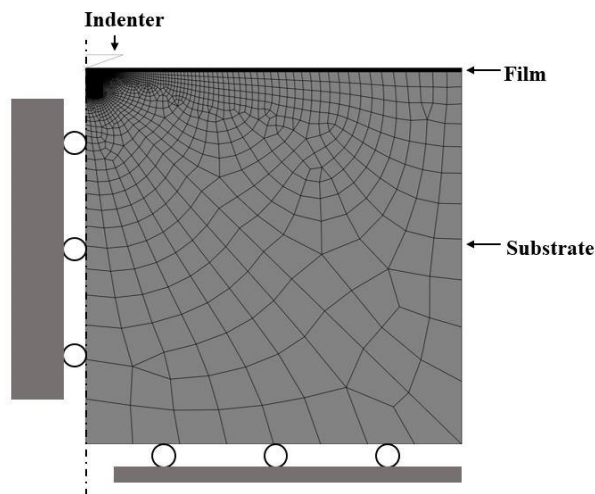


Figure 6.17. Illustration of a typical indentation load-displacement curve.

FE model

A finite element model was built for the indentation tests on a coated system using the commercial code ABAQUS 6.14-1. The model is presented by Figure 6.18. As Figure 6.18 (a) shows, a conical indenter with a half angle of 70.3° was used for the axisymmetric model (CAX3R and CAX4R elements). The selected indenter angle gives the conical indenter the same contact area-to-depth ratio as the Berkovich and Vicker pyramid indenters [155]. The horizontal displacement on the vertical axis of symmetry and the vertical displacement in the bottom surface were set to zero. Figure 6.18 (b) shows the refined mesh used for the coating layer due to the expected high plastic strains and the relatively coarse mesh used for the substrate material to reduce the time of computation. The substrate thickness, 2.5mm, is much larger than the coating thickness, $25\mu\text{m}$. Both the coating and substrate materials were modelled as elastoplastic. The indenter was modelled as rigid body, i.e. the indentation deformation was neglected. The friction between the indenter and the coating surface was assumed to be zero since its influence on obtaining the load-displacement curves from the indentation tests can be negligible [36]. A downward displacement was applied to the indenter to achieve a maximum indentation depth, i.e. 50% of the coating thickness. The reaction force on the indenter tip was recorded together with the indenter displacement to produce the load-displacement curves.



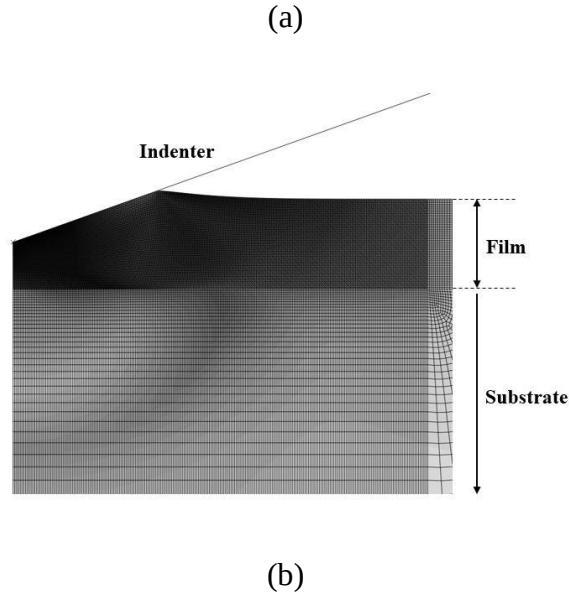


Figure 6.18. FE model of a conical indenter on a coated system: (a) boundary conditions (b) film mesh

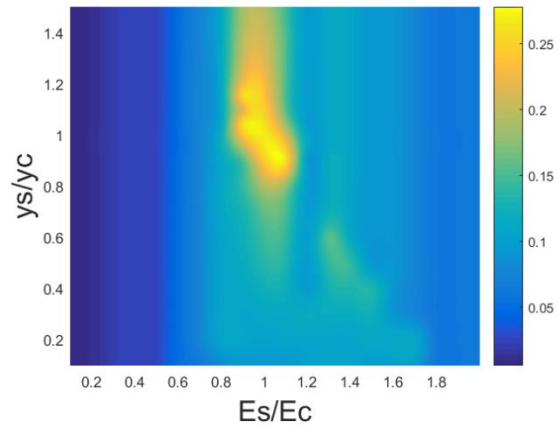
6.4.4.1.3. Parametric Study Results

The relationships between the CRID and $\frac{E_s}{E_c}$ and $\frac{\sigma_{ys}}{\sigma_{yc}}$ were investigated in this study. The critical indentation depth, h_c , is defined as the indentation depth at which the deviation of the coated system indentation response, P_i , from the indentation response of the single coating material, P_0 , exceeds 0.5% or 1%: $\frac{P_i - P_0}{P_0} > 0.5\%$ or $\frac{P_i - P_0}{P_0} > 1\%$. Therefore, the indentation response of the single coating material was obtained from FE modelling first. The CRID values were identified for different substrate material properties after the simulations of the indentation tests on the coated system were completed. Two different coating materials (different Young's modulus and yield stress) were used in the present study. Simulations were carried out for a range of the Young's modulus and yield stress, i.e. $0.1 < \frac{E_s}{E_c} < 2$, $0.1 < \frac{\sigma_{ys}}{\sigma_{yc}} < 1.5$ both with the increment of 0.1, where E_s , E_c , σ_{ys} and σ_{yc} are the Young's modulus and yield stress of the substrate and coating, respectively. It should

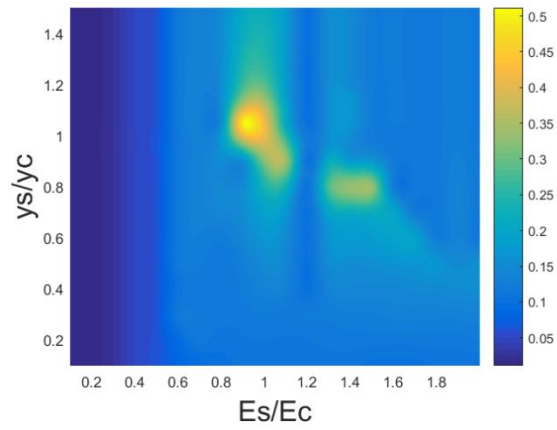
be noted that the Young's modulus and the yield stress of the coating material were set as constant and the material mismatch was achieved by altering the Young's modulus and the yield stress of the substrate material. The Poisson's ratio was assumed to be 0.3 for simplicity.

Coating material 1

Figure 6.19 shows the variation of the CRID with $\frac{E_s}{E_c}$ and $\frac{\sigma_{ys}}{\sigma_{yc}}$ for the first coating material. Figures 19 (a) and (b) show the results obtained from the critical error tolerance of 0.5% and 1%, respectively. It can be found that the patterns of the two figures are similar, indicating good consistency of the simulations. It can also be found that the rule-of-thumb suggesting the maximum indentation depth to be less than 10% of the coating thickness is applicable only when $\frac{E_s}{E_c} > 0.5$, approximately. When $\frac{E_s}{E_c} < 0.5$ the 10% rule-of-thumb is not applicable. For example, when $\frac{E_s}{E_c} = 0.3$, the CRID is less than 5%. In most cases, the CRID is mainly dependent of $\frac{E_s}{E_c}$ and relatively independent of $\frac{\sigma_{ys}}{\sigma_{yc}}$, i.e. the CRID is almost constant for a given value of $\frac{E_s}{E_c}$. However when $\frac{E_s}{E_c}$ is close to 1, the CRID is dependent of $\frac{\sigma_{ys}}{\sigma_{yc}}$, especially for $\frac{\sigma_{ys}}{\sigma_{yc}} > 1$.



(a)



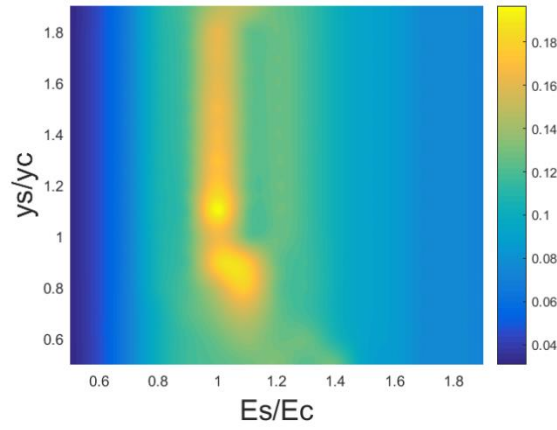
(b)

Figure 6.19. CRID maps of material 1 in terms of $\frac{E_s}{E_c}$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}}$: (a) error tolerance of 0.5% (b) error tolerance of 1%.

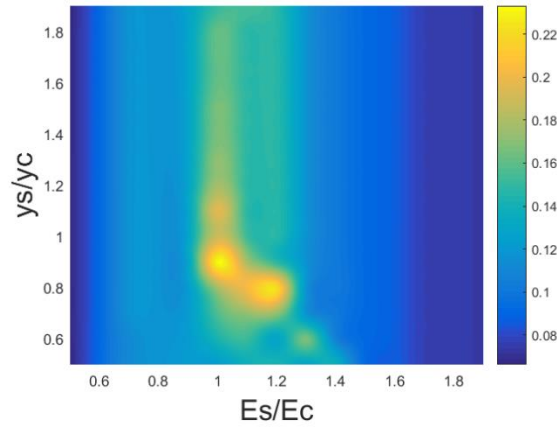
Coating material 2

Figure 6.20 shows the variation of the CRID with $\frac{E_s}{E_c}$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ (both ranged from 0.5 to 1.9) for the second coating material. It can be found that the patterns of the CRID are similar to the ones from Figure 6.19 for the same

range of $\frac{E_s}{E_c}$ and $\frac{\sigma_{ys}}{\sigma_{yc}}$: the CRID mainly is mostly dependent of $\frac{E_s}{E_c}$ and relatively independent of $\frac{\sigma_{ys}}{\sigma_{yc}}$. These similarities indicate that the effects of the mechanical material ratios between the substrate and coating on the CRID is generally independent of the mechanical properties of the coating.



(a)



(b)

Figure 6.20. CRID maps of material 2 in terms of $\frac{E_s}{E_c}$ and $\frac{\sigma_{ys}}{\sigma_{yc}}$: (a) error tolerance of 0.5% (b) error tolerance of 1%.

6.4.4.1.4. Discussion

A quantitative expression for the CRID of the indentation on a coated system with a conical indenter has been proposed by Gamonpilas [153] based on a series of dimensional analysis and numerical simulations as follows:

$$\text{CRID} = \exp^{-1.695+0.501/\bar{\sigma}-0.444 \ln \bar{\sigma}+0.065/\bar{E}-0.17 \ln \bar{E}} \quad 6.5$$

where $\bar{\sigma} = \frac{\sigma_{y_c}}{\sigma_{y_s}}$ and $\bar{E} = \frac{E_c}{E_s}$. The CRID values calculated from Eq. 6.5 for the similar range of substrate/coating material properties ratios investigated in the present study are shown in Figure 6.21. Figure 6.21 shares some common features with Figure 6.19 and 6.20: (a) The CRID is generally more sensitive to $\frac{E_s}{E_c}$ than to $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ within the current range of investigation and (b) the CRID is relatively small for a hard coating on a soft substrate, i.e. $\frac{E_s}{E_c} < 1$. However, Figure 6.21 differs from Figure 6.18 and 6.19 as the larger values of CRID are not seen when $\frac{E_s}{E_c}$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ are close to 1 but when $\frac{E_s}{E_c} > 1.5$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}} > 1$. This result is not expected since when $\frac{E_s}{E_c}$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ are close to 1 the coating and substrate should show similar indentation responses and hence the CRID is expected to be relatively larger. It should be noted that the fitting equation Eq. 6.5 [153] is obtained from a different range of material mismatch as the current study, e.g. $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ is generally less than 1 and as low as 0.02. It should also be noted that only a few different values of $\frac{E_s}{E_c}$ are investigated by Gamonpilas [153], therefore the case of $\frac{E_s}{E_c}$ and $\frac{\sigma_{y_s}}{\sigma_{y_c}}$ close to 1 may not be fully

taken into account for the fitting expression and the calculations of CRID for the extended range of $\frac{E_s}{E_c}$ may not be applicable.

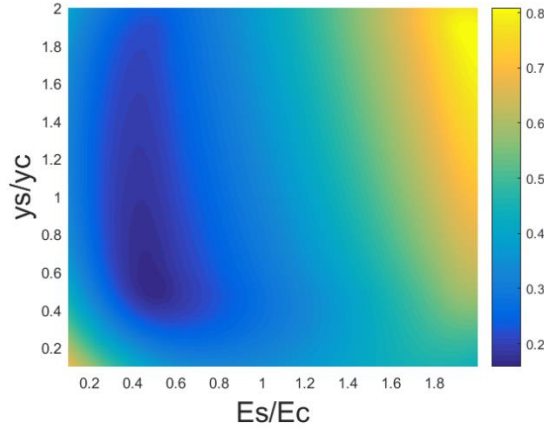


Figure 6.21. CRID map generated from reference [153].

The current parametric study provides some insight to the relationships between the CRID and $\frac{E_s}{E_c}$ and $\frac{\sigma_{ys}}{\sigma_{yc}}$ for a two-material system. The information obtained from this numerical study could provide as a preliminary guidance for selecting a combination of materials for indentation tests on the coating-substrate specimens, though the CRID maps need comprehensive experimental results for verification.

6.4.4.2 Indentation Tests on a CoNiCrAlY Coating at RT

6.4.4.2.1. Nano-Indentation Testing Method

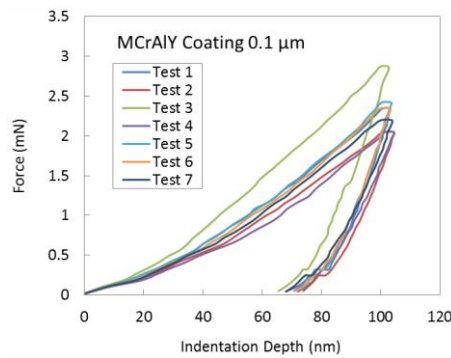
The nano-indentation tests were carried out using the Micro Materials Ltd. (MML) Nanotest NTX platform 2 and a diamond Berkovich indenter at RT as displacement-controlled tests, i.e. a maximum indentation depth (h_{max}) from 0.1 to 2 μm was set for each set of tests with a constant loading rate of 0.01 $\mu\text{m/s}$. The indenter was held in position at the maximum depth for 10 seconds. Five to seven repeated tests were performed for each depth. The

indents were spaced in the distance of 50 μm (larger than the required minimum distance for the standard of BS EN ISO 14577 [49] to avoid the possible interference).

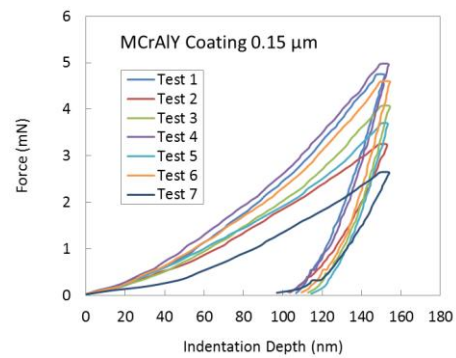
6.4.4.2.2. Nano-Indentation Testing Results

The load-displacement curves obtained from the nano-indentation tests for the CoNiCrAlY coating are presented in Figure 6.22. It can be found that the scattering is more significant for the tests of smaller indentation depth, e.g. $h_{max} < 0.5 \mu\text{m}$. It could be due to the effects of the possible variations in the surface finish between different specimens and local defects, such as porosity. Therefore, it was suggested to apply a relatively large maximum indentation depth to ensure the reliability of the nano-indentation tests.

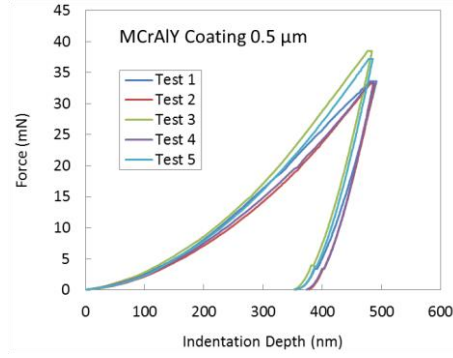
The load-displacement curves obtained from the indentation tests can be used, together with the surface profile of the indented specimen, to determine the mechanical properties of the material (demonstrated in Chapter 4). However the surface finish of this indented coating specimen was not good enough for the use of atomic force microscopy (AFM), i.e. the relatively large surface roughness and the local defects make it difficult to identify the indentation imprints. Therefore, improved surface finish will be required for future tests.



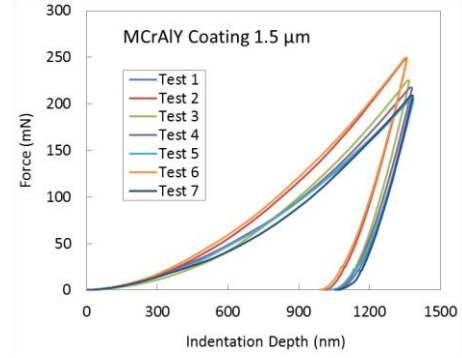
(a)



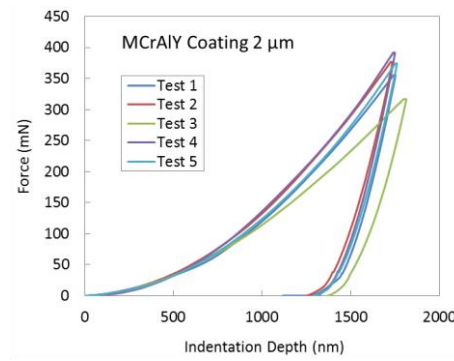
(b)



(c)



(d)



(e)

Figure 6.22. Nano-indentation tests on the CoNiCrAlY coating: (a) $h_{max} = 0.1 \mu\text{m}$ (b) $h_{max} = 0.15 \mu\text{m}$ (c) $h_{max} = 0.5 \mu\text{m}$ (d) $h_{max} = 1.5 \mu\text{m}$ (e) $h_{max} = 2 \mu\text{m}$

6.5 Conclusions on Characterisation of CoNiCrAlY Coating

A series of experimental data of the CoNiCrAlY Coating have been presented in this section, including SPT testing, miniature tensile testing and indentation testing. The results from the SPT and the miniature tensile testing have been found to be comparable regarding the DBTT and fracture surface characterisation. The DBTT has been found to be 600-700°C. The tensile testing results could provide valuable data for the future study on the coating-on-substrate specimens. Consistent load-displacement curves have been obtained from the nano and micro indentation tests at RT for the coating material. The surface conditions need to be improved in the future before AFM examination could be carried out.

Chapter 7 – Conclusions and Future Work

This chapter includes the conclusions of this study and the suggestions for future work regarding 1) the characterisation of coatings using the miniature specimen testing methods and 2) the development and applications of the inverse methods.

7.1. Characterisation of Coatings Using the Miniature Specimen Testing Methods

7.1.1. Conclusions

An analytical study was carried out to develop a characterisation method for a two-material coated system. It was demonstrated that the material properties of an unknown coating could be determined from the compound testing results and the known material behaviours of the other material (substrate) in conjunction with an inverse method.

SPT tests were carried out for the CoNiCrAlY coating from RT to 700°C. This coating material exhibits a clear ductile-to-brittle transition: brittle fracture is observed using SEM for temperatures from RT to 500°C while typical ductile fracture is observed at 700°C. Also, the fracture energy, which remains relatively constant between RT and 500°C, tends to increase rapidly when temperature is increased from 500 to 700°C. A notable phenomenon observed for the SPT tests at lower temperatures is the sudden drop in the loading force at an early stage due to cracking, which could contribute to the observed scattering of the low temperature SPT test results. In summary, the SPT testing is less effective and reliable as a mechanical testing technique for brittle materials in terms of deriving elastic-plastic properties from such a test. However, it could be a potential tool to investigate the cracking and fracture mechanics of the materials.

A novel miniature specimen testing method was developed and used for the miniature tensile and creep tests of a P91 steel and the same CoNiCrAlY coating used for the SPT tests. The results of the miniature tensile tests show good agreements with the SPT tests regarding the DBTT of this coating material; the material exhibits a relatively low ductility from RT to 500°C and very high ductility at 700°C. The mechanical properties determined from the miniature tensile tests were also able to provide comparable predictions of the corresponding SPT tests, including the early cracking at lower temperatures, using FE modelling. The tensile behaviours acquired from the miniature tensile tests on the coating specimens also provide the source of data for the characterisation of the two-material system. It should be noted that, dissimilar to the SPT tests, the repeatability of the miniature tensile tests on the coating material is excellent at all temperatures, indicating that this testing method has the potential to become an alternative to the SPT testing and a universally applicable miniature specimen testing technique for mechanical characterisation.

Heat treatment was performed for the P91 steel to replicate the one received by a typical thermal barrier coating during the manufacturing process. Miniature tensile and creep tests on the heat-treated P91 steel provide the data of the single-material elastic-plastic and creep behaviours of the substrate material for the future work of the characterisation of a two-material coated system. The repeatability and consistency of these miniature tests were very good, though the results would be more improved if repeated experiments could be carried out for a more comprehensive range.

7.1.2. Future Work

Miniature creep tests can be carried out on the single-material CoNiCrAlY coating specimens to obtain the ‘reference’ material behaviours. Miniature tensile and creep tests can be carried out on the two-material coated specimens consist of the CoNiCrAlY coating and the heat-treated P91 steel.

The compound tensile and creep behaviours can be used to extract the material behaviours of the CoNiCrAlY coating. The obtained CoNiCrAlY coating material properties will be compared to the ‘reference’ single-material behaviours.

Regarding the micro/nano-indentation tests on the coating specimens, the next step should be taking accurate measurements of the surface profile for the indented specimen. For example, atomic force microscopy (AFM) can be used to measure the surface displacement across the indentation imprint. It should be noted that this technique requires very good surface finish, i.e. small roughness and minimised local defects, which could be challenging for the coating specimens. Indentation tests could also be carried out on the two-material coated specimens. For example, it is worth investigating the critical indentation depth of the two-material coated specimens.

7.2. Development and Applications of the Inverse Methods

7.2.1. Conclusions

Inverse methods were developed to determine the material properties from the experimental results based on the nonlinear optimisation function and FE modelling or analytical solutions. During the development stage, the influence of using a load-controlled or displacement-controlled FE model in the commercial code ABAQUS was investigated. Generally, the displacement-controlled model gives faster and more accurate convergence for the optimisation algorithm. The scaling coefficients of the multi-objective optimisation, which increase or decrease the ratio of the target function value to the total function value, were found to have significant effects on the efficiency and accuracy of the inverse method. Some parametric studies are necessary for finding the appropriate scaling coefficients for the specific scenario. The inverse method was firstly demonstrated taking the theoretical indentation testing as an example and proved to be able to extract the material properties with enough input of ‘experimental’ results. The inverse method

was also modified and used for more advanced applications, such as 1) determining the full-stage creep and damage properties from the uniaxial creep tests, 2) determining the elastic-plastic properties of a coating from the miniature tensile tests at different temperatures and 3) determining the unknown coating material properties from the compound testing results of a two-material system.

7.2.2. Future Work

The inverse method's ability of including the predefined constraints can be strengthened. For example, the minimum strain rate and the time to failure obtained from the creep tests could be crucial constraints for the determination of the full-stage creep and damage properties. The efficiency and accuracy of the inverse method could potentially be well improved if those constraints can be effectively applied. Also, it is worth developing a method to monitor the progress of the FE modelling of creep testing and to terminate it under the predefined conditions. For instance, the time increment becomes very small, close to zero, when the FE modelling of the creep deformation evolves close to failure, but the FE modelling could continue for quite a long time. If the total deformation, the current time increment, and the current displacement/strain rate can be monitored and the FE modelling can be terminated under predefined conditions, the overall computing time of the inverse method could be reduced significantly.

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