Modelling damage and fracture evolution in plasma-sprayed ceramic coatings: effect of microstructure

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Modelling Damage and Fracture Evolution in Plasma Sprayed Ceramic Coatings: Effect of Microstructure

by Jian Zhao

A Doctoral Thesis
Submitted in partial fulfilment of the requirements for the award of
Doctor of Philosophy of Loughborough University

March 2005
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Abstract

Ceramic coatings are thin layers bonded to substrates (typically metal ones) for protection from wear, chemical attack and high-temperature environments. They have been increasingly used in industry for many years. However, stresses induced by the properties mismatch between coatings and substrates, as well as brittleness of ceramics, may lead to failure of coatings in service, and hence they have been the principal problem encountered with ceramic coatings. It has been known that the microstructure of ceramic coatings has a significant effect on their mechanical and thermal properties. However, there has been neither a standard account for the effect of microstructure on damage and fracture evolution in coatings nor an adequate quantitative description of that effect. Little is known about crack nucleation processes at the microscale, while extension of damage and fracture mechanics to microstructures has become an urgent concern. These challenges await serious studies.

Thus, the purpose of this research was to investigate damage and fracture evolution in ceramic coatings under mechanical and/or thermal loading, as well as to study the effect of microstructure on properties of ceramic coatings and their behaviour under loading, by means of numerical and experimental studies. The principal points of this thesis are as follows:

- Various experiments were employed in order to determine some material properties of plasma sprayed alumina coatings and to study their damage and fracture phenomena under loading. The experiments involved nano-indentation, three-point bending, laser-induced thermal shock and infrared thermography.
- With the use of the image analysis technique and statistical analysis, microstructure of plasma sprayed alumina coatings was quantitatively characterised using such parameters as the size, shape and number (density) of microvoids and the porosity level in the coatings. Based on this approach, the effective mechanical and thermal properties of the coatings with low porosity (less than 10%) were calculated; the effect of microstructure on the material properties was investigated quantitatively and qualitatively.
- A constitutive relation with consideration of damage and fracture evolution in heterogeneous brittle materials was proposed by extending a damage evolution theory from isotropic materials to anisotropic ones. Then, a framework for the multiscale analysis of damage and fracture in ceramic coatings combined with the constitutive relation was proposed in terms of the finite element mesh superposition method.

- Based on the proposed methods, failure mechanisms in plasma sprayed alumina coatings under three-point bending and thermal loading were investigated quantitatively and qualitatively with the account for the microstructure of ceramic coatings. The obtained results agree with experimental ones and indicate that the numerical models allow for predictions of the stress-strain state and distributions of damage in the coating under loading, as well as the possible positions, where failures may occur.

Little has been done so far to model damage and fracture evolution of ceramic coatings under loading, especially for multi-axial loading cases, with the consideration of their microstructure, from the point of view of continuum damage mechanics. This thesis contributes to this area of study.
Acknowledgement

I am grateful to many people. Without them, this research would not have been possible.

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My family for their love, care and support during this research.

Last but not least, the EPSRC for the studentship.
Publications

The research undertaken during the course of this project has given rise to publications listed below.


5. Jian Zhao and Vadim V. Silberschmidt, Micromechanical analysis of effective properties of ceramic coatings, submitted to *Journal of Materials Science and Engineering A.*
# Notation and Abbreviations

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<thead>
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<th>Symbol</th>
<th>Meaning</th>
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<tbody>
<tr>
<td>$A$</td>
<td>Area</td>
</tr>
<tr>
<td>$a$</td>
<td>Length of major axis of an elliptical void; the maximum size of voids</td>
</tr>
<tr>
<td>$a_0$</td>
<td>Material parameters; crack size</td>
</tr>
<tr>
<td>$b$</td>
<td>Length of minor axis of an elliptical void</td>
</tr>
<tr>
<td>$C_{ij}$</td>
<td>Components of a stiffness matrix</td>
</tr>
<tr>
<td>$c_1, c_2$</td>
<td>Microstructure-dependent material constants</td>
</tr>
<tr>
<td>$c_p$</td>
<td>Specific heat</td>
</tr>
<tr>
<td>$D$</td>
<td>Damage tensor</td>
</tr>
<tr>
<td>$D$</td>
<td>Damage</td>
</tr>
<tr>
<td>$D_0$</td>
<td>Initial damage</td>
</tr>
<tr>
<td>$D_i$</td>
<td>Damage in the principal direction $i$</td>
</tr>
<tr>
<td>$D_c$</td>
<td>Damage variable induced by cracks</td>
</tr>
<tr>
<td>$D_v$</td>
<td>Damage variable induced by voids</td>
</tr>
<tr>
<td>$d$</td>
<td>The distance between the neutral axis and the surface of the coating in the bending tests</td>
</tr>
<tr>
<td>$E$</td>
<td>Young's modulus</td>
</tr>
<tr>
<td>$E_0$</td>
<td>Young's modulus of an undamaged material</td>
</tr>
<tr>
<td>$E_i$</td>
<td>Young's modulus of the indenter in the nanoindentation tests</td>
</tr>
<tr>
<td>$E_e$</td>
<td>Effective Young's modulus</td>
</tr>
<tr>
<td>$E_L$</td>
<td>Young's modulus in spray direction</td>
</tr>
<tr>
<td>$E_r$</td>
<td>Reduced Young's modulus in the nanoindentation tests</td>
</tr>
<tr>
<td>$E_s$</td>
<td>Young's modulus of a sample in the nanoindentation tests</td>
</tr>
<tr>
<td>$E_T, E_{ex}$</td>
<td>Young's modulus in transverse direction</td>
</tr>
<tr>
<td>$e_{ij}$</td>
<td>Parameters dependent on the Poisson's ratio of an undamaged solid</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
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<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>$F$</td>
<td>Force</td>
</tr>
<tr>
<td>$F_c$</td>
<td>Failure force</td>
</tr>
<tr>
<td>$G$</td>
<td>Energy release rate</td>
</tr>
<tr>
<td>$G_0$</td>
<td>Nominal bulk modulus</td>
</tr>
<tr>
<td>$G_e$</td>
<td>Effective bulk modulus</td>
</tr>
<tr>
<td>$h$</td>
<td>Penetration depth</td>
</tr>
<tr>
<td>$h_1, h_2, h_3$</td>
<td>Shape factors of microvoids</td>
</tr>
<tr>
<td>$H$</td>
<td>Hardness</td>
</tr>
<tr>
<td>$H_1$</td>
<td>Thickness of a coating</td>
</tr>
<tr>
<td>$H_2$</td>
<td>Thickness of a substrate</td>
</tr>
<tr>
<td>$I_c$</td>
<td>The centroidal moments of inertia of the area of a coating</td>
</tr>
<tr>
<td>$I_s$</td>
<td>The centroidal moments of inertia of the area of a substrate</td>
</tr>
<tr>
<td>$K_0$</td>
<td>Nominal shear modulus</td>
</tr>
<tr>
<td>$K_e$</td>
<td>Effective shear modulus</td>
</tr>
<tr>
<td>$K_1$</td>
<td>Stress intensity factor</td>
</tr>
<tr>
<td>$K_{ic}$</td>
<td>Critical stress intensity factor</td>
</tr>
<tr>
<td>$k_0$</td>
<td>Thermal conductivity of an undamaged material</td>
</tr>
<tr>
<td>$k_{ext}, k_{ey}, k_{ez}$</td>
<td>Effective thermal conductivity of a material in $x, y, z$ directions</td>
</tr>
<tr>
<td>$L$</td>
<td>Length</td>
</tr>
<tr>
<td>$\Delta L$</td>
<td>Crack extension</td>
</tr>
<tr>
<td>$M$</td>
<td>General representation of a material modulus (bulk, shear or Young's modulus); actual number of the microdefects in a material</td>
</tr>
<tr>
<td>$M_0$</td>
<td>Modulus of an undamaged material or a zero-porosity material</td>
</tr>
<tr>
<td>$M_e$</td>
<td>Effective modulus of a material</td>
</tr>
<tr>
<td>$m$</td>
<td>Weibull parameter</td>
</tr>
<tr>
<td>$N$</td>
<td>Average number of flaws in a material</td>
</tr>
<tr>
<td>$N_0$</td>
<td>Total number of microvoids in an area</td>
</tr>
<tr>
<td>$N_{i}$</td>
<td>The number of the $i$-th type of microvoids in a RAE</td>
</tr>
<tr>
<td>$n_0$</td>
<td>Material parameters</td>
</tr>
<tr>
<td>$n_a$</td>
<td>Voids density</td>
</tr>
<tr>
<td>$n_{ss}$</td>
<td>Number of microvoids types</td>
</tr>
<tr>
<td>$p, p_0$</td>
<td>Porosity</td>
</tr>
</tbody>
</table>
\( p_i \) Probability of the \( i \)-th type of microvoids
\( P \) Contact force in the nanoindentation tests
\( P_L \) Laser power
\( q_{\text{max}} \) The maximum heat flux in the thermal shock tests
\( R \) The radius of an isotropic elastic solid
\( R_x, R_y \) Normalised coordinates
\( r \) The distance from the crack tip; the spot radius containing 86.6% of the laser power in the thermal shock tests
\( r_0 \) Material parameters
\( S \) Support span in the bending and thermal shock tests; contact compliance in the nanoindentation tests
\( S_f \) Shape factor
\( T \) Temperature
\( \Delta T \) Thermal shock resistance or temperature difference
\( \Delta t \) Interaction traction tensor between microvoids
\( \Delta t_0 \) Initial time step
\( t_c \) Thickness of a coating in the bending tests
\( t_s \) Thickness of a substrate in the bending tests
\( V \) Volume
\( W \) Width
\( W^* \) Energy absorption capacity
\( W_{\text{eq}} \) The equivalent width of a specimen in the bending tests
\( W_{\text{perf}} \) Strain energy of an undamaged material
\( x, y, z \) Coordinates
\( \Delta x \) Mesh size
\( Y \) A geometric function dependent on the crack size normalized to characteristic dimension of a material
\( \alpha \) Coefficient of thermal expansion
\( \beta \) Voids (hole) density tensor
\( \beta_M \) A function of \( v_0 \) corresponding to each \( M \) (bulk, shear or Young's modulus)
\( \gamma \) \hspace{1cm} \text{Surface energy of an intergranular cracked interface}

\( \theta \) \hspace{1cm} \text{Angle}

\( \nu_0 \) \hspace{1cm} \text{Poisson's ratio of an undamaged material}

\( \nu_e \) \hspace{1cm} \text{Effective Poisson's ratio}

\( \nu_i \) \hspace{1cm} \text{Poisson's ratio of the indenter in the nanoindentation tests}

\( \nu_s \) \hspace{1cm} \text{Poisson's ratio of a sample in the nanoindentation tests}

\( \delta \) \hspace{1cm} \text{Deflection}

\( \delta_{11} \) \hspace{1cm} \text{A very small positive number}

\( \rho \) \hspace{1cm} \text{Density of a material}

\( \rho_0 \) \hspace{1cm} \text{Average number of voids in a unit area}

\( \rho_{i,v} \) \hspace{1cm} \text{Voids density of the } i\text{-th type of microvoids in a RAE}

\( \varepsilon \) \hspace{1cm} \text{Strain; a very small positive number}

\( \varepsilon_i \) \hspace{1cm} \text{Major principal strain}

\( \sigma \) \hspace{1cm} \text{Stress}

\( \sigma_c \) \hspace{1cm} \text{Fracture strength}

\( \sigma_{ij} \) \hspace{1cm} \text{Components of the stress tensor}

**Abbreviations**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Meaning</th>
</tr>
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<tbody>
<tr>
<td>BCs</td>
<td>Boundary conditions</td>
</tr>
<tr>
<td>CCD</td>
<td>Charge coupled device</td>
</tr>
<tr>
<td>CDM</td>
<td>Continuum damage mechanics</td>
</tr>
<tr>
<td>CSM</td>
<td>Composite sphere method</td>
</tr>
<tr>
<td>DM</td>
<td>Differential method</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite element method</td>
</tr>
<tr>
<td>FM</td>
<td>Fracture mechanics</td>
</tr>
<tr>
<td>GCS</td>
<td>Global coordinate system</td>
</tr>
<tr>
<td>GMC</td>
<td>Generalized method of cells</td>
</tr>
<tr>
<td>GSCM</td>
<td>Generalized self-consistent method</td>
</tr>
<tr>
<td>IR</td>
<td>Infrared</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
</tr>
<tr>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>LCS</td>
<td>Local coordinate system</td>
</tr>
<tr>
<td>MSA</td>
<td>Minimum solid area</td>
</tr>
<tr>
<td>MTM</td>
<td>Mori-Tanaka method</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical vapour deposition</td>
</tr>
<tr>
<td>QWIP</td>
<td>Quantum Well Infrared Photon,</td>
</tr>
<tr>
<td>RAE</td>
<td>Representative area element</td>
</tr>
<tr>
<td>RVE</td>
<td>Representative volume element</td>
</tr>
<tr>
<td>SCM</td>
<td>Self-consistent method</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>TBC</td>
<td>Thermal barrier coating</td>
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Chapter 1 Introduction

1.1 General Introduction

Ceramic coatings are thin layers bonded to substrates for protection from wear, chemical attack and high-temperature environments. They have been increasingly used in industry for many years. For example, Thermal Barrier Coatings (TBCs) are widely used in aerospace engineering. They can increase the temperature range of gas turbines by 90 to 200°C above the level that is now achievable with most advanced super-alloys [1]. Such an increase could provide an improvement of up to 20% in fuel economy. The coatings are often made of ceramics, which usually demonstrate a high stiffness-to-weight ratio, wear resistance, and high-temperature stability, while substrates are typically metals and alloys. Ceramic coatings allow for increased operating temperatures, increased efficiency, improved component durability, simplified designs, improved fuel economy, and sometimes the use of less expensive metallic substrates. Thus, TBCs represent an attractive approach to enhance the high-temperature limits for systems such as gas turbines, aircraft engines and diesel engines.

However, significant differences in properties between a ceramic coating and a substrate, as well as severe temperature gradients in such systems under their service conditions, cause thermal mismatch stresses that may affect resistance of the coating to wear and fatigue crack propagation. The presence of the stresses may promote delamination and spallation of the coating, especially in the case of thicker coatings, due to the increasing release of stored elastic strain energy induced by stress redistribution/relaxation [2].

In another aspect, ceramics are brittle and usually suffer from low fracture resistance. The main issues in designing with a brittle material are that a very large scatter in strength (under tensile stress), a lack of capacity for mitigating stress concentrations via plastic flow, and relatively low energy absorption prior to failure, dominate the mechanical behaviour. Thus, designers traditionally viewed brittle materials as unreliable when exposed to the tensile stress. However, since the early 1980s, brittle materials,
advanced ceramics in particular, have demonstrated reliable performance in many highly stressed structural applications, such as bearings, cutting tools, turbocharger rotors, missile guidance domes, etc. [3, 4]. Some methods [5, 6] have been proposed to overcome the problem of brittleness of ceramics. A major factor underlying these successes has been the development of a more thorough understanding of the role of micro voids/flaws in damage and fracture of ceramics.

Thermal stress-induced spallation, caused by brittleness of ceramic coatings and large thermal expansion mismatch between the coating and substrate, has been the most obvious failure mode for TBCs. Most commercial applications to date have had to be designed so as to be non-catastrophic in case of coating failure [1]. In order to overcome the problems with TBCs, much research has been carried out. Most solutions are related to reducing Young's modulus and residual stresses in the coatings. These require a fundamental knowledge of the coatings' damage and fracture mechanisms, which have been poorly understood so far because of the complex microstructure of ceramics. It is known that the use of TBC systems [1, 7] is a function of their architecture, composition, morphology and adhesion to the metal surface, on which they are applied. This is due to porosity of these systems, and pores in TBCs are believed to be able to reduce the layer's thermal conductivity and increase its in-plane compliance. Thus the properties of the layer are particularly dependent on the pore morphology, which is determined by the method used to apply the coating.

For thin coatings (usually denser and produced by deposition from the vapour phase), the stress level can be considered as essentially uniform and the effect of microstructure could be neglected, while for thicker ones (commonly formed by much faster processes such as droplet spraying), through-thickness variations in the stress level can be significant. These make full analyses for damage and fracture evolution in coatings much more complicated. Figure 1-1 shows a cross-sectional micrograph of a typical alumina coating [8]. It can be seen that there are various voids of different sizes and shapes distributed in the coating. Some studies [9-13] have reported that the voids do have a significant effect on material's properties of ceramics. Hitherto, there has been neither a standard account for the effect of voids on damage and fracture evolution in coatings nor an adequate quantitative description of that effect. Especially, little is known about crack nucleation processes [14] at the microscale, while extension of damage and fracture mechanics to microstructures has become an urgent concern. These challenges await serious studies.
1.2 Aims and Objectives of This Research

The purpose of this research was to investigate damage and fracture evolution in ceramic coatings under mechanical and/or thermal loadings, as well as to study the effect of microstructure on the properties of ceramic coatings and their behaviour under loading, by numerical and experimental studies. It was expected that these studies provide an explicit account for arbitrary microstructural morphologies and microscopic fracture patterns, and thus, make it easier to identify and design microstructural configurations that enhance fracture toughness, and hence could lead to improvements in the manufacturing of ceramic coatings.

There are various types of damage such as brittle damage, creep damage, plastic damage, fatigue damage and dynamic damage [15, 16]. In this research, the brittle damage, which plays the major role in the response of ceramics to external loadings, was studied. The research mainly aimed at examining damage and fracture evolution in ceramic coatings by means of numerical simulations within the framework of Continuum Damage Mechanics (CDM). Then CDM models were related to fracture mechanics (FM) and statistical schemes so that numerical simulations could reflect not only the damage
Chapter 1 Introduction

evolution process but also damage-induced anisotropy and randomness of fracture in ceramic coatings. In other words, the study focused on exploring the effects of such micro-structural parameters as the size, shape and number (density) of microvoids and the porosity level on the processes of damage and fracture evolution in alumina coatings.

Since externally applied thermal or mechanical loads in many practical cases are at the structural level, the suggested models should also be capable of correlating the micro-structural response with the overall macroscopic behaviour. However, coupling micro and macro scales still remains one of the most challenging goals despite all the developments in the area of micromechanics. Although in some cases fair comparisons with micrographs of damaged specimens have been reported [17-20], only a few of these studies have actually made a direct connection with experimental data in order to gain strong conclusions about failure mechanisms and the real macroscopic response of brittle materials. Accordingly, multiscale numerical simulations and their validation for damage analysis would be necessary. Through computation of stress/strain fields and damage distributions in real and idealized microstructures, different mechanisms controlling the macroscopic material’s response can be better understood.

Material properties of ceramic coatings are closely related to their composition and deposition processes. Plasma spraying process is one of the most common coating deposition processes used in industry, and alumina is considered to be a suitable material as an oxygen barrier of the TBCs for its low oxygen diffusivity at high temperature. Thus, plasma sprayed alumina coatings were selected in this research as modelling objects. Some parameters of bulk alumina under loading, for development of CDM models, have been obtained in the experimental results [21]. It is still necessary to conduct further experiments to determine the parameters and other properties of plasma sprayed alumina coatings, as well as to validate and justify the numerical results.

The objectives of the thesis are therefore:

• To study the development and applications of CDM, FM and multiscale finite element methods in analysing microstructure, effective properties and loading behaviour of plasma sprayed ceramic coatings.

• To undertake comparative experimental analyses of plasma sprayed alumina coatings on the basis of mechanical and thermal tests and thus provide verification for the numerical simulations.
• To model microstructure of plasma sprayed ceramic coatings by means of image analysis technique and statistical methods; and based on the proposed model, to investigate the effect of microstructure details on effective properties of plasma sprayed alumina coatings by means of analytical methods.

• To expand the definition of damage and the damage evolution theory into multiaxial states and to develop a multiscale finite element method to simulate damage and fracture evolution in heterogeneous ceramic coatings.

• To apply the proposed theory and method to simulations of plasma sprayed alumina coatings on metallic substrate under mechanical and thermal loading, to investigate damage and fracture evolution in the coatings and their loading behaviour.

1.3 Outline of the Thesis

This thesis is organised as follows.

Chapter 2 reviews the research methods used to study damage and fracture evolution in brittle materials. Various definitions of damage, damage evolution and presentation of damage at different scales are discussed and classified. Necessity for multiscale analysis of brittle materials and its applications are also introduced. This provides an essential background for the development and application of CDM, fracture mechanics and their combination used in failure analysis of brittle materials.

Chapter 3 introduces the related literature on the damage and fracture evolution in ceramic coatings from points of views of damage mechanics, fracture mechanics and applied mathematics. Plasma spraying process is briefly introduced and the resulting microstructure and effective properties of ceramic coatings are characterised in terms of recent publications. Current advances, including numerical and experimental studies, on the loading behaviour of ceramic coatings are analysed.

Chapter 4 respectively presents the performed experiments and their results including nano-indentation, three-point bending tests and laser-induced thermal shock tests. Some nominal properties of the coatings as well as phenomena observed in these tests are also presented.

Chapter 5 characterises and models microstructures of alumina coatings and their properties on the basis of image analysis and statistical modelling using an analytical
method. The effect of microstructure on the material's properties is investigated qualitatively and quantitatively.

Chapter 6 defines a multi-scalar damage variable for the case of multi-axial loading states, proposes a novel theory of damage and fracture evolution in ceramic coatings and their constitutive relationship, based on which, a framework of the multiscale analysis for damage and fracture in ceramic coatings is suggested. These methods serve as a basis for numerical simulations described in the following chapters.

Chapter 7 presents numerical simulations for alumina coatings under conditions of the three-point bending test, based on the finite element method applied at both the macroscale and mesoscale. Crack initiation, found in the tests in Chapter 4, are compared with and explained by, the numerical results.

In Chapter 8, the behaviours of alumina coatings under the laser-induced thermal shock are numerically simulated. The behaviour is presented in terms of distributions of transient temperature and its evolution, as well as fields of stresses and damage in the coating.

Chapter 9 summarizes the outcome of the research, presents conclusions drawn on the basis of the experimental results and numerical simulations, and gives recommendations for possible future work on the topic.
Chapter 2. Continuum Damage and Fracture Mechanics

This chapter reviews the related research that studies damage and fracture evolution in brittle materials. Firstly, it presents the definition of damage, damage evolution and representation of the damage at different scales, as well as classification of the methods and major tasks to be addressed by these methods. Secondly, it reviews various methods based on phenomenological models and this is followed by a discussion on damage evolution laws in the models. Thirdly, various methods based on micromechanical models are reviewed. Finally, methods of multiscale analysis for brittle materials are introduced.

2.1 Overview

Since Kachanov [22] initially postulated a damage model to study failure of materials under creep conditions, various damage and fracture approaches to the characterisation and prediction of failure modes and damage evolution have been proposed in various fields, including physics, applied mathematics, materials science and engineering, fracture mechanics and damage mechanics. In these approaches, material damage is generally considered as the presence of microscopic voids, cavities or cracks in the material. The process of void nucleation, growth and coalescence, which initiates the macrocracks and causes progressive material degradation through strength and stiffness reduction, is called damage evolution [15].

The damage and fracture approaches may be referred to three scales: the atomic scale, the microscale and the macroscale. At the atomic scale, the state of material’s damage is determined by the configuration of atomic bonds, the breaking and re-establishing of which constitute the damage evolution. At the microscale, the material’s structure is piecewise discontinuous and heterogeneous and the state of damage in a volume of the material can be determined by the number of microcracks or voids and their size and configuration. At the macroscale, the material’s state with the damage evolution is
approximated by introducing the so-called "effective state variables", e.g., effective Young's modulus, Poisson's ratio, etc., based on the equivalence principles — strain equivalence, stress equivalence, elastic energy equivalence or total energy equivalence. In all cases of various equivalence hypotheses, the true distribution of individual microvoids and microcracks is neglected and homogenized by a selection of the properly defined internal variables that characterize the damage state and are called "the damage variables". They may be scalar, vector or higher-rank tensor variables.

Besides the definition of damage variables, the other two major tasks in these approaches are to formulate damage evolution laws and a constitutive relationship for materials with damage. The former describes damage accumulation, i.e., effects of stresses and strains on damage development in materials. The latter is used to conduct a structural analysis and determine conditions for crack initiation and macroscopic failure of the structure. To approach these tasks, phenomenological and/or micromechanical models are generally used together with the equivalence principles. In phenomenological models, details of the material's microstructure are indirectly described in terms of internal damage variables (of different tensorial orders) without deep research into physical mechanisms, and the damage evolution is generally derived from thermodynamics. Hence, various definitions for damage and its evolution law have been proposed to describe the response of different materials to external loading [23-27]. In the micromechanical models, fracture mechanics or discrete damage mechanics (e.g., cohesive zone methods) are usually employed. The global material's response to loading and fracture evolution are studied using a direct detailed discretization of the heterogeneous microstructure for a representative volume element (RVE), assuming periodic repetition of RVEs in a macroscopic volume. In other words, the material's behaviour at the structural level is reduced to that of a single RVE.

In addition to these two types of models, some other approaches such as atomic models [28, 29], particle models [30] etc. have also been developed to describe the damage and fracture evolution. But the current computational facilities limit their application. Thus, in the following sections, the models[26] mainly at macroscale and microscale are reviewed.
2.2 Damage Variables and Effective Properties

The general stress-strain relation in continuum damage mechanics (CDM) is usually represented in the following form:

\[
\sigma = E_e \varepsilon ,
\]

where

\[
E_e = E_0 (1 - D) \quad \text{or} \quad D = 1 - \frac{E_e}{E_0}.
\]

Here \( \sigma \) and \( \varepsilon \) are stress and strain components, \( E_e \) and \( E_0 \) the Young's modulus of the damaged and undamaged material, respectively; \( E_e \) is also called the effective Young's modulus which can be used for macroscopically homogeneous and linear elastic materials with microstructure. The case of damage measure \( D = 0 \) corresponds to the undamaged state, while \( D = 1 \) is equivalent to complete local failure of the material. Actually, the damage measure \( D (0 \leq D \leq 1) \) is usually of non-uniqueness. It can be a scalar function in the simplest case [25], a vector function in a more complex case [31], or be characterized by a tensor of second [26] or higher (even) rank [31]. Further discussions can be seen in Section 2.2.4. Approaches to the problem of the effective modulus (\( E_e \), Poisson's ratio \( \nu_e \) etc.) of solids with non-periodic distributions of pores/voids/defects have been proposed by researchers from various points of view. These approaches can be divided into three distinct groups: analytical models, numerical models and empirical models. The first group studies various mutual positions and orientation statistics of defects, as well as periodic arrangements. But their predictions for the properties are limited by existing analytical solutions of elasticity, which are only available for regular voids shapes—spherical or ellipsoidal in the 3-D analysis, and circular, elliptical or right polygons in 2-D. Numerical simulations can be used for all kinds of shapes, but they require substantial computational power and are not universal. Empirical models are generally created in terms of a mathematical fit to experimental data corresponding to some materials. In the following subsections, these models are further reviewed.

2.2.1 Analytical Models

Many analytical models for determining effective properties of porous materials have been proposed in the past decades. These models were generally developed within the
framework of the small-deformation linear elasticity theory for heterogeneous materials and predicted the effective constitutive response at the macroscopic level from parameters of their microstructural behaviour. Most of the models are reasonably effective in predicting equivalent material properties for relatively simple geometries and for low porosity.

Some of the common analytical models are well known: the self-consistent, differential, Mori–Tanaka, composite sphere and minimum solid area models etc. All of these models basically involve two steps: (1) to establish stress and strain distributions for a simple two-phase geometry (matrix plus inclusions), and then (2) to modify these distributions taking into account interaction between inclusions. With regard to the second step, it can be divided into two categories: non-interacting and interacting models. The non-interacting models are based on assumptions that neighbouring pores/voids do not interact and that the overall effect can be obtained by the summation superposition of effects from the individual pores/voids. These assumptions are accurate only for the cases of low porosity. In the interacting models, the interaction of pores and cracks in porous materials must be considered when one evaluates the material's effective properties. The applicability of the analytical models was compared in [32], using the published experimental data on ceramic materials for a porosity range of 0-40% and on a cellular material with porosity of about 90%. In the remaining part of this section, the basis and validity of the analytical models are reviewed.

- **Self-Consistent Method (SCM)**

In this method, a matrix containing an inclusion of a simple geometry is assumed to have unknown effective moduli. Then, average stress and strain distributions in the matrix and inclusion are determined for an external application of a uniform stress or strain of an arbitrary value. Thus, the effective moduli are established, using these average stress and strain distributions, which, in their turn, are expressed as functions of effective moduli in the SCM.

The SCM for inhomogeneous materials was initially proposed in [33-35] and used to calculate the effective moduli of solids with random cracks by Budiansky and O'Connell [36] for porous solids. In this method, the effective bulk modulus $G_e$ and shear modulus $K_e$ are expressed [34, 35]:

\[
G_e = \frac{1}{2} \left( \frac{1}{1 - 2\nu} + \frac{2\nu}{1 - 2\nu} \right) G_0 + \frac{1}{2} \frac{2\nu}{1 - 2\nu} \frac{c^2}{K_0},
\]

\[
K_e = \frac{1}{2} \left( \frac{1}{1 - 2\nu} + \frac{2\nu}{1 - 2\nu} \right) K_0 + \frac{1}{2} \frac{2\nu}{1 - 2\nu} \frac{c^2}{G_0},
\]

where $G_0$ and $K_0$ are the bulk and shear moduli of the matrix, respectively, and $c$ is the volume fraction of the inclusions.
2-3 \left( \frac{G_e}{G_0} \right)^2 \left[ f_1(p, \nu_0) \left( \frac{G_e}{G_0} \right) + f_2(p, \nu_0) = 0, \right.
\end{equation}

\begin{equation}
\frac{K_e}{K_0} = \frac{1 - p}{1 + f_3(p, \nu_0) \left( \frac{G_e}{G_0} \right) p},
\end{equation}

where $f_1$, $f_2$ and $f_3$ are functions of the Poisson ratio $\nu_0$ and the fractional porosity $p$.

The method was also explored by many other researchers [37-43]. Furthermore, the Generalized Self-Consistent Method (GSCM) [44] for composite materials was proposed and adopted for cracked solids by embedding a crack in a 2D or 3D circular matrix that was embedded in an effective medium [45, 46].

The SCM or GSCM has been used in many research although some analysis [32, 47] indicate that the method seems to over-estimate the effective compliance, particularly for narrow and crack-like holes.

- **Differential Method (DM)**

This method is actually an improved SCM, in which an infinitesimal porosity is introduced in the material treated as a homogeneous medium possessing the unknown effective moduli $M_e$. Then, a differential equation is established in terms of parameter $M_e$, and the solution of this equation can be obtained either in a closed form or by numerically. After the DM was proposed in [48, 49], it has been used to evaluate effective moduli of microcracked solids by some researchers [50-52]. Respective equations, obtained in [51] have the following form:

\begin{equation}
\frac{G_e}{G_0} = (1 - p^2) \left[ f_3(p, \nu_0) + f_2(p, \nu_0) \left( \frac{G_e}{G_0} \right) ^{\frac{1}{3}} \right] = 0,
\end{equation}

\begin{equation}
\frac{K_e}{K_0} = \frac{G_e}{G_0} \left( 1 + f_3(p, \nu_0) \right)^{-1} = 0,
\end{equation}

where $f_1$, $f_2$ and $f_3$ are functions of $\nu_0$ and $p$. $f_3 = \frac{(1 - 5\nu_0) \left( \frac{G_e}{G_0} \right) ^{\frac{3}{2}}}{1 - \nu_0}$. These equations can be solved iteratively and have been shown to yield an exponential type of the empirical equation [53]. A comparative analysis [32] shows that DM and SCM tend to
over-estimate the effective modulus. The accuracy of this method is about 5% when \( \nu_e \) is around 0.3, but when \( \nu_e \) is around 0.15, the error can be as high as 15% [32].

- **Mori-Tanaka Method (MTM)**

  Originally introduced by Mori and Tanaka [54] and applied to porous materials in [55], this method involves complex manipulations of the field variables such as eigenstrain and backstress associated with multiphase geometries of materials. It is assumed in the MTM that, for randomly located cavities, the interaction tractions \( \Delta t \) reflect the average stress environment \( \sigma \) in the solid phase, while a solid with traction-free cavities is represented as a superposition of several solids containing one defect each, as shown in Fig. 2-1. Thus, taking \( \Delta t = n \cdot \sigma \) constitutes the MTM. This method has received a theoretical support from some researchers [56, 57] and also been verified by experimental data [58, 59].

![Figure 2-1 Stress superposition for a solid with cavities or cracks. \( \Delta t \) denotes the interaction tractions (from [47]).](image)

However, the main limitation of the above models is that they cannot provide a unified description of the effective properties of materials with arbitrary microstructures (shapes, sizes and orientation of cavities). The root of the limitation is that the morphology parameters of cavities density are not identified—the moduli are mainly given in terms of porosity. To overcome this drawback, the elastic potentials for a solid with cavities were constructed by Kachanov [47] to deal with the morphology parameters, which depend on shapes of defects. Then, the effective moduli were characterized in terms of not only porosity but also shape factors, for example, "eccentricity" for elliptical holes. The analysis was based on two approximations: the approximation of non-
interacting cavities and the approximation of the average stress field according to MTM. The results from the analysis indicated that MTM appears to be more reasonable if mutual positions of defects are random.

- **Composite Sphere Method (CSM)**

Hashin and his co-workers [60, 61] have introduced CSM in which a composite sphere — a sphere of the matrix material with another spherical phase concentrically placed into it — was postulated. The real geometry of the two-phase material was approximated as an assembly of such composite spheres of different sizes but with the same volume fraction of the second phase in each sphere. For an isolated spherical pore, the equations are expressed as [62]

\[
\frac{M_e}{M_0} = \frac{(1-p^2)}{(1+\beta_mp)} \\
\nu_e = f_m(\nu_0, p),
\]

where \(M\) represents the bulk, shear or Young's modulus, \(\nu\) is the Poisson's ratio, \(p\) is the fractional porosity, the subscript "e" denotes the effective modulus, "0" represents the zero-porosity modulus, and \(\beta_m\) is a function of \(\nu_0\) corresponding to each \(M\) (bulk, shear or Young's modulus).

The accuracy of such types of models is good, with an error margin of about 5%, when the zero-porosity Poisson ratio \(\nu_0\) is around 0.3 [32]. But it was assumed in this method that each composite sphere experiences the same pressure. This assumption is not realistic for porous solids where the second phase (e.g., voids) has a zero modulus.

- **Minimum Solid Area (MSA) Models**

In MSA models [9], the effective properties of porous materials are associated with pore shapes and packing arrangements by correlating these geometric characteristics with the minimum area (load-bearing area) that transfers the load in porous materials. The scheme of MSA concept is given in Fig. 2-2.
For a homogeneous solid (A) the minimum and average solid cross sections are the same; however, the flux or stress transmission in (B) must be dominated by the MSA normal to the direction of the stress or flux (dashed lines delineate the cell structure). (C) and (D) represent the definitions of the MSA for pores between spherical particles (C) and spherical pores (D).

There are two versions of MSA models in general. The first version refers to the foam/honeycomb MSA models and the second version is for bodies consisting of packed uniform spherical particles and resultant pores between the particles [9]. The MSA models can clearly and directly predict the critical porosity value for a given pore model. But further improvements and extensions are needed in aspects of the anisotropy of properties.

- Other methods

In [63], a generalized method of cells (GMC) was used to calculate effective elastic properties and inelastic response of porous materials. This method considers a material that possesses a periodic structure with a repeating RVE, which consists of a number of subcells. Macroscopic average stresses and strains are defined from the corresponding microscopic quantities (homogenisation) on the assumption of continuity of displacements and tractions at the interfaces between the subcells, as well as between the RVEs. Thus, the resulting overall (macroscopic) constitutive equation of the multi-phase media, as well as the effective stiffness tensor can be obtained. It was shown in [63] that the results from the GMC are in good agreement with that from some empirical models for a given pore geometry.

An effective medium theory was used in [64] to obtain a closed-form expression for the dependence of bulk moduli of ceramics on porosity. The theory is based on three
principal aspects: firstly, there is a reference system, usually of an idealized nature, in which all of the relevant mathematics can be performed; secondly, there is a renormalization of a metric (e.g., a length scale) which reflects the transformation of the ideal system into the non-ideal system; thirdly, there is a consistency condition that ensures that the effective medium conforms to a known characteristic of the non-ideal system. The theory is illustrated by applications to data for high-purity alumina that spans a volume fraction of porosity ranging from 0.5% to 90%.

2.2.2 Numerical Models

Numerical models for calculating effective properties are generally implemented by means of a random or digital representation [65] of a heterogeneous material as a network of springs or finite elements. In the random representation, a microstructure of the material is generated based on statistical microstructure modelling while in the digital one, the microstructure is introduced from a captured CT or x-ray image or scanning electron micrograph. In these models, the microstructure is discretized into elements and then directly used for computational solutions of the elasticity equations using FEM [66, 67]. The method uses a variational formulation of the linear elastic equations and the solution is found by minimizing the elastic energy by means of the fast conjugate gradient method [68]. The digital image is assumed to have periodic boundary conditions. Thus, computational results, which can be expressed simply by two- or three-parameter relations, correspond to a particular microstructure and explicitly show the dependence of properties on the nature of porosity. In the numerical models [68, 69], effect of porosity on the Young’s modulus, Poisson’s ratio and thermal conductivity were investigated. But the effect of other microstructural details – size, shape, number etc. of pores/voids – was neglected because they were too complex to be considered in the numerical models. To overcome this problem, Tsukrov and Novak [70] made an analysis using the combination of numerical and analytical techniques: the elasticity problem for each type of defects was solved numerically, and this solution was used in the analytical procedure. It was performed within the framework of linear elasticity, while non-linear effects caused by closing of holes due to compressive stresses (stiffness increasing with compression, etc.) were not covered. Shape, size and porosity were considered in this analysis.

Actually, the numerical models can be regarded as a kind of micromechanical models, which are presented in Section 2.4.
2.2.3 Empirical Models

Empirical or semi-empirical models generally provide a reasonable means of describing data, extrapolating results, and comparing data among materials. However, because they lack a rigorous connection with microstructure, these results offer neither predictive nor interpretive power. The following are some two-parameter empirical or semi-empirical relations introduced in [4, 71]:

\[ M = M_0 (1 - c_2 p), \]  
\[ M = M_0 (1 - c_1 p + c_2 p^2), \]  
\[ M = M_0 (1 - c_1 p)^c, \]  
\[ M = M_0 \exp(-c_2 p), \]  
\[ M = M_0 \exp\left(-\left(c_1 p + c_2 p^2\right)\right) \]  
\[ M = M_0 \frac{1 - p}{1 + c_2 p}, \]  
\[ M = M_0 \left(1 - c_2 p^3\right)^{\frac{2}{3}}, \]

where \( M \) is the modulus at porosity \( p \), and the adjustable parameters are \( M_0 \) (the zero-porosity modulus), \( c_1 \) and \( c_2 \) are widely believed to be dependent on microstructure. The comparison of the models with large sets of experimental data [71] suggested that the exponential fit is better than the linear fit in the approximation of the experimental data whereas the linear relation has the smallest deviations in comparing polycrystalline moduli for the near-zero-porosity. The test of self-consistency indicated that the linear relation provides better agreement with experimental results.

2.2.4 Other Models

There are many different damage models for various types of material damage, microscopic mechanisms and features due to the complex nature of damage. So far, there is no general agreement regarding the definition of damage variables although the classic CDM models presented by Eqs. (2-1) and (2-2) have been used by most researchers. As discussed in [23], selection of a damage variable is largely a matter of taste and convenience and often has no obvious physical basis. Here we review some representative models.
Compared to the classic CDM models, which is deduced in terms of the strain-equivalence principle \[5, 25\], a CDM model based on strain-energy-equivalence principle was proposed by some researchers \[24\]. In this model, the damage variable is defined as \( D = \left( \frac{a}{R} \right)^2 \) for a 2D case or \( D = \left( \frac{a}{R} \right)^3 \) for a 3D case and effective stiffness

\[
\overline{C}_{ij} = C_{ij}(1 - e_{ij}D),
\]

where \( R \) is the radius of an isotropic elastic solid, \( a \) is the half length of an elliptical void in the solid, \( C_{ij} \) are the components of stiffness matrix of the undamaged solid, \( e_{ij} \) are parameters dependent on the Poisson's ratio of the undamaged solid. The advantage of the model is that it accommodates damage associated with general microdefects and models the progress of local damage in a continuum sense.

A displacement-equivalence damage model \[72, 73\] for brittle materials was also proposed. Based on the concept of the effective stress, the virtual undamaged configuration was introduced in the model, and the assumption of displacement equivalence was proposed to correlate the damaged and the virtual undamaged configurations. The damage deactivation criterion in the model, which depends on both the stress and strain states of the materials, was defined for the principal direction as

\[
D_i = \begin{cases} 
D_i & \text{when } \sigma_i \geq 0 \text{ or } \varepsilon_i \geq 0 \\
0 & \text{when } \sigma_i < 0 \text{ and } \varepsilon_i < 0
\end{cases},
\]

while the damage variable \( D_i \) was defined as

\[
D_i = 1 - \frac{A_i}{A_i^e},
\]

where \( A_i \) and \( A_i^e \) is the area and effective area, respectively, for resisting load \( \sigma_i \) corresponding to the principal directions. Then, a constitutive relationship was proposed as

\[
\begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{bmatrix} = \frac{1}{E} \begin{bmatrix}
1 & -\nu \sqrt{(1-D_1)} & -\nu \sqrt{(1-D_1)} \\
-\nu \sqrt{(1-D_2)} & 1 & -\nu \sqrt{(1-D_2)} \\
-\nu \sqrt{(1-D_3)} & -\nu \sqrt{(1-D_3)} & 1
\end{bmatrix} \begin{bmatrix}
\sigma_1^e \\
\sigma_2^e \\
\sigma_3^e
\end{bmatrix},
\]

where effective stress \( \sigma_i^e = \frac{\sigma_i}{1 - D_i} \). Further work and assumptions were performed to deal with the unsymmetric compliance matrix.

In another damage model \[74\], total damage variable \( D \) is decomposed into two parts: damage variable \( D_v \) and \( D_c \) induced by voids and cracks, respectively, shown in Fig. 2-3. It is defined that \( D = 1 - (1 - D_v)(1 - D_c) \).
Chapter 2. Continuum Damage and Fracture Mechanics

2.3 Damage Evolution

Apparently, the damage variables discussed above vary with the change in stress and strain levels that may lead to new damage/cracks in the materials. The evolution rules of the damage are generally obtained by three methods: the experiment-based method, the micromechanical method, and the thermodynamics-based method. The first method includes curve fitting [75] and statistics based or stochastic methods [76]. The experiment-based method is able to offer rational results for the tested cases, but is unable to be generalized to other more complicated cases. As for the second method, the micromechanical derivation of microcrack growth laws is currently achievable only for the case of originally homogenous linear isotropic elastic solids without microcrack interaction [77].

Thermodynamics-based methods can be divided into three categories: the associated method, the non-associated method and the direct method. The associated method [78-80] introduces a damage surface, which defines the reversible domain. The evolution of the damage should be normal to the damage surface (flux rule) and guarantee that the state variables of the material stay on the succeeding damage surface (consistent condition). The associated method can also be deduced equivalently from the principle of maximum dissipation [81], or the principle of minimum free energy [82]. In the non-associated method [25, 77, 83], however, it is not necessary for the flux rule and consistent condition to be derived from the same surface. For example, in [83], the damage growth
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was related to the Von Mises equivalent strain. As for the direct method, it directly utilizes certain principles and relations in thermodynamics, for example, the Onsager relations [84], or specifies a dissipation function in the space of thermodynamics to deduce the evolution rule of damage by a differential relation [27]. The difficulty of the thermodynamics-based method lies in the choice of the dissipation potential and damage surface in the stress space or thermodynamics space. Usually, the experimental observation is resorted for this [21, 25, 85], while certain requirements in thermodynamics are considered, for example, the damage surface should be expressed by a homogeneous function of degree unity.

In the damage theory [27] for isothermal tensile loading, a thermodynamic damage evolution law was derived in the following form:

$$D = D_0 \exp \left( \frac{E_0 \varepsilon_i^2}{2W^*} \right),$$  \hspace{1cm} (2-17)

where initial damage $D_0$ reflects the effect of the initial state of the material, $\varepsilon_i$ is the major principal strain, $W^*$ is the continuum damage energy absorption capacity of the material. It can be seen from Eq. (2-17) that damage develops only if there is some initial damage (i.e., $D_0 > 0$), while its amount depends on ratio of the strain energy $E_0 \varepsilon_i^2$ to $2W^*$. On the basis of the theory [27], the continuum damage and failure evolution in inhomogeneous ceramic materials was given in [21, 86]. The authors analysed the effects of energy dissipation and acoustic emission, the load-carrying capacity, the post-critical response in dependence of the heterogeneity realisations and the size effect of damage inhomogeneity. The numerical procedure to study the local failure and interaction of elements was presented and some very useful parameters of alumina property were obtained for various loading temperatures [21].

In addition, for elastic brittle damage, the nucleation and growth of microscopic cracks caused by elastic deformations in terms of CDM are investigated in [87]. The author discussed the change of the effective stiffness and compliance due to the strength reduction and elastic modulus drop with damage evolution. Also, the general thermodynamically-based theory for a constitutive law and damage evolution of elastic-brittle materials (high strength concrete) was presented in [26]. The theory was based on the Helmholtz free energy as a function of the elastic strain tensor $\varepsilon^e$, the second rank damage tensor $D$, and another scalar damage variable. By establishing a single
dissipation potential, a unified description was possible instead of a separate formulation of constitutive and damage evolution equations.

When an element fails \((D = 1)\) under some loading, it means the presence of a crack induced by the loading and also implies an abrupt reduction of the local stiffness of the element. In some cases \([80, 88, 89]\), the damage measure \(D\) is taken as a number very close to 1, e.g., 0.99, so that the constitutive stiffness/compliance matrix based on CDM keeps definitely positive in the numerical calculations. In other cases, it is related to spacing of the crack \([90]\) or a model parameter \([72]\), which can be determined in terms of micromechanical analysis discussed in the following sections.

### 2.4 Fracture and Micromechanical Models

#### 2.4.1 Fundamentals of Fracture Mechanics

In FM, the geometry and location of the growth of one or more macrocracks is explicitly represented. A crack propagation through a solid with a homogeneous microstructure under tensile stress field is represented by an unstable growth of its length. In the simplest case, when linear fracture mechanics is used, the crack is assumed to be surrounded by a linear homogeneous and isotropic elastic solid and a corresponding failure mode is perfectly brittle. Thus, fracture starting from flaws or defects in materials can be described using the methods of continuum mechanics. The basic assumption is the description of the flaws as two-dimensional defects with a sharp tip, which are called cracks. Whereas three-dimensional flaws such as pores have a finite radius, the idealized cracks have a radius of zero at the tip. Two equivalent approaches exist to describe the mechanics of crack extension: the energy approach developed by Griffith \([91]\) and the stress approach developed by Irwin \([92]\), shown in the following equations, respectively.

\[
G_1 = \frac{K_1^2}{E},
\]

\[
\sigma_{ij} = \frac{K_1}{\sqrt{2\pi r}} f_y(\theta),
\]

where \(G\) is called "the energy release rate", \(K\) is called "the stress intensity factor", the subscript 1 represents loading mode 1, \(E\) is the Young's modulus, \(f_y(\theta)\) are known
angular functions, $\sigma_{ij}$ are stresses near the crack tip and $r$ is the distance from the crack tip.

The stress intensity factor $K_I$ indexes the crack tip stress field singularity by delineating the magnitude of the stress field at a crack tip where it occurs. $K_I$ differs for different crack and body configurations, structural constraints, and loading conditions, and thus needs to be evaluated for each specific situation.

### 2.4.2 Statistical Models of Fracture Mechanics – Weibull Theory

It has been known that brittle fracture is caused by natural flaws such as inclusions, pores, damaged grain boundaries, etc. The fracture strength, i.e., the maximum stress that a material can withstand, varies unpredictably from component to component even if a set of nominally identical specimens is tested under the same conditions. This is due to the effect of the natural flaws, which are randomly distributed in the component and determine the tolerable load. To account for the random nature of failure, Weibull [4, 93] proposed the strength distribution function, correlating the fracture strength $\sigma_c$ and maximum crack size based on a weakest-link theory:

$$F(\sigma_c) = 1 - \exp\left(-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right),$$

(2-20)

where $\sigma_0 = \frac{K_{lc}}{Y}\sqrt{a_c N/m}$, $K_{lc}$ is the critical stress intensity factor – fracture toughness, $Y$ is a geometric function dependent on the crack size normalized to characteristic dimension of the material, $N$ is the average number of flaws in the material, $m$ is called "Weibull parameter" dependent on the material and also the size of the component. In [94], the correlation between the parameters $\sigma_c$, $\sigma_0$ and $m$ was discussed.

The critical value $K_{lc}$ depends on the crack tip constraint and specimen thickness and geometry. Once $K_{lc}$ has been determined, either the fracture strength can be predicted for the given crack length, or if the stress at failure is known then the critical crack length can be estimated. Alternatively, knowledge of the fracture strength and the assessment of the crack shape and size permit an estimation of the fracture toughness [95]. It is generally assumed that fracture toughness is a materials property independent of crack geometry. However, it is frequently observed that fracture toughness can be a function of
the crack length. In such cases the material is said to exhibit the R-curve behaviour. Such behaviour is generated by microstructural features, which exert closure forces on the growing crack.

In the corresponding mathematical models based on the Weibull theory, the following basic assumptions are used: (i) a strength value can be attributed to each flaw and the value depends on the size of the flaw; (ii) the strength $\sigma$ is a random variable with a probability distribution function $F(\sigma)$ (ref. Eq. (2-20)); (iii) alternatively, the size $a$ of a flaw can be considered as a random variable with a probability distribution $F(a)$; (iv) interaction effects among random variables can be neglected; (v) the worst flaw determines the maximum tolerable load; (vi) the number of flaws per component is a Poisson distributed random variable. Further discussion about the assumptions can be seen in Section 3.2.1.

The statistical Weibull fracture theory has acquired success in many engineering areas. It examines the "weakest link" in a statistical manner in the material volume under consideration. However, it is too difficult mathematically to be applied to complicated parts or structures. Especially, failures can occur in structures that have no visible initial cracks. This necessitates studies of the evolution of internal microscopic damage before the formation of the visible macroscopic defect. Therefore it was highly appreciated by the scientific community when Kachanov [22] published a simple model of material damage which subsequently could be extended to brittle elastic, plastic or viscous materials under conditions of uniaxial or multi-axial, quasi static or cyclic loadings, so that it may be considered as nearly universal.

2.4.3 Micromechanical Models

As reviewed in the above sections, the growth of the macrocrack through a solid with a heterogeneous microstructure may be studied using FM approaches, while continuum damage accumulation prior to the macrocrack may be determined using CDM approaches. The relation between the two approaches is actually a question of different characteristic sizes of microcracks and macrocracks (see Fig. 2-4). It also means that a question of scale refers not to the size of the crack considered but to the medium surrounding it [96]. The classical characteristic parameters used in the approaches, such as stress intensity factor $K$ or energy release rate $G$, are based on the local theory. However, in order to analyse the global behaviour of materials, as well as interaction
between the cracks, non-local approaches are usually required, although a so-called local approach to fracture based on CDM and FEM is used as a practical tool for coupled elastic-brittle damage fracture analysis [97]. Thus, micromechanical models have been proposed to introduce and develop FM or/and CDM from the macroscale to microscale.

![Diagram](image)

**Figure 2-4 Microcrack growth, single macrocrack initiation and propagation in a crystalline material (from [15]).**

Instead of proceeding phenomenologically at the macroscopic scale, the principles and tools of micromechanics are applied to phenomena occurring at the microscale. The effective constitutive relationship is usually derived by means of FM or/and CDM approaches. In other words, details of the material's microstructure (micro crack/voids evolution, coalescence etc.) are either directly described using the FM method or in terms of the internal damage variable at the microscale. The micromechanical models generally provide an explicit account for arbitrary microstructural morphologies and microscopic fracture patterns, making it easier to identify and design microstructural configurations. The advantages of the models also include explicit modelling of fracture in a non-constrained manner and direct analysis of the stochastic nature of fracture in heterogeneous microstructures. Therefore, the crack initiation, growth, and coalescence
are a natural outcome of the material's response, applied loading, and boundary constrains.

A direct idea of analysing the effect of deficiencies is to discretize the material at the microscale and then to use the finite element method. But the problem is that firstly, geometry modelling of the material is very difficult because such parameters as the number, size, location etc. of defects are randomly distributed in the material; secondly, and this is the most important point, such computations at the microscale require huge amounts of data that current computers are not capable enough to deal with. Thus, to study the transition between the microscale and effective material properties at the macroscale, a RVE is generally introduced in micromechanical models. It is important that the size of RVE is large enough to include a sufficient number of microvoids or microcracks but, at the same time, it must be small enough for the stress and strain state to be considered as homogeneous or with a small inhomogeneity allowed. Further details of representation of the RVE has been reviewed in [90, 98].

Accordingly, the first step in micromechanical models is representation of the microstructure of the RVE in a realistic manner, while the other steps are a choice of the numerical techniques to solve the boundary-value problem and identification of the constitutive equations for the constituents.

Generally, there are two main types of methods to model the microstructure of a RVE: the digital image-based methods and the random methods. In methods of the former type, cross-sectional images are captured by some devices, e.g., CCD camera or micro-CT scanner etc., and then processed by means of numerical methods [65, 99, 100]. An example of the images is shown in Fig. 2-5, the dimension of which is 250×250 μm and each microcrack is modelled explicitly as an evolving internal boundary. In the random methods (see Fig. 2-6(b)), the size, number and location of voids/pores/inclusions in a RVE are generated in terms of some statistical distributions, which are compatible with information obtained from 2D observations: porosity, morphology etc.
Chapter 2. Continuum Damage and Fracture Mechanics

Figure 2-5 The digital image-based microstructure modelling (from [99]).

Figure 2-6 Creating extended microstructure (a) a mesh of macroscopic elements with an underlying microstructure of repeated RVEs; (b) the extended microstructure by Voronoi tessellation (from [17]).

To solve the boundary-value problem, the conventional FEM, as in Fig. 2-5(b), is generally employed. Some other methods, e.g., the unit cell method [101, 102], Voronoi tessellation method [103, 104], have also been developed for microscopic stress/strain analysis. In these methods, constitutive equations describing the local behaviour of each constituent in the heterogeneous material are the weakest point in the computation of microstructures. The reason is that the local properties of one phase may differ
significantly from that determined in the bulk material. To overcome the problem, the homogenisation theory is developed based on continuum mechanics and mathematics and generally applied in the analysis. In this theory, the heterogeneous RVE is homogenized by an equivalent material element under the two assumptions. One is that the microstructures are periodically arrayed in the structure globally, and the other is that the macroscopic fields are uniform.

To account for arbitrary microstructural morphologies, a mesh generator was developed in [105] to create polygons based on Dirichlet tessellation, which discretizes the domain into a network of multi-sided convex Voronoi polygons containing one inclusion at most. These polygons can serve as the elements in the finite element analysis of porous materials. The stiffness matrices of polygonal elements are derived by assuming compatible displacement fields along inter-element boundaries and a stress distribution in the interior of each element. This is appropriate for \( n \)-sided polygonal elements having a varying number of nodes since interpolation of displacement field is only needed along the element boundary. The element formulation is based on the principle of minimum complementary energy and the details of this formulation may be found in [18]. This tessellation method was employed to analyse a microstructural response of materials with arbitrary distribution, shapes and sizes of heterogeneities [17, 19, 106].

The above methods account for the initiation of cracks, their coalescence etc. But it is difficult using them to study damage-induced anisotropy and they cannot describe adequately the growth of a dominant crack leading to macroscopic failure since it is not suitable to homogenisation. To overcome these limitations, models based on a discrete approach were developed, e.g., lattice cell models [107], cohesive zone models, etc. Especially, the latter has been accepted in many publications to analyse crack initiation and propagation.

In cohesive-zone models, special cohesive interface elements are used in simulations and the fracture characteristics of the material are accounted for by a cohesive surface traction - displacement relation. Degrading mechanisms in front of an actual crack tip are lumped into a discrete line or plane - the cohesive element. In a finite element analysis, cohesive elements are introduced at the boundary between volumetric elements either from the beginning of the analysis [104, 106, 108, 109] or after the corresponding interface is predicted to start failing [110]. These models have been used successfully,
especially in cases that the crack path is known in advance. By inserting interface elements between continuum elements along the potential crack path, a cohesive crack can be modelled exactly. However, crack initiation and propagation in brittle porous materials tend to be random due to the effect of microstructure [19, 104]. More details of the cohesive-zone models including their advantages and limitations have been addressed by some authors [111-113]

2.5 Multiscale Analyses

It is known that in general structural stress analysis or fracture mechanics, the finite element method is applied extensively to study the behaviour of various materials. In these studies, the materials are generally considered to be homogeneous. However, for heterogeneous materials, in which there generally exist various defects like cracks, voids, pores or inclusions at the microscale, general analyses at the macroscale are not enough to describe the effect of such defects on the material’s behaviour. Thus, models that can span multiple length scales are inevitably required to study the correlation between the microstructure and the macroscopic properties.

Since the applied thermal or mechanical loads are at the structural level, the models should be capable of correlating the microstructural response with the overall macroscopic behaviour. Different to conventional local models at a single scale, multiscale models are generally gradient or nonlocal and assume that the stress at a material point is defined not only by the strain at the point but also by its spatial derivatives (the gradient models) or by the strains in a finite neighbourhood of that point (nonlocal models [83, 114]).

For instance, some researchers [17, 18] suggested a multi-level computational model for multi-scale damage analysis in composite and porous materials. In the model, the microstructural analysis was conducted with the Voronoi cell finite element method [18], while a conventional displacement-based FEM code executed the macroscopic analysis (as shown in Fig. 2-6) and the detail analysis procedure was presented. Coupling between the scales in regions of the periodic microstructure was accomplished through asymptotic homogenisation.

In [102], a three-scale model was proposed with the consideration of microstructure, macrostructure and the fracture origin such as an interface or a crack. This model combined the asymptotic homogenisation method [18, 115] and the finite element mesh
superposition method [116, 117]. The overall behaviour of material was analysed by means of the homogenisation of the heterogeneity expressed by the unit cell model, while the fracture origin (at the middle scale) was modelled directly with the microscopic heterogeneity by another microscopic mesh, which was superposed onto the macroscopic mesh. The most advantageous feature of this model is that it can analyse the non-periodic microscopic stress at the crack tip under a non-uniform macroscopic strain field with a high gradient. Some similar models, based on FM [118] or combination of CDM and FM [89], have also been proposed to study crack propagation by using the mesh superposition method.

Other methods [119, 120], based on a statistical approach, achieved bridging between micromechanical and continuum models where the microstructural material's randomness was considered below the level of a single finite element. Likewise, a finite element-based Monte Carlo method that can be used to predict the scatter in the nominal elastic constants and fracture of thin films was developed in [121].

Despite all these advances in the area of micromechanics, bridging between micro and macro scale still remains one of the most challenging goals. Although in some instances comparison with experimental findings and microscopy studies have been done [19], the majority of the contributors omit comparison and correlation with experimental data.

### 2.6 Summary

Analysis of the current research into damage and fracture evolution in brittle materials indicates that there is no general agreement regarding the definition of damage variables due to its complex nature. To some degree, definition of the damage variable is largely a matter of taste and convenience and often has no obvious physical basis. But such a definition is necessary in CDM so that it can reflect the effect of presence and evolution of a large number of randomly distributed microcracks of irregular shapes that are random in size and orientation on the response, failure and reliability of the embedding material. Thus, damage can be directly defined in terms of variables that can represent the relative decrease in material properties, e.g., elastic modulus or thermal conductivity; it can also be indirectly represented by microcrack parameters (crack size, number etc.) or variation of stress/strain.
Two major approaches — phenomenological and micromechanical— have been developed to study damage and fracture evolution over the last decades. The pure phenomenological definitions are consummated in theories, but it is not easy to provide an explicit account for arbitrary microstructural morphologies and fracture patterns. Although the micromechanical definitions could be regarded as the accurate ones and are easy to explain some complicated phenomena associated with microcracking evolution, usually they are too complicated for practical use. A purely micromechanical theory may never replace a properly formulated macroscopic phenomenological theory as a design tool. Thus, quasi-phenomenological or quasi-micromechanical models that combine the concepts and methods of the two kinds of conventional approaches seem promising to construct an applicable damage constitutive theory with a firm physical basis.

In order to provide a powerful tool in understanding mechanisms that lead to macroscopic failure and to refine theories of damage utilized in continuum descriptions, or continuum/discrete models, development of multiscale models is necessary and expected. Some models have been proposed and the results have also presented some agreement with experiment. But, further efforts are still necessary.
Chapter 3. Damage and Fracture Evolution in Ceramic Coatings

This chapter reviews current research results on damage and fracture evolution in ceramic coatings. It firstly overviews the points that need to be studied in this area. Then, plasma spraying process, the resulting microstructure and its effects on elastic and thermal properties of the plasma sprayed ceramic coatings are reviewed. Next, modelling of damage and fracture of ceramics and coatings is briefly reviewed. Current advances including numerical and experimental studies on loading behaviours of the ceramic coatings are introduced in later section. Finally, this chapter is summarized and some ideas, which are applied in our simulations described in later chapters, are proposed based on the above reviews.

3.1 Introduction

Ceramics have been extensively used as coatings on metallic substrates due to their unique thermal, mechanical, chemical and electrical properties for heat, corrosion, and wear protection. However, ceramics usually suffer from low fracture resistance, which may lead to failure of coatings under loading and have been the principal problem encountered with ceramic coatings. It has been known that the fracture behaviour and effective properties of the coatings are closely related to their microstructure. But, so far, there has been no standard answer for the effect of microstructure on damage and fracture evolution in the coatings. Further research on the effect is accordingly important and necessary.

Microstructure of ceramics is generally characterized by porosity, shape, size, number etc. of voids, pores or grains in the coatings. Statistical distribution of the factors must be considered due to their random nature. The effect of microstructure on the properties of materials are investigated both by experimentalists and from the point of view of theoretical modelling, which are usually based on the empirical, analytical or numerical methods discussed in Chapter 2. To be noted is that there are large differences between the effective properties of plasma sprayed ceramic coatings and that of the comparable bulk ceramics. This is due to a unique splats-based microstructure of the
coatings and it is dependent on thermal spraying technology, which is gaining increasing application in industry for high performance coatings.

Besides stresses induced by external loading such as a bending moment or a thermal shock etc., stresses mainly induced by elastic or thermal mismatch are major sources of damage and fracture of ceramic coatings. The mismatch stresses are present in most surface coatings and may promote debonding and spallation of the coatings. This becomes increasingly likely as the thickness of the coating is increased, since the release of stored elastic strain energy as the stresses become relaxed can drive this debonding and the quantity of energy released, per unit area of interface, normally rises more or less linearly with coating thickness. For such a case, numerical calculations combined with microstructural analysis must be employed to study the damage and fracture behaviours of the coatings.

Generally speaking, the research on the damage and fracture of ceramic coatings involves microstructure characterisation, determination of effective properties and analyses for stress/strain and damage and fracture behaviour of the coatings under various loadings. In the following sections, the detail of the points is introduced.

3.2 Characterisation of Ceramic Coating Microstructure

3.2.1 Methods to Characterise Microstructure of Ceramic Materials

A number of publications [4, 9] have investigated ceramic microstructure and its dependence on the ceramic properties. In the simplest case, microstructure of ceramics can be only characterized by the parameter – volume fraction porosity. However, with the development of micromechanics, other factors are required to describe the microstructure so that heterogeneity of the ceramics can be determined. Table 3-1 outlines some factors of microstructure characterisation to accompany suitable property measurements.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Characterisation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>Volume fraction or density</td>
</tr>
<tr>
<td>Amount</td>
<td>Average value; statistical variation of porosity spatial distribution</td>
</tr>
<tr>
<td>Size</td>
<td>Average and approximate range; size distribution</td>
</tr>
</tbody>
</table>
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<table>
<thead>
<tr>
<th>Geometry</th>
<th>Shape, spacing, orientation and their distribution of microdefects – voids/pores/cracks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Related microstructure</td>
<td>Average size, shape, orientation of grains and their distributions</td>
</tr>
</tbody>
</table>

In most cases, the microdefects are clearly observable by microscopy and the porosity can be obtained by stereological measurements, since the area fraction occupied by the microdefects on a random cross-section of a sample is also a volume fraction. The microdefects in most simulations is generally idealized as circular, elliptical or slit voids although they actually have irregular shapes. Some other shaped voids are also used in some publications. For instance, cross, cube or cylinder shaped pores are employed in [63] and hexagonal pores are assumed in [10]. Especially in [47], various shapes (see Fig. 3-1) of voids including mapping functions of each shape are summarized. As for the grain shapes, they are generally modelled using Voronoi tessellation [122].

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{3} \zeta^2\right) \quad \quad h_1 = 7/2, \quad h_2 = h_3 + h_2
\]

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{2} \zeta^2\right) \quad \quad h_1 = 17/8, \quad h_2 = 1/8, \quad h_3 = 9/2
\]

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{3} \zeta^3\right) \quad \quad h_1 = 153/14 = 10.71, \quad h_2 = 0.344, \quad h_3 = 2.660
\]

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{5} \zeta^3\right) \quad \quad h_1 = 1887/176 = 10.65, \quad h_2 = 0.521, \quad h_3 = h_1 + h_2
\]

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{6} \zeta^3\right) \quad \quad h_1 = 335/154 = 1.526, \quad h_2 = 0.344, \quad h_3 = 2.618
\]

\[
z = R\left(\frac{1}{\zeta} + \frac{1}{4} \zeta^4\right) \quad \quad h_1 = 99/42 = 2.357, \quad h_2 = 0.690, \quad h_3 = h_1 + h_2
\]

Figure 3-1 Various void shapes, corresponding mapping functions \(z(\zeta)\) and shape factors \(h_i\) (from [47]).
The amount of the microdefects in a material sample is essentially probabilistic. The actual number \( (M) \) of the microdefects in the material may be larger or smaller than an average value \( N \). Thus, the probability of the sample containing exactly \( M \) defects is generally given \([4, 122, 123]\) by the Poisson distribution

\[
P(M) = \frac{N^M \exp(-N)}{M!},
\]

(3-1)

where \( N = n_v V \) or \( N = n_a A \); the sample has the volume \( V \) or the area \( A \); \( n_v \) is the voids density which is defined as the number of microdefects in a unit volume or unit area of the material sample. In general, the void density strongly decreases with the increase of the void size \( a \).

In terms of the Poisson distribution, some authors \([4]\) determined the cumulative distribution of the maximum size \( a \) of the voids in the material as

\[
H(a) = \exp\left(-N \left(1 - F(a)\right)\right).
\]

(3-2)

where \( F(a) \) is the probability of the \( M \) voids that have size \( a \) in the \( M \) voids. The relation between the size and material strength then results in the strength distribution – Weibull distribution, which is discussed in Section 2.4.2. The frequency distribution \( F(a) \) was approximated in \([124]\) by a simple inverse power law

\[
F(a) = n_0 \left(\frac{a}{a_0}\right)^{-\sigma},
\]

(3-3)

where \( n_0 \), \( a_0 \), and \( \sigma \) are material parameters. An observation \([125]\) from optical microscopy shows that the size \( a \) of most pores in high-quality alumina ceramics is less than 25 \( \mu m \), which can be seen from Fig. 3-2. Another research \([126]\) also shows that flaw size in ceramics with low porosity is about 20 \( \mu m \) (see Fig. 3-3). These results are similar to those observed in alumina coatings discussed in the following sections, while there are larger differences between the elastic properties of the coating and the comparable bulk materials. This suggests that attributes such as number, porosity, shape etc. of microvoids in materials may have a larger effect on their properties.
3.2.2 Plasma Spraying Process and the Resulting Microstructure of Ceramic Coatings

There are various deposition processes, e.g., thermal spraying, physical vapour deposition (PVD) etc., which involve coating a specimen/component with a layer of material to meet the requirements of specific service environments. Thermal plasma spraying is one of the most common processes for high performance coatings and has been increasingly applied in industry [127]. This process involves feeding a material powder (e.g., ceramics) mixed with a carrier gas/fluid into a high voltage electric arc. The gas forms an expanding plasma, which both heats the powder particles to a molten or semi-molten state and accelerates them towards a substrate (typically metal). The molten droplets are deposited by impact at high temperature and velocity on a cooler substrate and are rapidly cooled. Such a process can be divided into flame, electric arc, plasma arc and detonation gun techniques. In spray fusing, the coating is heated after deposition (usually by a torch) to fuse the material into a dense alloyed structure and produce a diffusion bond to the substrate. In industry [127], the process can be automated and accurately controlled, with robot manipulation of the gun, rotation of the component being sprayed, and computer control of the spray parameters. Layer thickness is built up by moving the spraying apparatus (plasma-gun) transversely across the substrate so successive splats can be deposited and cooled.
Compared to the microstructure of bulk ceramics, plasma sprayed TBC layers generally exhibit a more complex microstructure (see Fig. 3-4) composed of splats and various types of voids \([10, 128-130]\) that may be divided into interlamellar and translamellar. The splats are discontinuously bonded to each other forming very narrow, slitlike inter-splat voids. Other types of more spherical inter and intra-splat voids are also visible. These voids are ubiquitous, especially in ceramic deposits, and reside either within a single splat or over several splats (see Fig. 3-4(b)).

Figure 3-4 (a) Schematic of microstructure of a plasma-sprayed ceramic coating; directions of elastic moduli along spray direction \(E_L\) and transverse direction \(E_T\) (from \([10]\)); (b) A cross-sectional micrograph of the type of coating (from \([130]\)).
Different deposition techniques of coatings may lead to considerably different microstructures in them. For example, unlike the laminar microstructure obtained in plasma sprayed coatings (Fig. 3-5(a)), the TBC layers produced by electron beam PVD (Fig. 3-5(b)) have a columnar microstructure with elongated intercolumnar pores that become predominantly aligned perpendicular to the plane of the coating as its thickness increases. A finer distribution of intracolumnar pores also exists. These differences should be considered during modelling microstructures of the coatings. In the following sections, microstructures of plasma sprayed coatings are mainly studied.

Figure 3-5 Schematic illustrations of microstructures of (a) plasma sprayed ceramic coating showing its disc-like pores aligned parallel to the substrate surface and (b) electron beam PVD coating with elongated pores aligned perpendicular to the substrate surface (from [131]).

Using stereological image analysis (IA) technique, some researchers [130] have determined the void size-shape distribution of thermally sprayed coatings. The shape of
voids was assumed to be an oblate spheroid, length of major and minor axis of which was \(a\) and \(b\). Correspondingly, a shape factor was defined as: 
\[
\text{shape factor} = 1 - \left(\frac{b}{a}\right)^2.
\]
In their results, about 90% of voids were observed within 0~25 \(\mu\)m. The shape of voids was generally very distinct; however, 0~3 \(\mu\)m voids existed in the entire range of the shape factor. The size of voids that have shape factors <0.9 was normally <3 \(\mu\)m. The small and spherical voids were most likely closed pores within individual splats. Fig.3-6 shows a bivariate size-shape distribution of voids per unit area in the ceramic coating. In addition, a typical pore size distribution (see Fig. 3-7, measured by a test) for a dense thermal sprayed coating were introduced in [132]. To be noted is that those small pores (less than 0.5 \(\mu\)m) cannot be seen by optical microscopy, but they also make significant contribution on the overall porosity.

![Figure 3-6 Bivariate size-shape distribution of voids per unit area in a plasma sprayed ceramic coating (from [130]).](image-url)
Chapter 3. Damage and Fracture Evolution in Ceramic Coatings

3.2.3 Effective Elastic and Thermal Properties – Effect of Microstructure

It has been known that the influence of microstructure on the overall behaviour of porous materials depends on the morphological characteristics such as porosity, size, shape, orientation and spatial distribution of micro voids or cracks in the materials. Based on the analytical, numerical or empirical methods discussed in Section 2.2, this problem has been addressed by a number of authors to determine effective elastic or thermal properties of ceramics with inhomogeneities. In [64], this problem was considered in an analysis for the porosity dependence of the bulk modulus of high-purity alumina, which spans a volume fraction of porosity ranging from 0.5% to 90%. In [68, 133], significant effect of porosity on Young's modulus and effect of pore shape on Poisson's ratio in porous ceramics were investigated. Further results on the effects of both porosity and pore shape were also obtained in [63]. These analyses were generally limited to the isotropic case of randomly oriented spheroids, under the additional assumption that all the spheroids have identical shapes. In another analysis [134] for the Hertzian contact damage in porous alumina ceramics, it was indicated that the influence of porosity on the indentation stress–strain behaviour and contact damage response have serious

![Figure 3-7 Pore size distribution for a dense plasma sprayed coating (from [132]).](image-url)
implications concerning the capacity of ceramics to sustain mechanical damage and to absorb energy in impacts. The results show that the performance of ceramic-based structures may be controlled by the level of porosity, and hence by processing strategies. Such is the case of porous coatings used for thermal barriers. In addition, effect of grain morphology on stiffness and strength of ceramics was also explored in [135].

However, for plasma sprayed ceramic coatings, their effective properties can be more complex and different from those of corresponding conventionally sintered ceramics. For example, Young’s moduli of plasma sprayed ceramic coatings are known [10, 129] to be much lower than that of comparable bulk materials. This is obviously a consequence of the unique splat-based microstructure that includes highly non-spherical pores with a high aspect ratio. Due to such a microstructure, the coatings generally exhibit anisotropic or transversely isotropic properties. This was confirmed in an experimental study [129] for elastic stiffness of plasma sprayed alumina coating. The results indicate that there is no significant difference between stiffness $C_{22}$ and $C_{33}$ in transverse directions (see Fig. 3-4) while the stiffness in spray direction $C_{11}=130$ GPa and $C_{22}=C_{33}=90$ GPa, compared to the stiffness of bulk $\alpha$-alumina (about 380 GPa). In the case of effect of voids, the fundamental contribution is due to the authors [136] who derived the effective elastic constants of plasma sprayed coatings, under the assumption of unidirectionally aligned and two-dimensional randomly distributed voids in coatings. Their results show that the effective elastic constants (Young's modulus, shear modulus, bulk modulus and Poisson's ratio) of the coatings exhibited a large variation with void concentration, crack density parameter and void aspect ratio. Further results for the effect of voids in plasma sprayed ceramic coatings were analysed in the literature [10] which used an analytical method introducing statistical distributions [130] of pore sizes and shapes. The analysis also revealed that nearly consistent moduli could be obtained for RVE sizes larger than 150 $\mu$m x 150 $\mu$m, which is about an order of magnitude greater than the sizes of larger pores. In terms of microstructural parameters and analytical methods, other researchers [137] also calculated the transversely isotropic elastic moduli of plasma-sprayed coatings. In the calculation, the dominant features of the porous space were identified as strongly oblate pores, which tend to be either parallel or normal to the substrate. In addition, some authors [138] studied the elastic behaviour of plasma sprayed materials under compressive stresses within the framework of continuum mechanics, more specifically,
the anisotropic nonlinear theory of elasticity. They concluded that Young's moduli of the materials increased several times at small deformations between 0 and -1%.

On the other hand, an analytical method was proposed in [139] to analyse transversely isotropic effective thermal conductivity of a material with inhomogeneities. This method was improved and employed in [11] to analyse both effective thermal conductivity and elastic properties of plasma sprayed ceramic coatings, assuming strongly crack-like pores. Similar work was also done by the authors [140] who calculated both thermal conductivity and elastic properties of plasma sprayed ceramic coatings using two numerical methods. They concluded that porous microstructure causes considerable reduction of effective thermal conductivity and modulus in both the spray and transverse directions. The reduction increases with the total porosity. Generally, the pores account for 40–75% of the reduction in the effective properties. Their analysis [140] also elucidated that the relative effects of pores on the thermal conductivity and the elastic modulus are not equivalent. With an identical porous model, a larger reduction was observed in the modulus than that in the conductivity.

3.2.4 Experimental Determination of Properties of Ceramic Coatings

Various material properties including thermal expansion, elastic modulus, Poisson's ratio, fracture toughness, thermal conductivity etc. of high purity bulk alumina were evaluated in [141] as a function of temperature ranging from 20°C to 1800°C. This evaluation was based on a comprehensive set of material property data from published physical, mechanical, and thermal properties of alumina specimens that conform to the constraints of a particular specification of sintered alumina. These properties can serve as a reference to compare with those of alumina coatings.

An evaluation [142] for the elastic anisotropy of plasma-sprayed ceramics by ultrasonic measurements indicate that the anisotropy ratio $C_{22,33}/C_{11}$ of the as-sprayed materials, e.g., alumina, ranges from 1.8 to 3.6 depending on the material deposition process. To measure elastic properties of ceramic coatings, indentation techniques are usually employed. For example, the researchers [143] measured elastic modulus ($E_t$) of a plasma sprayed alumina coating (porosity~9%) in the out-of-plane direction by nanoindentation tests and obtained a value 180 GPa. This value was considered to be the elastic modulus of a single splat because the depth (100–500 nm) of the nanoindenter penetration was assumed to be insufficient to include the modulus reduction by pores.
Compared to that value, another value 80 GPa was obtained as the out-of-plane elastic modulus of plasma sprayed alumina (porosity~8%) in a Hertzian indentation test [8], in which effect of pores was included due to a larger depth of the indentation. Another indentation results from [144] show that the elastic modulus of plasma sprayed alumina coating in spray direction decreases with the increase of the indentation depth owing to porosity and pore morphology. Some authors [145] also measured the elastic modulus and hardness of plasma sprayed ceramic coatings by indentation as functions of thermal cycles. In addition, the authors [146] developed relationships to determine in-plane elastic modulus $E_T$ of general coating layers by two experimental techniques: static bend test and dynamic resonance. These techniques can be also employed to evaluate in-plane properties of plasma sprayed coatings.

### 3.3 Brittle Fracture in Ceramic Coatings

#### 3.3.1 Stresses in Ceramic Coatings

A considerable elastic or thermal expansion mismatch for ceramic coatings and metallic substrates results in generation of mismatch stresses in such systems, even under purely thermal loading in the absence of mechanical loads. The stresses can be of considerable significance, since they may influence characteristics such as the resistance of the coating to heat, wear and fatigue crack propagation. Transient thermal stresses induced by thermal shock associated with fuel ignition in an engine or a turbine, for example, are superimposed onto the mismatch stresses. The combined stresses can cause cracks initiation and propagation and eventually lead to local failures of coatings. The damage and fracture development due to thermal stresses has been one of the principal problems encountered with ceramic thermal barrier coatings (TBCs).

The stress level in thin coatings or films, which are usually deposited by vapour deposition techniques and typically have thicknesses less than 10 μm, is essentially considered [147] to be uniform throughout the film, and equal biaxial in the plane of the film. The substrate bonded to such a coating is assumed to be much thicker than the coating and, thus, is stress-free. When such a coating specimen is under thermal loading, e.g., the temperature is changed from $T_0$ to a different level $T$. The thick substrate acquires a thermal strain, but remains stress-free. The coating also acquires a thermal strain $\varepsilon_T$, which differs from that of the substrate by
\[ \varepsilon_T = \int_0^T (\alpha_c - \alpha_s) dT \]  
\hspace{1cm} (3-4)

where \( \alpha_c \) and \( \alpha_s \) are the thermal expansion coefficients of the coating and the substrate, respectively. When the coating and the substrate are well bonded, the net in-plane strain in the coating must be the same as the thermal strain of the substrate. Consequently, the mismatch strain needs to be accommodated by elastic and inelastic deformation in the film. If the film remains elastic during the temperature change, this mismatch strain induces a biaxial stress in the plane of the coating, \( \sigma_T \), given by

\[ \sigma_T = \frac{E_c \varepsilon_T}{1-\nu_c} \]  
\hspace{1cm} (3-5)

where \( E_c \) is Young’s modulus and \( \nu_c \) is Poisson’s ratio of the coating.

Based on the Eqs. (3-4) and (3-5) as well as failure criteria of fracture mechanics, Evans and Hutchinson [148, 149] investigated the thermomechanical integrity of thin films and measurement methods of the residual mismatch stress in the film, as well as various modes of the film cracking.

However, the stress level in thicker coatings, which are commonly formed by much faster processes such as thermal spraying and typically have thicknesses in the range of 100 to 1000 \( \mu m \), varies significantly through the thickness. For such a case, Eqs. (3-4) and (3-5) are not fully practicable. Especially, additional stress can be generated, for example, by a bending moment applied to the coating-substrate composite. Indentation and scratching are other means to generate stress; however, the stress field so generated is complicated and the total stress in the coating is the sum of the thermal stress and the applied stress. Thus, numerical calculations combined with microstructural analysis and statistical description must be employed to determine such a stress field and the damage and fracture induced by the stress in the thicker coatings.

3.3.2 Characterisation of Damage and Fracture in Ceramic Coatings

In ceramic coatings, the stresses induced by thermal or mechanical loading or both of them may lead to damage and fracture in the materials. Such phenomena, either in bulk ceramics or in ceramic coatings, have been characterized by many researchers using the parameters based on various failure criteria. In most papers, the criteria of fracture mechanics are used and thus, the research focus on determining the fracture mechanics parameters. For instance, some authors [150-153] determined fracture toughness or
strength with consideration of effect of grain size or morphology on fracture strength of alumina ceramics, as well as their R-curve behaviour. Some authors [154] studied thermal shock behaviour of alumina by calculating the critical stress intensity factor. Yanaka etc. [155] studied cracking phenomena of brittle films by determining the fracture strength and concluded that the strength value was in the same range in both initial and multiple formation of cracks. Another researcher [132] gave typical fracture stresses of plasma-sprayed zirconia coatings in the range from 500 to 700 MPa while the corresponding fracture strains under tensile conditions were between 0.1 to 0.4 %.

However, as discussed in Section 2.4, the fracture mechanics parameters are not accurate enough to describe the failure at microstructure level and accordingly, some other criteria have been proposed. Some researchers [156, 157] proposed a Griffith-like strain-energy-based criterion \( \frac{1}{2} \sigma^* \varepsilon^*_y \frac{A_e}{2} \geq 2\gamma \), where \( A_e \) is the area of an element; \( \sigma^* \) and \( \varepsilon^*_y \) are the stress and strain of the element, respectively; \( \Delta L \) is the crack extension in element; \( \gamma \) is the surface energy of the (intergranular) cracked interface and assigned a value dependent on the material. Based on this criterion, critical temperature loading for microcrack formation in a RVE of a bulk alumina sample (isotropic, \( \gamma = 0.89 \text{ J/m}^2 \)) was predicted. Najar etc. [21] determined the change of the energy absorption capacity \( W^* \) (see Section 2.3 ), which characterizes the enhanced fracture energy losses at failure, with the temperature of bulk alumina samples. Their results are shown in Table 3-2:

Table 3-2: Continuum damage model parameters for bulk alumina samples at elevated temperatures [21].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Young’s modulus (GPa)</th>
<th>Fracture strength (MPa)</th>
<th>( W^* ) (kJ/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>394.5</td>
<td>285.8</td>
<td>52.7</td>
</tr>
<tr>
<td>400</td>
<td>386.7</td>
<td>269.9</td>
<td>47.5</td>
</tr>
<tr>
<td>750</td>
<td>373.4</td>
<td>255.2</td>
<td>44.3</td>
</tr>
<tr>
<td>1000</td>
<td>360.6</td>
<td>244.7</td>
<td>42.2</td>
</tr>
<tr>
<td>1500</td>
<td>326.7</td>
<td>223.8</td>
<td>38.9</td>
</tr>
</tbody>
</table>
Based on the parameters and the failure criteria, damage and fracture initiation and propagation in ceramic coatings under various loading are investigated analytically and experimentally.

### 3.3.3 Damage and Fracture Behaviour of Ceramic Coatings under Various Loading

Examination of the damage and fracture behaviour of ceramic coatings generally focus on the crack modes, the measurement of length, number etc. of the cracks induced by thermal or mechanical loading, as well as the correlation of the crack parameters. In addition, in many research on the coatings, effect of the coating geometry such as thickness, length etc. on the behaviours is seriously taken into account as well. In the case of thermal loading, the critical temperature loading – thermal shock resistance $\Delta T$ is also an important parameter describing the behaviours.

- **Thermal loading behaviours**

  In the investigation [158] of thermal fracture of plasma sprayed ceramic coatings under high heat flux laser heating, two different cracking modes were found to occur upon cooling after the heating: surface cracks and surface-interface cracks. A schematic diagram of these two types of cracking is given in Fig. 3-8. The surface cracks were perpendicular to the surface, and a surface-interface crack including a surface crack as well as an interface crack that was located at or above the interface between the coating and the bond layer. The surface cracks were all found to occur within the zone of the specimen heated by the laser while the interface cracks were always found directly below the surface cracks. However, the surface cracks did not necessarily extend through the coating to the interface.

![Figure 3-8 Schematic diagram of surface and interface cracks in plasma sprayed ceramic coating under laser-induced thermal shock.](image-url)
In another thermal shock test [159] by heating the surface of a coating specimen using combustion flame of hydrogen-oxygen gas mixture, the authors also found cracks generated vertically in the top surface layer of the coating in a specimen. The cracks deflected and propagated in the boundary between the coating and the substrate in the direction parallel to the interface. The authors considered that the deflection of crack propagation caused a surface segment of the coating to be spalled out by link-up of the cracks together. In a similar test [160], the authors found that increasing crack density resulted in decreased interfacial cracking at the end of the transient thermal shock.

In the test [158], coating thickness was found to influence the number, spacing and length of surface cracks generated by a laser-induced thermal shock. Increasing the coating thickness was found to decrease the number of surface cracks induced by the loading and increase the distance between the cracks. Surface cracks were found to extend further into thin coatings than thick coatings, which were produced from same material and with similar spray parameters, and thus were considered to have the same type of microstructure. Increasing the maximum surface temperature was found to increase the surface crack length. The thinnest and thickest coatings developed fewer and shorter interface cracks than coatings with intermediate thickness. These results indicate that the morphology of surface cracks in such coatings has a significant effect on the response of interface cracks to a thermal load produced by a high heat flux. Thus, thicker thermal barrier coatings could be made more resistant to interface crack propagation.

It has been known that the thermal stresses responsible for the thermal shock damage are primarily controlled by the temperature drop $\Delta T$ through the specimen thickness. In [158], higher maximum surface temperatures were found to cause longer surface cracks independent of coating thickness. Results from an indentation quenching test [161] for a bulk alumina specimen show no crack growth at very low temperature loading $\Delta T$, stable crack growth at medium $\Delta T$ and unstable crack growth above a certain $\Delta T$.

By heating one surface of an alumina sample while cooling its opposite surface so as to create a temperature $\Delta T$ as well as thermal stress gradient inside the sample, the authors [162] investigated thermal shock behaviour of the sample. They found that failure generally initiated at periphery of the top surface of the sample and the critical temperature difference $\Delta T_c$ was dependent on thickness of the sample and was higher for thicker samples. The tests were simulated using a finite element method to obtain the
temperature and thermal stress distributions experienced by the specimens and the results were compared with the experimental results.

- **Mechanical loading behaviours**

Besides effect of thermal loading on the damage and fracture in ceramic coatings, effect of mechanical loading is also an important research area. For example, the blades and vanes of gas turbines are often subjected to bending or tension. Thus, in recent years, some concentrated efforts [163-165] have been made in studying the mechanisms of crack initiation and propagation in ceramic coatings under controlled mechanical loading. Keeping this in view, the attempt [163] was made to study the crack propagation behaviour in coatings by bending test at room temperature as well as at a higher temperature. The results reveal that the crack path trajectory in the coatings often showed crack branching and deflection, while the cracks (length) grew linearly in the coatings with increase in bending load until the yield point of the substrate was about reached. Approaching the interface between the ceramic layer and the bond coat, a high threshold load was required to propagate the crack further into the bond coat. Once the threshold was surpassed, the crack grew rapidly into the brittle bond coat without an appreciable increase in the load.

In another bending test [166] for TBC systems at ambient temperature, the number and lengths of micro and macro cracks induced by the loading were measured. It was found that the crack behaviour (number, lengths etc.) of the TBC systems depended strongly on the topcoat microstructures as well as heat treatment after the plasma spraying. For TBC systems with large numbers of microcracks in the coatings, the macrocrack development was appreciably delayed, compared with those with only a few microcrack. This was believed mainly due to the effective stress relief associated with the opening of the individual microcracks. The compressive failures were also found rather incidental and dependent on the strength of topcoat at the interfacial region between the coatings and substrates.

Some authors [132] also examined the stress-strain behaviour of a thermal barrier zirconia coating in a bending test. They found that a strain of 0.1 to 0.2% resulted in the initiation of cracks at the coating surface. Then, it can be seen from Fig. 3-9 that these cracks grow perpendicular to the surface through the coating with further increase of the strain. Close to the bond-coat/coating interface, cracks become deflected and further
crack growth occurs parallel to the interface. Delamination of the coating was observed at tensile strains between 1.0 to 1.5%. In addition, it was found in the test that cracks always initiated at surface pores of the coatings and further increase of strain resulted in crack growth and linking up of cracks to form a crack network.

![Figure 3-9 A typical stress-strain behaviour of a TBC on coated samples in a 4-point bending test [132].](image)

3.3.4 Modelling Damage and Fracture Evolution in Ceramic Coatings

Numerical simulations for damage and fracture process in ceramic coatings have been done by a large number of authors. Most of them have modelled the process at macrostructure level in terms of fracture mechanics. Some representative and simple examples of such simulations [148, 149] have been introduced in Section 3.3.1. There are two main points for them: 1) assuming that a coating is homogeneous and the stress in the coating is uniform and equal biaxial; 2) using the criteria of fracture mechanics to evaluate failure of the coating under various loadings.

To explain further failure phenomena of the coatings, similar and more complex models are subsequently proposed by introducing the FEM. For instance, on the assumption of the coating being homogeneous, Buchmann etc. [167] simulated a thermal spraying process of coating using FEM to determine the thermal residual stresses. The heat supply during the process was modelled as a Gaussian distribution parallel to the surface. Likewise, on the assumption of the homogenized properties, Gao etc. [168] examined cracking patterns of coatings under tensile or thermal stress based on Eq. (3-5)
Chapter 3. Damage and Fracture Evolution in Ceramic Coatings

and theory of fracture mechanics and FEM. In another simulation for laser-induced thermal shock test [169], the authors calculated transient thermal stress field in an infinite plate and stress intensity factors for an embedded centre crack at macroscale.

Furthermore, the authors [160, 170, 171] discretized a coating into finite elements at microscale and then simulated surface and interface crack initiation and propagation in coatings under thermal shock. In the simulations, they assumed isotropic coating material and plane stress [160, 170] or plane strain [171] condition while effect of microstructure details such as microdefects etc. were still neglected. They firstly analysed thermal stresses at the thermal shock centre using finite element method. Then, they calculated the strain energy release rate for an introduced coating-substrate interface crack [160] under the centre surface and a surface crack [170] perpendicular to the interface, respectively. Similar work was also reported in [172, 173]. The authors used the homogeneous and isotropic model to obtain representative analytical results of stress field (plane stress) and crack behaviour in coatings. Especially, some authors [174] evaluated stress field in thermal barrier coatings under thermomechanical loading using a high-order theory [175]. They found that their results were overestimated by the analysis and suggested that microstructure details of the coatings should be explicitly taken into account in order to gain a better understanding of delamination growth induced by the loading.

However, due to microstructure complexity of ceramic coatings, only a few studies have been done with the consideration of effect of microstructure of coatings. As we have referred above, compared to the comparable bulk ceramics, ceramic coatings present more complicated behaviours due to their manufacturing process. Their anisotropy must be considered in analyses in order to obtain reasonable explanations for their behaviours, although this may lead to more time-consuming solutions or convergency problems in the analyses. An example of such studies can be seen in [176], which used the analytical method GSCM [44] to calculate the homogenized elastic and thermal properties with consideration of microstructural details. Then, based on the properties, the authors further calculated the local states of stress and strain of ceramics under a thermal loading $\Delta T$. At last, they proposed a failure criterion in terms of local conditions and the definition [177] of the thermal shock resistance to crack initiation and crack propagation. Another example is the work done by Zimmermann etc. [88], who examined microcracking due to thermal expansion and elastic anisotropy via computer...
simulations based on a Griffith-type failure criterion. They generated random microstructures of polycrystalline coatings using Monte Carlo Potts-model simulations. Then, critical mismatch strain leading to the onset of microcracking in a RVE was calculated using FEM and damage accumulation (number of failed elements) was correlated with the three-parametric Weibull distribution of microstructural parameters.

From a point of view of statistics, using finite element method associated to a statistical description of fracture stress, the authors [178] simulated damage and fracture process of inhomogeneous materials submitted to tension and 3-point bending, respectively. They found that the lower limit and the scattering of the fracture stress distributions were the dominant characteristics of the damage development in the two types of tests. High intervals of the stress distributions made easier a diffuse progression of damage whereas a localised development of macrocracks was observed in the case of low intervals. In addition, their results indicate that the damage development was clearly different in tensile and bending tests.

3.4 Summary

Microstructure of ceramic coatings is usually characterized by the parameters such as porosity and amount, size, shape etc. of microdefects as well as grains in the coatings. Real microstructures of coatings often demonstrate randomness in the distribution of microdefects making the periodicity assumption, usually used to analyse bulk materials, too restrictive. Various statistical distribution functions have been proposed to describe the parameters. Especially, thermally sprayed coatings are usually composed of splats and various types of voids and this leads to anisotropic or transversely isotropic properties of the coatings.

Effective elastic or thermal properties of ceramics with inhomogeneities have been investigated in many papers with the consideration of effect of microstructure. Young's moduli of thermally sprayed ceramic coatings are known to be much lower than that of comparable bulk materials due to the effect of the splat-based microstructure that includes highly non-spherical pores with a high aspect ratio. The ratio between the stiffness $C_{11}$ in spray direction (see Fig. 3-4) and that $C_{22,33}$ in transverse directions could be up to 0.5. The effective properties have also been determined by indentation tests, bending tests or dynamic resonance tests.
The stresses in various ceramic coatings generally consist of transient thermal stresses induced by thermal loading, tensile stresses induced by mechanical loading, and the mismatch stresses induced by elastic or thermal expansion mismatch for the coatings and metallic substrates. The combined stresses can cause cracks initiation and propagation and eventually lead to failures of the coatings. The cracks and failure are characterized by many researchers using the parameters and criteria of fracture mechanics. To introduce effect of microstructure, some improved or different parameters or criteria have been proposed and obtained by experimental and analytical method. But further validation on the parameters is still expected.

Various tests such as laser-induced thermal shock tests, mechanical bending tests etc. have been conducted to study the damage and fracture evolution of ceramic coatings during loading. These tests have also been simulated by the researchers at macroscale or microscale. The simulations can be divided into three groups. In the first group, the criteria of fracture mechanics are directly used to evaluate failure of the coating under various loadings on the assumption of homogeneous and isotropic properties. Effect of microstructure and coating geometry is neglected. In the second group, the criteria of fracture mechanics are used with the introduction of FEM either at macroscale or microscale. In the last group, effect of microstructure is considered in the simulations using analytical or micromechanical method accompanying new failure criteria and statistical description for the microstructure. Obviously, results from the last group should be more reasonable although further work is still expected.
Chapter 4. Experimental Results

This chapter investigates damage and fracture of thermally sprayed alumina coating under mechanical and thermal loading, respectively, based on experimental studies. The completed experiments included nano-indentation tests, three-point bending tests and thermal loading tests. Through the experiments, the material properties of the coating and its behaviour under thermal or mechanical loading were investigated.

Through a thermal plasma spray process (Poeton Ltd., U.K), described in Section 3.2.2, aluminium plates (each 5×114×114 mm, see Fig. 4-1) were coated with a thermal barrier coating (thickness approximately 250 μm) with an adhesive layer (thickness approximately 10 μm, 20% Nickel, 80% Chromium). The coating material used for this study was a high-density Apticote 800/55P alumina oxide with the properties shown in Table 4-1.

Figure 4-1 An alumina – aluminium plate.
Table 4-1 Properties of the test specimens provided by Poeton Ltd., U.K.

<table>
<thead>
<tr>
<th>Type</th>
<th>Coating: High-density Alumina Oxide (Al 98.5%, Silicon Dioxide 1%)</th>
<th>Aluminium alloy substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina-Aluminium</td>
<td>Density (g/cc) = 2.9/3.3</td>
<td>Young’s modulus: 71 GPa</td>
</tr>
<tr>
<td></td>
<td>Porosity &lt; 2%</td>
<td>Melting point = 550–660 °C</td>
</tr>
<tr>
<td></td>
<td>Melting point = 2000 °C</td>
<td></td>
</tr>
</tbody>
</table>

Each test specimen was cut out from the plate by a Microfine Bench Top Abrasive Cut-off Machine (D.R.BENNETT LTD, UK, see Fig. 4-2) in the shape of beams (see Fig. 4-3). The external coating surface was kept in the as-sprayed condition and the cross-sectional surface of each specimen was polished using grinding machine to a better surface finish for observing microstructures, crack formations during tests and post-test crack measurements. Before the tests, all specimens were checked under a microscope to ensure that there were no cracks induced during cutting. Figure 4-4 shows a micrograph of the cross-sectional surface of a plasma sprayed alumina coating.
4.1 Nano-Indentation Test

Nano-indentation is similar to conventional indentation testing, but it is performed on much smaller scale – nano or micro scale. This is important for very hard coating on
softer substrates in the case that the indentation depth may need to be less than 1/10 of the film thickness to avoid substrate effects.

Recent improvements in indentation equipment allowed accurate measurements of load $P$ down to micro-Newton ($\mu$N) and the minimum penetration depth $h$ down to nanometres (nm) although the depth used in this chapter was micrometres. To measure nanomechanical properties, a very small calibrated diamond probe is brought into contact with the sample surface at a specified applied load. The diamond probe is either Vickers or Berkovich indenter, the tip of which is a four-sided or three-sided pyramid, in contrast to the traditional sphere or a cone (a Rockwell indenter) usually used for hardness testing. The resultant displacement of this sharp diamond pyramid into the surface is monitored and displayed in real time as a function of applied load. Data is acquired, analysed and stored in ASCII format. Since depth resolution is on the scale of nanometres, it is possible to conduct indentation experiments even for very small surface areas. Two quantities, which can be extracted from nano-indentation experiments, are material's hardness and its Young's modulus. In this study, the nano-indentation tests are employed to obtain the Young's modulus of alumina coatings.

4.1.1 Instrumentation and Principle of Data Analysis

1) Instrumentation

The tests were performed with the NanoTest platform (Fig.4-5) made by Micro Materials Ltd, Wrexham, UK. The components of this device are shown in Fig. 4-6. This instrument possesses the following features:

- A load is applied by means of a coil and magnet located at the top end of the pendulum.
- The penetration of the probe is monitored with a sensitive capacitive transducer.
- All instrument calibrations are performed automatically.

The specification of the range and sensitivity for the displacement and load is shown in Table 4-2. The Berkovich indenter (Fig. 4-7) was used in all the tests.
Chapter 4. Experimental results

Figure 4-5 NanoTest platform.

Figure 4-6 NanoTest platform design.

Figure 4-7 Berkovich three-faced pyramid.
Table 4-2 Specification of the NanoTest Platform.

<table>
<thead>
<tr>
<th>Displacement range and sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range</td>
</tr>
<tr>
<td>Noise-floor</td>
</tr>
<tr>
<td>Theoretical resolution</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Load range and sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum resolution</td>
</tr>
<tr>
<td>Load ranges</td>
</tr>
<tr>
<td>X/Y/Z resolution/travel</td>
</tr>
<tr>
<td>Analysis area</td>
</tr>
</tbody>
</table>

2) Nanoindentation Data Analysis

The NanoTest measures the penetration depth of a calibrated diamond probe as a function of the applied load during a loading-unloading cycle (see Fig. 4-8). During unloading, the elastic component of the displacement starts to recover producing a sloped rather than horizontal unloading curve. It is from this slope that the elastic properties can be derived. The hardness is derived from the residual depth of the unloading curve as described below.

![Figure 4-8 Plastic depth determination in nanoindentation.](image-url)
After frame-compliance correction, the raw depth vs. loading and unloading data is fitted to a power law function of the form: \( P = a(h - h_t)^m \), where \( a \), \( h_t \) and \( m \) are constants. The true diamond contact depth is determined from the expression: 
\[
h_c = h_{\text{max}} - c_i S P_{\text{max}}
\]
where \( h_{\text{max}} \) is the maximum penetration depth, \( P_{\text{max}} \) is the maximum load, \( S \) is the contact compliance equal to the tangent at the maximum load, and \( c_i \) is the coefficient that depends on the indenter geometry.

Hardness \( H \) is determined from the peak load and the projected area of contact, \( A \):
\[
H = \frac{P_{\text{max}}}{A}.
\] (4-1)

To obtain the elastic modulus, the unloading portion of the depth-load curve is analyzed according to the following relation: 
\[
S = \frac{\pi \delta^2}{(2E_s A^0.5)},
\]
where \( E_s \) is the reduced modulus defined by
\[
\frac{1}{E_s} = \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_i^2}{E_i},
\] (4-2)
where \( E_s \) and \( \nu_s \) are the Young's modulus and Poisson's ratio for the sample, respectively; \( E_i (=1141 \text{ GPa}) \) and \( \nu_i (=0.07) \) are those for the indenter.

### 4.1.2 Specimen Preparation and Experimental Procedure

Specimens (see Fig. 4-9) of alumina deposited onto the aluminium substrate in the spray direction were examined in the tests. The dimension of each specimen was 10 mm x 16 mm x 5.5 mm. Before the tests, the top surface of the coatings was polished to exclude the effect of surface roughness. The tests were carried out in an automatic mode with a total duration of one day. There were four runs A, B, C and D, parallel to each other. The distance between two neighbouring indentation points of each run was 100 \( \mu \text{m} \). Each indentation point of each run was produced with a constant load of about 50, 100, 200 and 400 mN applied to the probe tip, corresponding to four runs A, B, C and D. The difference in the residual depth of the crater produced with the probe was automatically recalculated into hardness and the Young's modulus of the corresponding indentation place as has been shown above.
4.1.3 Experimental Results and Discussion

Alumina coating properties were obtained by the nano-indentation tests. Figures 4-10 and 4-11 show the obtained Young's modulus and hardness of the coating in different test points using the contact force 50, 100, 200 and 400 mN, respectively. The average value of the Young's modulus, hardness and other properties is given in Table 4-3. It is obvious that the values at different points fluctuate randomly, due to randomness of microstructure morphology of the ceramic coating. The level of scatter is higher for the case of lower loads. For example, there is a considerable difference between the maximum value (203 GPa) and minimum value (72 GPa) in the category 50 mN contact force. With the decrease in the load level and the corresponding maximum depth, the obtained values of both Young's modulus and hardness as well as their standard variance increase.
Figure 4-10 Young's modulus of the alumina coating from the nanoindentation tests.

Figure 4-11 Hardness of the alumina coating from the nanoindentation tests.
Table 4-3 Average values of alumina coating properties in nanoindentation test.

<table>
<thead>
<tr>
<th>Force (mN)</th>
<th>400</th>
<th>200</th>
<th>100</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test points</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Young's modulus (GPa)</td>
<td>104.95±15.69</td>
<td>106.29±21.89</td>
<td>116.20±23.05</td>
<td>140.25±35.02</td>
</tr>
<tr>
<td>Hardness (GPa)</td>
<td>5.06±1.78</td>
<td>6.79±3.25</td>
<td>8.74±3.01</td>
<td>10.19±3.23</td>
</tr>
<tr>
<td>Maximum depth (nm)</td>
<td>2195±481</td>
<td>1476±355</td>
<td>929±200</td>
<td>555±92</td>
</tr>
<tr>
<td>Plastic depth (nm)</td>
<td>1911±490</td>
<td>1244±354</td>
<td>784±203</td>
<td>445±80</td>
</tr>
<tr>
<td>Contact compliance (nm/mN)</td>
<td>0.94±0.07</td>
<td>1.55±0.08</td>
<td>1.93±0.15</td>
<td>2.93±0.55</td>
</tr>
</tbody>
</table>

These results suggest the following features of the process. Firstly, the lower force corresponds to the lower maximum penetration depth (e.g., 555 nm), which is considered to be insufficient to include the effect of the modulus reduction by microvoids. Thus the obtained values could reflect the local properties rather than global ones. This means that the mean value 140 GPa (corresponding to the force 50 mN) could be the actual Young’s modulus of the splats or grains in the coatings without the effect of microvoids. Secondly, with the increase in the depth for each load category, the Young’s modulus decreases. This is considered to be due to the effect of microvoids (number, shape, size, location etc.), which become more stable (smaller variance) in the case of larger penetration depth.

In addition, the properties of the aluminium substrate are also measured in the nanoindentation tests, shown in Figs.4-12 and 4-13. Corresponding to the load force 50, 100 and 200 mN, respectively, the average Young’s modulus is 76.7±6.0, 76.4±6.1 and 70.4±3.3 GPa; the hardness is 0.547±0.031, 0.527±0.023 and 0.495±0.016 GPa. They are in good agreement with the known properties of aluminium published in literatures.
Chapter 4. Experimental results

4.1.4 Summary

The values of the properties (Young’s modulus and nano-hardness), measured by nano-indentation at different points, fluctuate randomly. With the decrease in the load level, the obtained values for both Young’s modulus and hardness as well as their standard variance increase.
4.2 Three-Point Bending Tests

Various components, e.g., turbine blades, which are made of alloys with ceramic coatings, are often subjected not only to purely thermal loads but also to mechanical ones. Both types of loading can induce cracking in and spalling of the coatings. Thus, in this section, three-point bending tests were employed to study the effect of microstructure on crack initiation and propagation in the alumina coating under mechanical loading. In addition, some parameters of the alumina coating were determined by the tests. These parameters are tensile strength and the Young's modulus of the alumina coating in transverse direction (perpendicular to the spray direction). The tests were conducted at room temperature (20°C). The three-point bending device (see Fig. 4-14) was used. It consists of the test machine and the computer-based data acquisition system, which can automatically record the history of force, extension and maximum deflection of the tested specimen during loading. The technical specification of the device is given in Table 4-4.

Figure 4-14 The instrumentation (LLOYD Instruments Ltd, UK) used in three-point bending tests.

Table 4-4 Technical specification of the three-point bending device.

<table>
<thead>
<tr>
<th>Standard force range</th>
<th>0.5 N to 2.5 kN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force accuracy</td>
<td>Actual performance of 2.5 kN load cell: 1% accuracy of measured force between 1% to 100%; 0.5% accuracy of measured force between 2% to 100%. Actual performance of</td>
</tr>
</tbody>
</table>
lower force load cells depend on application, grips and sample.

<table>
<thead>
<tr>
<th>Load resolution</th>
<th>Maximum resolution better than 0.01% of load cell rating.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extension resolution</td>
<td>&lt; 5 µm</td>
</tr>
</tbody>
</table>

The recording system of the bending device was calibrated before testing. Figure 4-15 shows curves of force against deflection of a beam-shaped aluminium specimen (dimension $L \times W \times H_2 = 37 \text{ mm} \times 3.5 \text{ mm} \times 5 \text{ mm}$, Young's modulus $E = 70 \text{ GPa}$, span $S = 27 \text{ mm}$) during loading.

![Force versus deflection](image)

Figure 4-15 Force versus deflection recorded by the calibrated bending device and a dial gauge, as well as from the simple bending theory.

Triangular marks in Fig. 4-15 denote the force-deflection values measured using a dial gauge and the solid curve represents the values automatically recorded by the calibrated bending device. The dashed line represents a part of the force-deflection relation before the onset of yielding, fitted to test points. The dashed-dotted line represents the values calculated using the simple bending theory, in which the relation between the deflection and force is:
\[
\delta = \frac{FS^3}{48EI_z}, \tag{4-3}
\]

where \(\delta\) is the deflection, \(F\) is the force, \(S\) is the span, \(I_z\) is the second moment of the cross-sectional area of the beam about the neutral axis: 
\[
I_z = \frac{WH^3}{12} = \frac{3.5 \times 5^3}{12} = 36.458 \text{ mm}^4.
\]

It can be seen from the figure that the fitted line and the theoretical line agree very well. The bending strength of the aluminium beams, corresponding to the yield point A in Fig. 4-16, can be obtained:

\[
\sigma_{bc} = \frac{3FS}{2WH_z} \approx 192 \text{ MPa}. \tag{4-4}
\]

From Eqs. (4-3) and (4-4), the strain can be obtained:

\[
\varepsilon = \frac{\sigma}{E} = \frac{6\delta H_z}{S^3}. \tag{4-5}
\]

When the deflection values from the test are used in Eq. (4-5), the strain-load relation (see Fig. 4-16) can be obtained.

![Figure 4-16 Load versus maximum strain of an aluminium beam during bending.](image)

**4.2.1 Specimen Preparation**

The upper limit of loading that can be applied by the bending device in the tests is around 700 N. Therefore, the specimens' dimensions were designed so that the coating can fail at a load less than 700 N. Beam-shaped test specimens (see Fig. 4-3) were firstly
cut out from the plate (shown in Fig. 4-1) with the cut-off machine (Fig. 4-2). The full length of each specimen was about $37 \pm 1$ mm. A grinder-polisher was used to shape the specimens to the required size. The external top surface with coating was kept in the as-sprayed condition. The cross-sectional face of each specimen was polished manually using emery polishing paper of four increasingly finer abrasive grits (Fig. 4-17(a)). This was followed by further polishing the specimen with a finer polisher (Fig. 4-17(b)), cleaning with acetone and drying to a better surface finish for observing microstructures. Before the tests, each specimen was checked under an optical microscope to ensure that there are no cracks induced during cutting. The thickness of the coating (ceramic layer) was measured by optical microscopy. The width of each specimen was measured by a vernier. A typical SEM of the specimens is shown in Fig. 4-18.

Figure 4-17(a) Emery polishing paper with four increasingly finer grits 200, 400, 800 and 1200.
Figure 4-17(b) A polisher with 6 µm grits.

Figure 4-18 A SEM cross-sectional micrograph of a specimen.
4.2.2 Experimental Set-Up and Procedure

The radii of a central mandrel and two support rollers of the three point bending machine (see Fig. 4-19) is 10 mm. The specimen was placed so that its coating was in a tensile state. The loading speed was set as 2 mm/min and the loading force, time, extension and deflection for each specimen during loading can be recorded by the computer system during test. A high speed video system (PHOTO-SONICS PHANTOM V7, see Fig. 4-20) was used to observe and record the changes at the cross-sectional surface of the coating during loading. The system consists of a camera and a computer for recording. The camera use SR-CMOS sensors with a maximum resolution of 800 x 600 (21 μ) pixels at up to 4,800fps giving 1.2 seconds of recording time.

Figure 4-19 The experimental set-up of the three-point bending test.
Figure 4-20(a) High speed video system and three-point bending device.

Figure 4-20(b) High speed camera and experimental set-up.
Chapter 4. Experimental results

Before the tests, some specimens were loaded in order to determine the minimum critical loading force necessary to initiate the first crack in the coating.

Then, the test procedure was as following:
1) Install a specimen in the test machine (see Fig. 4-19).
2) Set maximum force and loading rate.
3) Start loading until the load arrives at the maximum value and then unloading.
4) Observe the specimen under microscope and record the details.
5) Mark the specimen and put it in a bag.
6) Repeat step 1) to 5) until all specimens were tested.

4.2.3 Experimental Results and Discussion

• Mechanical properties of the alumina coating

Twenty four specimens were tested and the failure force $F_c$, corresponding to the first crack in the coating during loading for each specimen, is shown in Table 4-5.

Table 4-5 Experimental results of three-point bending.

<table>
<thead>
<tr>
<th>Specimen's ID</th>
<th>$S$ (mm)</th>
<th>$W$ (mm)</th>
<th>Yield force ($F_c$ N)</th>
<th>$\frac{F}{\delta}$ (N/mm)</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$E_{\text{cx}}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>3.60</td>
<td>488</td>
<td>476</td>
<td>7979</td>
<td>328</td>
<td>140</td>
</tr>
<tr>
<td>S2</td>
<td>3.86</td>
<td>538</td>
<td>522</td>
<td>8231</td>
<td>285</td>
<td>111</td>
</tr>
<tr>
<td>S3</td>
<td>3.75</td>
<td>508</td>
<td>493</td>
<td>8461</td>
<td>350</td>
<td>153</td>
</tr>
<tr>
<td>S4</td>
<td>3.40</td>
<td>487</td>
<td>474</td>
<td>7531</td>
<td>345</td>
<td>140</td>
</tr>
<tr>
<td>S5</td>
<td>3.80</td>
<td>539</td>
<td>523</td>
<td>7959</td>
<td>264</td>
<td>99</td>
</tr>
<tr>
<td>S6</td>
<td>3.45</td>
<td>404</td>
<td>722</td>
<td>217</td>
<td>222</td>
<td>98</td>
</tr>
<tr>
<td>S7</td>
<td>3.50</td>
<td>473</td>
<td>816</td>
<td>399</td>
<td>179</td>
<td>128</td>
</tr>
<tr>
<td>S8</td>
<td>3.50</td>
<td>463</td>
<td>724</td>
<td>235</td>
<td>91</td>
<td></td>
</tr>
<tr>
<td>S9</td>
<td>3.50</td>
<td>461</td>
<td>743</td>
<td>273</td>
<td>109</td>
<td></td>
</tr>
<tr>
<td>S10</td>
<td>3.50</td>
<td>485</td>
<td>737</td>
<td>275</td>
<td>103</td>
<td></td>
</tr>
<tr>
<td>S11</td>
<td>3.50</td>
<td>493</td>
<td>763</td>
<td>329</td>
<td>128</td>
<td></td>
</tr>
<tr>
<td>S12</td>
<td>3.50</td>
<td>472</td>
<td>458</td>
<td>7706</td>
<td>317</td>
<td>134</td>
</tr>
</tbody>
</table>
Chapter 4. Experimental results

All the specimens had same thickness ($t_e$) 0.25 mm for the coating and ($t_s$) 5.15 mm for the substrate. During loading, the span (S) was either 28 mm for the specimens with width $W$ less than 4 mm or 30 mm for those with higher $W$. Six of them (S7 to S12) were made to have uniform width ($W$) 3.50 mm so that the corresponding results (see Fig. 4-21(a)) could be compared. One of them (S12) was loaded until it yielded while the others were loaded until the first crack could be observed. In all cases the first crack appeared before the onset of irreversible deformation of the specimen. A typical example of the force-deflection diagram for a specimen loaded in the three-point bending machine is shown in Fig. 4-21(b). The solid line represents the force-deflection relation fitted to the test data using the least square method and the ratio of force and deflection ($\frac{F}{\delta}$) for each specimen during loading is calculated (see Table 4-5).
Chapter 4. Experimental results

Figure 4-21(a) Fitted lines of the force vs. deflection diagram for six alumina-aluminium specimens (S7 ~ S12) tested in three-point bending.

Figure 4-21(b) The typical force vs. deflection diagram for the alumina-aluminium specimen (S12) tested in three-point bending.
The ratio \( \frac{F}{\delta} \) is used to calculate the Young's modulus of the coatings in terms of the simple bending theory for two-layer composite beams [33, 179]. The deflection increment \( \Delta \delta \) of the specimen at the surface of the coating are expressed as:

\[
\Delta \delta = \frac{S^3}{48E_s (I_s)_e},
\]

where \( \Delta F \) is the increment of the loading force, \( S \) is the support span, \( E_s (70\text{GPa}) \) is the Young's modulus of the substrate. The equivalent moment of inertia of the specimen's area is

\[
(I_s)_e = I_s + t_c \cdot W_{ec} \left( \frac{d - t_c}{2} \right)^2 + t_s \cdot W \left( t_c + \frac{t_s}{2} \right)^2,
\]

where \( I_c = \frac{Wt_c^3}{12} \) and \( I_s = \frac{Wt_s^3}{12} \) are centroidal moments of inertia of the area of the coating and the substrate respectively, \( t_c \) and \( t_s \) are the thickness of the coating and the substrate respectively, \( W \) is the width of the specimen and the equivalent width \( W_{eq} = \frac{E_{cs}W}{E_{cs}W + t_c \cdot W + t_s \cdot W} \) is the Young's modulus of the coating, \( d = \frac{t_c \cdot W_{ec} \cdot \left( \frac{t_c}{2} + t_s \right) \cdot W \left( t_c + \frac{t_s}{2} \right)}{t_c \cdot W_{ec} + t_s \cdot W} \) is the distance between the neutral axis and the surface of the coating. In terms of the Eqs. (4-6), (4-7) and the values of the force and deflection in Table 4-5, the value \( E_{cs} \) for each specimen can be calculated implicitly and is given in Table 4-5 and Fig. 4-22. The normal cumulative distribution of the relative modulus \( \left( \frac{E_{cs}}{E_{ava}} \right) \) of the coatings is shown in Fig. 4-23. These values can serve as a basis for describing microstructure of the alumina coating in our numerical simulations in following Chapters.
Chapter 4. Experimental results

Figure 4-22 The Young's modulus of the alumina coating for the alumina-aluminium specimens.

Figure 4-23 Normal cumulative distribution of the relative Young's modulus of the alumina coating.
Stress/strain values corresponding to the force-deflection curves are also evaluated. The maximum stress \( \sigma_{\text{ex}} \) at the centre of the coating surface is expressed as

\[
\sigma_{\text{ex}} = \frac{E_{\text{ex}}}{E_s} (\sigma_{\text{ex}})_e.
\] (4-8)

where the equivalent maximum stress at the surface of the coating \((\sigma_{\text{ex}})_e = \frac{M_s}{(I_s)_e^3} d \). Thus, the bending strength \( \sigma_{\text{cmax}} \) (see Table 4-5) of the coating for each specimen can be calculated as following:

\[
\sigma_{\text{cmax}} = \frac{E_{\text{ex}}}{E_s} \frac{M_s}{(I_s)_e^3} d = \frac{E_{\text{ex}}}{E_s} \frac{F_c \cdot S \cdot d}{4(I_s)_e^3}.
\] (4-9)

Based on these values and the corresponding Young's modulus \( E_{\text{ex}} \), the energy absorption capacity \((W^*)\) of the alumina coating is determined in Chapter 5.

It can be seen from Fig. 4-21(a) that the critical force, corresponding to failure of the coating, and the force-deflection ratio varies for different specimens (S7 to S12) although they have uniform dimensions and loading conditions. The material properties (see Table 4-5), obtained for other specimens, also show fairly large differences. These fluctuations are believed to be due to two aspects. The first aspect is the effects of friction and shift of contact lines, which may lead to an error of 5~11% and have been discussed in the literature [4]. The second aspect is attributed to the effect of microstructure of the alumina coating. Essentially, failure phenomenon is a local behaviour of materials and it depends on both local stress states and material properties. During three point bending tests in this chapter, stresses usually concentrated in the central area of the coating, at which local microstructures of each specimen were different due to the random nature of microstructure of ceramic coatings and thus might have different local properties. Consequently, this effect induces the large variance of material properties shown in Fig. 4-22.

- **Crack initiation and propagation behaviour of the alumina coating**

Failure processes in the coated specimens during loading were recorded by the high speed video system. After the tests, each specimen was thoroughly observed under optical microscope or using SEM. The first crack induced by loading is usually situated in the central area of the coatings shown in Fig. 4-24. The level of the critical load inducing the first crack varies (see Table 4-5) for different specimens although some of
them (S7 to S12) had same dimension (length, width and thickness) and a span during loading. The observed cracks generally start at the top surface of coatings, propagate through their thickness, penetrate bonds and stop at the interfaces between the bonds and substrates. The deflection of the first crack, corresponding to the critical load, is about 1 to 5 μm. Figure 4-24(b) shows a typical example of the failure status for the coating S9 with three macrocracks (1) and two microcracks (2) being initiated almost at same load 477 N before yielding of the specimen. The distance between the neighbouring cracks is about 1 mm. This implies that once a crack in the coatings is initiated, the fracture energy is also released in a zone with the length 0.5~1 mm.

Figure 4-24(a) The first crack induced by three-point bending in specimens S7, S8, S10 and S11.

Figure 4-24(b) The first cracks induced by three-point bending in specimen S9.

Observations by means of SEM indicate that the crack path has four types of trajectories: arced (Fig. 4-25(a)), inclined (Fig. 4-25(b)), branched (Fig. 4-25(c)) and nearly straight cracks (Fig. 4-25(d)). With the increase in the load, the number of cracks and deflection of each crack increase. No signs of spallation are found for coated alumina-aluminium specimens during in all the tests even if some specimens have yielded. This indicates that no larger interface cracks are produced between the coating and the substrate during loading.
Figure 4-25(a) Arced trajectory of crack path in the coating.

Figure 4-25(b) Inclined trajectory of crack path in the coating.
Figure 4-25(c) Branched trajectory of crack path in the coating.

Figure 4-25(d) Straight trajectory of crack path in the coating.
In order to study the failure behaviour of the alumina-aluminium specimen under three-point bending after the first crack has been initiated in the coating, the specimen S6 was loaded in three cycles shown in Fig. 4-26. In the first cycle (cycle 1 in Fig. 4-26), the specimen was loaded until 450 N at which the first crack (crack 1, Fig. 4-27(a)) was initiated. After the second cycle (cycle 2), the residual deflection was about 0.015 mm as it can be seen from Fig. 4-26. The specimen was loaded until 458 N in this cycle and another crack (crack 2) appeared (Fig. 4-27(b)). In the last cycle (cycle 3), three other cracks (crack 3a, 3b and 3c, Fig. 4-27(c)) were initiated when the load reached 470 N. The distance between the crack 1 and crack 2 is 4 mm, while spacings between neighbouring cracks after the third cycle are practically the same (1 mm).

![Figure 4-26 Three cycles of three-point bending for an alumina-aluminium specimen.](image-url)

---

**Figure 4-26 Three cycles of three-point bending for an alumina-aluminium specimen.**
Chapter 4. Experimental results

Figure 4-27(a) Failure status of the coating in the first cycle.

Figure 4-27(b) Failure status of the coating in the second cycle.

Figure 4-27(c) Failure status of the coating in the final cycle.
4.2.4 Summary

- Three-point bending was used to determine some mechanical properties of the alumina coating. The Young's modulus and strength of the plasma sprayed alumina coating is $128\pm21$ GPa and $299\pm41$ MPa, respectively. These values can be used to determine the parameter $W^*$ in the following Chapter.

- Four typical types of crack path trajectory are found in the coatings under three-point bending: arced, inclined, branched and straight cracks. The initial deflection of the first crack is from 1 to 5 $\mu$m. With the increase of the load, this value as well as the crack numbers increases. The distance between the neighbouring cracks is about 1 mm. No spallation of the coatings from the substrate was found in the tests.
4.3 Thermal Shock Test

Thermal Barrier Coatings (TBCs) have been used for many industrial applications. The coating material is usually ceramic deposited onto a metallic substrate. Coatings can protect metallic substrates (components) from high temperature environments and allow for an increase in operating temperatures, efficiency etc. of the coated components. However, thermal stresses can be generated in coatings under service conditions, e.g., thermal excursions, thermal shock etc. that can lead to their failure. It is well known that the microstructure of ceramics has a significant effect on the material's behaviour under loading. But this effect for ceramic coatings still awaits for further studies. Thus, in this section, laser-induced thermal shocks were applied to specimens with the alumina coating to study the effect of microstructure of the coating on its damage and fracture evolution under thermal loading. The results from the tests are used to validate our computational models described in Chapter 8.

In this section, an instrumentation used in the tests is introduced. Then, the manufacturing process for and specification of the specimens used in these tests are discussed. An experimental set-up and testing procedures are presented in the further sub-section. Next, the experimental results and their discussions are given in this section, finished by some conclusions.

4.3.1 Instrumentation

The instrumentation used in these tests consists of two parts: a 1.2 kW CO₂ laser machine (Fig. 4-28) manufactured by Convergent Energy and a thermal imaging system ThermaCAM SC3000 (see Fig. 4-29) manufactured by FLIR Systems AB. The laser power \( P_L \) can be varied from 100 to 1200 W by controlling the laser frequency \( f_l \) (1–1000 Hz), the laser pulse length \( T_p \) and the current setting \( I_{set} \). The heat flux distribution, generated by the laser machine, on the specimen's top surface is considered to be the Gaussian distribution with a diameter 12 mm of spot area (Fig. 4-30), which contains 86.6% of the power \( P_L \). The wavelength of the laser is 10.6 μm, at which the absorptivity of alumina is 76±5% [180].
Figure 4-28 A 1.2 kW CO₂ laser machine manufactured by Convergent Energy.

Figure 4-29 A thermal imaging system (FLIR ThermaCAM SC3000).
Chapter 4. Experimental results

Figure 4-30 The spot area of the 1.2 kW CO₂ laser.

The thermal imaging system includes an infrared (IR) camera and a computer recording system. The camera features the new Stirling-cooled Quantum Well Infrared Photon (QWIP), which is a long wave self-cooling analysis system with a cool down time less than 6 minutes. The IR detector has a thermal sensitivity of 20 mK at 30°C, a spectral range of 8 to 9 μm and can measure temperature in a range of -20°C to 2000°C. It has an accuracy of ± 1% or 1°C for measurement ranges up to +150°C and ± 2% or 2°C for measurement ranges above +150°C. The camera also includes a close-up lenses with a field of view 34x25 mm and the minimum focus distance 100 mm. The produced image has a resolution of 320x240 pixels with 14-bit digital data. The data is acquired with the real-time digital recording and temperature system Tracer Plus Package via a high capacity PC-card and then stored on the hard drive of a MS Windows-based PC (see Fig. 4-29). The information acquired, can be analysed using the ThermaCAM researcher software installed on the accompanying PC, or it can by converted into other formats such as Matlab or Excel. The accompanying PC is a Pentium IV 1.8 GHz running MS Windows 2000.

To measure the temperature accurately, it is necessary to compensate for the effects of a number of different radiation sources. This is automatically performed by the system itself. But, to implement this procedure, the following parameters must be supplied: the ambient temperature, the distance between the object and the camera, the relative humidity of the air and the emissivity of the object. The first three parameters are adjusted to the current values, and the only parameter that needs additional calibration is an emissivity of the cross-sectional surface of the alumina coating.
The emissivity is a measure of how much radiation is emitted from the object compared to that if it were a perfect black body. Normally, materials for various surface treatments exhibit emissivity ranging from approximately 0.1 to 1. An oxidised or painted surface usually has much higher emissivity than other surfaces. We need to know the emissivity in order to be able to calculate its temperature. Thus, before the tests, the emissivity was calibrated as 0.95–1.00 using the known melting point (about 1750°C) of the alumina coating. This value agrees fairly well with the data (see Fig. 4-31) of alumina obtained by other authors [181].

![Graph showing measured emissivity of alumina samples with different surface roughness](image)

Figure 4-31 Measured emissivity of alumina samples with different surface roughness (from [181]).

### 4.3.2 Specimen Preparation

Two types of specimens were used in the thermal shock tests. One type was alumina-aluminium specimens, described above (see also Table 4-1). They were cut in the shape of beams (see Fig. 4-3) out from the plate (Fig. 4-1) using the Cut-off Machine (Fig. 4-2). Another type was alumina-titanium specimens, which were manufactured by the
Engineering Research Centre in Biomaterials, Sichuan University, China. Their properties and dimensions are given in Tables 4-6 and 4-7. For this type of specimens, α-alumina powders were deposited on beam-shaped titanium substrates by a thermal plasma spray process. Preparation processes for the later specimens, such as polish and observation, are same as that described in Section 4.2.1. Figure 4-32 shows an example of the specimens.

Table 4-6 Properties of alumina-titanium specimens provided by the manufacturer.

<table>
<thead>
<tr>
<th>Coating</th>
<th>Substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Alumina:</td>
<td>Titanium (ISO 5832/11):</td>
</tr>
<tr>
<td>Particle size distribution:</td>
<td>Ultimate tensile strength &gt; 345 MPa;</td>
</tr>
<tr>
<td>98-154 μm (for 10% porosity);</td>
<td>Yield strength &gt; 230 MPa;</td>
</tr>
<tr>
<td>14-98 μm (for 7% porosity);</td>
<td>Elongation &gt; 20%;</td>
</tr>
<tr>
<td>&lt;14 μm (for 4% porosity).</td>
<td>Young's modulus: 110 GPa</td>
</tr>
<tr>
<td>Melting point = 1750°C</td>
<td>Melting point = 2000°C</td>
</tr>
</tbody>
</table>

Figure 4-32 An example of the specimen with alumina coating.
Table 4-7 Dimensions and properties of test specimens.

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Length $L$ (mm)</th>
<th>Width $W$ (mm)</th>
<th>Coating porosity level</th>
<th>Coating thickness level $H_1$ (mm)</th>
<th>Substrate thickness $H_2$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina-aluminium</td>
<td>30</td>
<td>3</td>
<td>&lt;2%</td>
<td>0.25</td>
<td>5.15</td>
</tr>
<tr>
<td>Alumina-titanium</td>
<td>30</td>
<td>3</td>
<td>Level A (&lt;4%)</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.40</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.50</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.65</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.80</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Level B (&lt;7%)</td>
<td>0.35</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Level C (&lt;10%)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4.3.3 Experimental Set-Up and Procedures

The experimental set-up for the laser-induced thermal shock tests is shown in Fig. 4-33 (a) and (b). It comprises the 1.2 kW CO$_2$ laser machine, the thermal imaging system, an alumina coating specimen and a hollow support.

![Figure 4-33 (a) Scheme of laser-induced thermal shock tests.](image-url)
Chapter 4. Experimental results

Figure 4-33 (b) Experimental set-up for laser-induced thermal shock tests.

The operating procedure, used in experiments, is as follows:

1) Set up a specimen so that the laser beam can be easily projected onto the top surface of the coating.

2) Set up the FLIR ThermaCAM SC3000 system so that the camera can focus on the central area (see Fig. 4-33(a)) of the coating and the full temperature distribution in the coating can be measured.

3) Set the laser power as required.

4) Assure that the FLIR THERMACAM SC3000 system is ready. Switch on the system.

5) Switch on the laser machine and heat the top surface of the coating only once, for a required time (0.6 ~ 5 s).

6) Allow the specimen to cool in ambient air for some time.

7) Use a glove and / or tongs to handle the specimen into a ready bag, mark an ID number without scratching the cross section surface of the specimen coating.

8) Repeat step 1) to 7) until all the test specimens are tested.
4.3.4 Experimental Results and Discussion

Total 34 alumina-titanium specimens were tested in experiments in which a single pulse of the CO₂ laser was used to initiate a thermal shock on the top surface of the coatings. In all the tests, the ambient temperature was 20°C. The distance between the specimen and the camera was 100 mm and the relative humidity of the air was 50%. The view angle of the camera was 15°. Full length of each specimen was 30 mm. The coating thickness and applied laser power for each specimen during heating are shown in Table 4-8. Duration of laser heating was controlled not to exceed 3 s and the heating was stopped if the maximum temperature of a specimen was larger than the melting point of the alumina coating. Only in one (S9) of the 34 specimens, a macrocrack crossing the coating was found in the central area. The coating in four specimens spalled during the heating.

<table>
<thead>
<tr>
<th>ID</th>
<th>Coating Porosity</th>
<th>Coating thickness (mm)</th>
<th>Duration of laser heating (s)</th>
<th>Laser power (kW)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>4%</td>
<td>0.10</td>
<td>2.57</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S2</td>
<td></td>
<td>0.12</td>
<td>2.29</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S3</td>
<td></td>
<td>0.16</td>
<td>1.70</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S4</td>
<td></td>
<td>0.16</td>
<td>1.81</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S5</td>
<td></td>
<td>0.24</td>
<td>2.04</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S6</td>
<td></td>
<td>0.25</td>
<td>2.66</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S7</td>
<td></td>
<td>0.28</td>
<td>2.50</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S8</td>
<td></td>
<td>0.30</td>
<td>2.37</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S9</td>
<td></td>
<td>0.36</td>
<td>1.03</td>
<td>0.95</td>
<td>Macro crack</td>
</tr>
<tr>
<td>S10</td>
<td></td>
<td>0.40</td>
<td>2.00</td>
<td>0.85</td>
<td></td>
</tr>
<tr>
<td>S11</td>
<td></td>
<td>0.40</td>
<td>1.37</td>
<td></td>
<td>Spalled</td>
</tr>
<tr>
<td>S12</td>
<td></td>
<td>0.44</td>
<td>2.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S13</td>
<td></td>
<td>0.44</td>
<td>1.71</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S14</td>
<td></td>
<td>0.48</td>
<td>No record</td>
<td></td>
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</tr>
<tr>
<td>S15</td>
<td></td>
<td>0.48</td>
<td>2.42</td>
<td></td>
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</tr>
<tr>
<td></td>
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<td>---</td>
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<td>---</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S16</td>
<td>0.50</td>
<td>1.49</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S17</td>
<td>0.55</td>
<td>1.00</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S18</td>
<td>0.55</td>
<td>1.00</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S19</td>
<td>0.68</td>
<td>1.37</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S20</td>
<td>0.70</td>
<td>1.11</td>
<td>1.2</td>
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<tr>
<td>S21</td>
<td>0.72</td>
<td>1.42</td>
<td>0.85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S22</td>
<td>0.72</td>
<td>0.85</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S23</td>
<td>0.72</td>
<td>No record</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S24</td>
<td>0.28</td>
<td>1.44</td>
<td>0.85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S25</td>
<td>0.28</td>
<td>1.50</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S26</td>
<td>0.18</td>
<td>3.01</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S27</td>
<td>0.18</td>
<td>2.85</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S28</td>
<td>0.20</td>
<td>1.01</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S29</td>
<td>0.22</td>
<td>2.51</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S30</td>
<td>0.24</td>
<td>0.61</td>
<td>0.85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S31</td>
<td>0.24</td>
<td>1.50</td>
<td></td>
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<tr>
<td>S32</td>
<td>0.26</td>
<td>1.45</td>
<td></td>
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</tr>
<tr>
<td>S33</td>
<td>0.28</td>
<td>2.50</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S34</td>
<td>0.34</td>
<td>0.95</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **Effect of thickness**

Evolution of the maximum temperature on the specimens' top surface for typical specimens with different thickness of the coating is shown in Fig. 4-34(a). A general trend in this figure is obvious: for the same power level, the top surface temperature rises faster for the thicker coatings (Fig. 4-34(b)). Obviously, due to the increase in the coating thickness, heat transfer in the coatings was additionally hindered during laser heating. Hence, the heat at the top surface was accumulated rapidly leading to the temperature rise. Fig. 4-35 shows a typical whole field temperature distribution in a specimen S1 after the top surface was heated with the laser to a maximum temperature of 1200°C. A development of the temperature distribution along the line L101 in Fig. 4-35 with time is shown in Fig. 4-36. Generally, the heat flux distribution provided by the laser is close to the Gaussian distribution. It can be seen from Fig. 4-36 that the initial temperature
distributions, which are close to the Gaussian ones (at time $t_0$ and $t_0+0.6$ agree) lose their symmetry with time, with local fluctuation appearing near the edges of the laser spot area (diameter 12 mm). This phenomenon has been found in most tested specimens. It suggests that rapid damage evolution at these locations during laser heating considerably affects material properties, e.g., thermal conductivity, with the rise in the temperature. Based on these data, some thermal properties of the alumina coating and some parameters of the Gaussian distribution can be determined (see Chapter 8 for further discussions).

Figure 4-34 (a) Evolution of the measured maximum temperature on the top surface for coatings with different thickness (alumina-titanium), laser power $P_L=0.85$ kW.
Chapter 4. Experimental results

Figure 4-34 (b) Effect of thickness on maximum temperature on top surface after 1.0 s laser heating.

Figure 4-35 A typical temperature distribution in a specimen (S1) with maximum temperature 1200 °C on its top surface.
Chapter 4. Experimental results

Figure 4-36 Evolution of the temperature distribution versus time along line L101 in Fig. 4-35.

- **Effect of porosity**

  Figure 4-37 demonstrates evolution of the maximum temperature on the coating top surface for specimens (alumina-titanium) with different porosity levels. The three specimens have porosity levels 4% (S7), 7% (S24) and 10% (S33), respectively, while they have same coating thickness 0.28 mm and they were exposed to similar heating conditions with laser power $P_L = 0.85$ kW. It can be seen from this figure that there are obvious differences in temperature rate for tested specimens. The increase in the porosity level of the coating is linked to higher temperature rates. This is due to the increase in thermal conductivity of the coatings with the increase in the coating porosity level. A relatively small change in porosity of the coating may lead to considerable changes in temperature on their surface under conditions of the high temperature thermal shock.
Figure 4-37 (a) Evolution of the maximum temperature on the top surface versus time for the coatings with different porosity (alumina-titanium), $P_L=0.85$ kW; (b) Effect of porosity on maximum temperature on the top surface after 1.0 s laser heating.
• **Spallation of the coatings**

It is found that three out of four spalled alumina-titanium specimens were all heated with higher power laser (1.2 kW) and another one was with power 0.85 kW. They have thickness 0.40, 0.68, 0.72 and 0.72 mm, respectively, for S11, S19, S22 and S23. Most values of thickness are generally larger than that of the majority of specimens. The instant of spallation of the coating in specimen S23 is registered in Fig. 4-38. The change in the recorded top surface temperature with heating time for three specimens (S11, S19 and S22) is shown in Fig. 4-39.

![Figure 4-38 An instant of spallation of a coating due to laser heating (specimen S23, $P_L=1.20$ kW).](image)

![Figure 4-39 Evolution of the maximum temperature on the top surface before spallation.](image)
Figure 4-38 indicates that, during laser heating, at first, a vertical crack (perpendicular to the interface between the coating and substrate) is initiated in the central area of the laser spot, followed by delamination along the interface between the coating and the substrate, and, finally, the coating breaks at the edge of the spot area. It can be seen from Fig. 4-38 and 4-39 that the four specimens spall when the maximum temperature in the coatings exceeds 1000°C. For the two spalled specimens (S19, S22) in Fig. 4-39, their top surface temperature before spallation is close to the melting point 1750°C of the alumina coating, while the respective temperature is just about 1200°C for specimen S11.

The temperature distributions in this specimen (S11) just before and after spallation are shown in Fig. 4-40. The time difference taking Figs. 4-40(a) and Fig. 4-40(c) during the experiment was 6 ms. Fig. 4-40(b) shows temperature distribution along the line in Fig. 4-40(a). Figure 4-40 vividly demonstrates that the temperature distribution around the edge of laser spot area had been different from the Gaussian distribution. Actually, all the four specimens failed in the same manner. This can be explained in the following way: vertical cracks through the coating are initiated near the edges and centre of the laser spot area and, subsequently, interface cracks (parallel to the interface) under the vertical crack near the centre area propagate to the edge of the spot area. In addition, for the four specimens (see Table 4-8) with larger thickness (around 0.70 mm) and heated by higher power laser (1.2 kW), three out of them (S19, S22 and S23) spalled during the tests. For the other specimens, only one of them (S11) spalled. This indicates that there exists a thickness limit of the alumina coatings under a higher heat flux loading in this study. Once the thickness of the coatings is larger than this limit, thermal stresses induced by the loading in the coatings may lead to spallation of them.

At last, coalescence of the cracks leads to the spallation of the specimen as shown in Fig. 4-40(c). This explanation is confirmed by analysis of micrographs (see Fig. 4-41) of alumina-titanium specimen S12 that shows the situation before spallation. At the edge of the laser spot area in the coating, the size of microvoids in alumina after laser heating (Fig. 4-41(b)) looks larger than that before heating (Fig. 4-41(a)), while there are no evident differences in other parts of the coating.
Figure 4-40 Temperature distribution in specimen S11: (a) spatial distribution just before spallation (time $t_0$); (b) distribution along the line L101 in (a) at time $t_0$; (c) spatial distribution just after spallation (time $t_0 + 0.006 \text{s}$).
Figure 4-41 Micrograph of alumina coating near the edge of the laser spot area (a) before and (b) after laser heating in specimen S12.
• **Cracking of a coating**

In 34 tested specimens exposed to laser-induced heating, cracks, other than spallation, were only found in one of them (S9). The thickness of this specimen was 0.36 mm and its porosity level was 4%. It was heated by the laser with power 0.95 kW, which was larger than that used in other specimens with similar thickness and porosity and thus was considered to be the main cause leading to the cracks. Two vertical surface cracks (perpendicular to the top surface) were found at the centre of the laser spot area and each was accompanied by an interface crack. The distance between the two cracks was about 3.7 mm. An example of the surface crack and interface crack is shown in Fig. 4-42. The surface crack propagates through the coating and thus has nearly the same length as the coating thickness (0.36 mm), while the interface crack is just under the surface crack and at the interface or above it between the coating and the substrate. From above results, it can be concluded that mismatch stresses induced by laser heating were not large enough to cause fracture in the coatings, especially in the case of thinner coatings (~0.5 mm) and lower laser power (~0.85 kW). The damage and fracture evolution in alumina coatings are related not only to external loading and coating properties but also to substrate properties and their thickness.

![Cracks induced by laser heating at cross-sectional surface of an alumina coating (S9).](image-url)
Chapter 4. Experimental results

- Thermal loading behaviour of alumina-aluminium specimens

Alumina-aluminium specimens (see Tables 4-1 and 4-6) were also heated by the laser to study the temperature distribution and crack behaviour under transient thermal shock. The coating thickness of the specimens is about 0.25 mm and the coating porosity is less than 2%. Due to the lower melting point (~660°C) of aluminium, the specimens were heated by the laser with power 500 W for 3 to 5 s so that the temperature of the aluminium substrate was less than the melting point during heating. Figure 4-43 shows the evolution of measured maximum temperature on the top surface for the coatings of 5 specimens (AL01 to AL05). During the laser heating, the temperature rises swiftly to about 350°C and then ascends gradually. This is due to the effect of temperature on thermal conductivity, which has larger values at higher temperature for the alumina coatings.

![Figure 4-43 Evolution of the measured maximum temperature on the top surface of coatings (alumina-aluminium).](image)

Figure 4-43 Evolution of the measured maximum temperature on the top surface of coatings (alumina-aluminium).
Chapter 4. Experimental results

After the heating, each specimen was examined under a microscope. For most of them, no evident surface cracks were found while the microstructure of the coatings looks denser. For the specimen (AL04), some damage phenomena (continuous microcracks) were observed as shown in Fig. 4-44. The maximum temperature at the coating of this specimen is around 707°C and the damage is located at the edge of the laser spot area. It is considered to be a trend line for probable crack propagation in the coating.

![Figure 4-44 Damage/crack trend line in a coating after laser heating (alumina-aluminium).](image)

In another specimen (AL05), in which the aluminium substrate demonstrated some signs of melting, a few microcracks were found (Fig. 4-45). They were near to at the interface between the coating and the substrate but not exactly on it. This phenomenon was also found in another type of the coating (Fig. 4-42(a)). It suggests that damage induced by the heating has the highest value in that area, due to either stress concentration or weaker values than the bonding strength (further discussion in Chapter 8).
Chapter 4. Experimental results

4.3.5 Summary

- Time-dependent temperature distributions in alumina coatings under the laser-induced thermal shock were obtained. These results can serve as a reference for numerical simulations in the following Chapters.

- Cracks, induced by heating in the coating, generally were initiated at the centre of the laser spot area, propagated along the interfaces between the coatings and the substrates and caused breakage at the edge of the area. The crack paths were generally irregular.

- The thickness and porosity of the coatings had significant effects on their loading behaviour under thermal loading. Thermal resistance of the coatings increased with the increase in thickness or the porosity level. However, thicker coatings easily spalled during heating.

- Microstructures of the cross-sectional surface of the alumina coating before and after laser-induced heating were analysed. High heat flux heating was found to cause changes in the material, probably leading to a denser material around the heating spot and to damage/fracture near the edges of the area of heating. Induced microcracks were found near to the coating-substrate interface not exactly at this interface.
5.1 Overview

Plasma sprayed ceramic TBCs demonstrate porous lamellar microstructures consisting of elongated and flat splats (see Fig. 3-4). Various types of microvoids/pores/cracks can exist in coatings due to the nature of manufacturing processes of their deposition. These attributes greatly influence the effective mechanical properties of coatings. In general, the inhomogeneous microstructure reduces the overall stiffness, strength and integrity of coatings. Plasma sprayed TBCs are also anisotropic, which increases complexity of their mechanical characterization. A number of experiments have been made to measure the effective, or average, properties of coatings. For plasma sprayed coatings, the measured values of these properties of coatings made with the same feed materials have shown large variations, depending on the fabrication processes. Also, measurements have yielded inconsistent results, even with similar specimens.

In the first section of this chapter, image analysis for plasma sprayed alumina coating is firstly used to determine some parameters such as porosity and voids density. Then, details of microstructure are characterised by the introduced parameters – shape, size, orientation and number of microdefects – defined in a reference volume. Statistical distributions of these parameters are proposed by means of image analysis and the quantitative evaluation [130]. In the second section, micromechanical models are described, used to determine effective properties of the modelled microstructure of alumina coatings in terms of analytical methods. Effects of the microstructure on mechanical properties of alumina coatings are investigated in the next section. Especially, the results from the models are compared to those from the nano-indentation tests and bending tests described in Chapter 4. At last, some conclusions are given.
5.2 Modelling Microstructure of Ceramic Coatings

5.2.1 General Presentation of Microstructure of Ceramic Coatings

In terms of the review in Chapter 3, the basic structural unit of thermally sprayed ceramic coatings is considered as an ellipsoid-shaped splat. Various types of microvoids are present in the splats or along boundaries between them. Due to effects of the splat boundaries, microvoids and even of some impurities, microstructures of ceramic coatings are very different from those of corresponding bulk materials. Such layered microstructure leads to the transversely isotropic properties of ceramic coatings and their moduli are usually lower than those of bulk materials.

Microvoids are also termed as voids, microcracks, microdefects, pores etc., depending on their aspect ratios (ratio of the major axis over the minor axis). In this chapter, these defects are collectively treated as microvoids and considered to be the main factors affecting the material’s properties. Microvoids exist in coatings at random locations and their interaction is neglected on the assumption that the characteristic interaction length is smaller than dimensions of elements and spacing between the neighbouring microvoids. Thus, to describe microstructure of ceramic coatings, the following parameters are introduced:

- **Porosity**

Porosity $p$ is generally defined as a volume or an area fraction of microvoids in ceramic coatings. In this study, the following relation is used:

$$p = \frac{1}{V} \sum_{i=1}^{n} V_i \quad \text{or} \quad p = \frac{1}{A} \sum_{i=1}^{n} A_i ,$$

(5-1)

Where $V$ and $A$ is a volume or an area of a reference cross-sectional surface of a coating containing $n$ microvoids, $V_i$ and $A_i$ are volumes or areas of individual microvoids.

- **Number and voids density**

Voids density $\rho_a$ is defined as an average number of voids in a unit area and can be determined by micrograph analyses. The total number $N_a$ of microvoids in the area $A$ is

$$N_a = \rho_a A .$$

(5-2)

- **Shape and size**
Experimental observations of the microstructure of ceramic coatings in Chapter 4 indicate that microvoids in the coatings generally present irregular shapes, while the data that can be obtained from the observations are areas, perimeters, maximum and minimum dimensions of them. Thus, the shape of microvoids is considered as an ellipse, which can be characterised using lengths of its major and minor axes.

- **Orientation**

  In terms of the quantitative evaluation of microvoids in plasma sprayed alumina coatings [10], the orientation angle between the major axis of microvoids and the spray direction ranges randomly from $-20^\circ$ to $20^\circ$. Such microstructure results in the difference in the Young's moduli in the plane, parallel to the substrate-coating interface, and along the perpendicular to this plane (deposition direction). Accordingly, in this chapter, all microvoids are assumed to have an orthotropic attribute.

  Thus, these parameters – porosity, voids density/number, shape, size and orientation of microvoids – characterize the microstructure of ceramic coatings in this chapter. Effective properties of the coatings are significantly affected not only by the average values but also by distributions of parameters, which are further determined by means of image analyses for micrographs of coatings, described in the following section.

### 5.2.2 Image Analyses and Statistical Distribution of Microvoids in Ceramic Coatings

Image analyses for 10 cross-sectional micrographs (each 260 μm x 200 μm) of plasma sprayed alumina coatings in the alumina-aluminium specimens (see Table 4-1) were made using the software package ImageC (AQUINTO Co., Germany). Figure 5-1 shows a typical example of such micrographs. Areas of 5252 microvoids in the micrographs were measured. The average voids density, porosity (area fraction) and size of microvoids in the coatings were obtained, shown in Table 5-1 in comparison to the results from another image analysis [130] of plasma sprayed alumina coating. It can be seen from the data that there is no obvious difference in voids density for alumina coatings with different porosity (less than 10%), while the void size for the coating with lower porosity is by 60% lower than that for the coating with higher porosity.
Figure 5-1 A micrograph of the cross-sectional surface of a plasma sprayed alumina coating.

Table 5-1 Comparison of results from image analyses for plasma sprayed alumina coatings with two different porosity levels.

<table>
<thead>
<tr>
<th></th>
<th>Present image analysis</th>
<th>Another image analysis [130]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>1.76%</td>
<td>9.5%</td>
</tr>
<tr>
<td>Voids density (μm$^{-2}$)</td>
<td>0.0101</td>
<td>0.0099</td>
</tr>
<tr>
<td>Size of most microvoids (μm)</td>
<td>0 ~ 16</td>
<td>0 ~ 27</td>
</tr>
<tr>
<td>The largest size of microvoids (μm)</td>
<td>30</td>
<td>50</td>
</tr>
</tbody>
</table>

Then, based on the data extracted from the analysis [130], a shape-size probability distribution for microvoids in plasma sprayed alumina coatings with different porosity is
determined as shown in Table 5-2 and Fig. 5-2. All microvoids are divided into $n_s=28$ discrete types in terms of the shape and size obtained from the image analyses. The shapes of microvoids are divided into 10 levels using a shape factor $S_f = 1 - \left( \frac{b}{a} \right)^2$, with its extreme values 0 and 1, corresponding to circular voids and cracks, respectively. The size $a$ (length of a major axis, see Fig. 5-3) of elliptical voids is divided into 9 levels starting from less than 3 $\mu$m up to more than 27 $\mu$m. The size $b$ is a length of a minor axis of the voids, determined by the size $a$ and the respective factor. Probability $p_i$ of each type of voids is determined as

$$p_i = \frac{\rho_i}{\rho_s},$$

where $\rho_i$ is a density of the $i$-th type of voids (defined as a number of voids in a unit area) and $\rho_s = \sum_i \rho_i = 0.01 \mu m^2$ is the total void density of the coating.

Table 5-2 Voids types of alumina coating with porosity 9.5%.

<table>
<thead>
<tr>
<th>Voids type ID</th>
<th>Voids density ($\times 10^4 \mu m^2$)</th>
<th>Voids size (\mu m)</th>
<th>Shape factor</th>
<th>Probability</th>
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<tr>
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<td>0.0189</td>
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<tr>
<td>2</td>
<td>1.37</td>
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<td>0.11</td>
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<tr>
<td>3</td>
<td>2.73</td>
<td>3.0</td>
<td>0.22</td>
<td>0.0284</td>
</tr>
<tr>
<td>4</td>
<td>4.10</td>
<td>3.0</td>
<td>0.33</td>
<td>0.0425</td>
</tr>
<tr>
<td>5</td>
<td>4.10</td>
<td>3.0</td>
<td>0.44</td>
<td>0.0425</td>
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<tr>
<td>6</td>
<td>2.50</td>
<td>3.0</td>
<td>0.56</td>
<td>0.0260</td>
</tr>
<tr>
<td>7</td>
<td>2.50</td>
<td>3.0</td>
<td>0.67</td>
<td>0.0260</td>
</tr>
<tr>
<td>8</td>
<td>2.28</td>
<td>3.0</td>
<td>0.78</td>
<td>0.0236</td>
</tr>
<tr>
<td>9</td>
<td>2.73</td>
<td>3.0</td>
<td>0.89</td>
<td>0.0284</td>
</tr>
<tr>
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<td>3.0</td>
<td>0.98</td>
<td>0.1039</td>
</tr>
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</tr>
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<tr>
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<td>6.0</td>
<td>0.22</td>
<td>0.0059</td>
</tr>
<tr>
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<td>0.57</td>
<td>6.0</td>
<td>0.33</td>
<td>0.0059</td>
</tr>
<tr>
<td>15</td>
<td>0.57</td>
<td>6.0</td>
<td>0.44</td>
<td>0.0059</td>
</tr>
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</table>
## Chapter 5. Modelling Microstructure and Effective Properties of Ceramic Coatings

<table>
<thead>
<tr>
<th>16</th>
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<th>0.56</th>
<th>0.0083</th>
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<td>0.67</td>
<td>0.0083</td>
</tr>
<tr>
<td>18</td>
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<td>6.0</td>
<td>0.78</td>
<td>0.0059</td>
</tr>
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<td>19</td>
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</tr>
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<td>6.0</td>
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<td>9.0</td>
<td>0.89</td>
<td>0.0071</td>
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<td>21.0</td>
<td>0.98</td>
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</tr>
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<td>27</td>
<td>0.50</td>
<td>24.0</td>
<td>0.98</td>
<td>0.0052</td>
</tr>
<tr>
<td>28</td>
<td>0.25</td>
<td>27</td>
<td>0.98</td>
<td>0.0026</td>
</tr>
</tbody>
</table>

**Figure 5-2** A shape-size-probability distribution of microvoids in alumina coating with porosity 9.5%.
Chapter 5. Modelling Microstructure and Effective Properties of Ceramic Coatings

Figure 5-3 Scheme of microvoids in coating.

For a transversely isotropic coating with total porosity $p$, the relation

$$p = \frac{4\pi \sum_{i=1}^{n_e} (a_i^2 b_i \rho_{vi})}{3}$$  \hspace{1cm} (5-4)

holds, which means that a variation in the porosity level affects both the length of major and minor axes of microvoids in a case of the constant void density in this study. It is assumed that the variation affects the lengths of the two axes, i.e., the microvoids size $a_i$, $b_i$ and $S_{Gi}$ of the $i$-th type of microvoids are determined as

$$a_i = a_{0i} \left( \frac{p}{p_0} \right)^{\frac{1}{4}},$$

$$b_i = b_{0i} \left( \frac{p}{p_0} \right)^{\frac{1}{2}},$$

$$S_{Gi} = 1 - (1 - S_{f0i}) \left( \frac{p}{p_0} \right)^{\frac{1}{2}},$$  \hspace{1cm} (5-5)

where $a_{0i}$, $b_{0i}$ and $S_{f0i}$ are the sizes of $i$-th type (28 types) of microvoids and shape factors in the coating with porosity $p_0$. It can be seen from Eq. (5-5) that the variation affects more the length of the minor axis. In other words, microvoids in coatings with lower porosity are more flat in the spray direction. The shape factors in Fig. 5-4 show an example of their distribution for the alumina coating with porosity 1.76%.
5.2.3 Monte-Carlo Simulations Of Microstructure Of Ceramic Coatings

In numerical simulations, it is assumed that ceramic coatings with different total porosity have the probability distribution described in Section 5.2.2. Then, the number $N_{Ai}$ of the $i$-th type of microvoids in a representative area element (RAE) are determined by the Monte-Carlo simulations using the following procedure:

1) Divide the interval $(0, 1)$ into 28 sub-intervals shown in Table 5-3:

<table>
<thead>
<tr>
<th>Sub-interval</th>
<th>Probability range</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Delta_1$</td>
<td>$(0, p_1)$</td>
</tr>
<tr>
<td>$\Delta_2$</td>
<td>$(p_1, p_1 + p_2)$</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>$\Delta_i$</td>
<td>$(p_1 + p_2 + ... + p_{i-1}, p_1 + p_2 + ... + p_i)$</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>
In Table 5-3, $p_i$ is the probability of each shape-size type (28 types) of microvoids from the discrete distribution described in Section 5.2.2 and Eq. (5-3).

2) Determine overall number of microvoids $N_{all}$ in the RAE in terms of the Eq. (5-2).

3) Sample number $r$ with a uniform random distribution between $0.0$ and $1.0$.

4) If the number $r$ falls in the sub-interval $\Delta_i$ ($1 \leq i \leq n_{ss}=28$), the sample – the simulated microvoid – belongs to the $i$-th type of microvoids.

5) Repeat steps 3 and 4 $N_{all}$ times ($N_{all} = \sum_{i=1}^{n_{ss}} N_{Ai}$). Then, voids density of the $i$-th type of microvoids in the RAE is

$$\rho_{Ai} = \frac{N_{Ai}}{A}.$$  \hspace{1cm} (5-6)

It is assumed that a representative volume element (RVE) has same porosity as the corresponding RAE and the volume of the RVE is $V=A^{3/2}$. Then, the actual voids density in the RVE is

$$\rho_{vi} = \frac{3\rho_{Ai} \sum_{j=1}^{n_{ss}} \left( a_j b_j \rho_{Nj} \right)}{4 \sum_{j=1}^{n_{ss}} \left( a_j^2 b_j \rho_{Nj} \right)}.$$  \hspace{1cm} (5-7)

**5.2.4 Simulation of Microstructure of Sub-Elements in a Representative Element**

Considering that the actual number of microvoids of the $i$-th type in a sub-element of the RAE, which can be considered as a local region in Section 6.5, may deviate from the number $N_{Ai}$, probability of a sub-element containing exactly $M_i$ (from 0 to 10 or more) microvoids is given by the Poisson distribution

$$P(M_i) = \frac{N_{Ai}^{M_i} \exp(-N_{Ai})}{M_i!}.$$  \hspace{1cm} (5-8)

It is worth mentioning that an introduction of the Poisson distribution into description of the crack size and strength of materials in fracture mechanics leads to the Weibull
distribution [4], which has been widely applied in statistical fracture analysis for brittle materials. Then, an exact number of the \( i \)-th type microvoids \( n_{ip} \) in each sub-element of the local region is also determined by the Monte-Carlo simulations based on the similar procedure, described above, but with the following difference: probability \( p_i \) is determined in terms of the Poisson distribution, Eq. (5-8).

### 5.3 Micromechanical Modelling – Effective Properties of Ceramic Coatings

Based on the microstructures modelled in the last section, effective parameters (Poisson’s ratio \( \nu \) and Young’s modulus \( E_{ex} \) and \( E_{eq} \) in transverse direction and \( E_{eq} \) in spray direction, see Fig. 5-3) of an element for materials with a transversely isotropic distribution of elliptical voids can be explicitly expressed in terms of the number of microvoids, porosity \( P \) and the crack density (hole density tensor \( \beta \) [47, 182]) as following:

\[
E_{e1} = E_{e2} = E_0 \left[ 1 + \frac{32(1-\nu_i^2)}{3(2-\nu_0)} \frac{\beta_{1i}}{(1-p)} + \frac{3(1-\nu_0)(9+5\nu_0)}{2(7-5\nu_0)} \frac{p}{(1-p)} \right]^{-\frac{1}{3}},
\]

\[
E_{e3} = E_0 \left[ 1 + \frac{32(1-\nu_i^2)}{3(2-\nu_0)} \frac{\beta_{3i}}{(1-p)} + \frac{3(1-\nu_0)(9+5\nu_0)}{2(7-5\nu_0)} \frac{p}{(1-p)} \right]^{-\frac{1}{3}},
\]

\[
G_{e12} = G_0 \left[ 1 + \frac{32(1-\nu_0)}{3} \frac{\beta_{1}}{(1-p)} + \frac{15(3-\nu_0)}{2(7-5\nu_0)(1-p)} \frac{p}{(1-p)} \right]^{-\frac{1}{3}},
\]

\[
G_{e13} = G_{e23} = G_0 \left[ 1 + \frac{32(1-\nu_i^2)}{3(2-\nu_0)} \frac{\beta_{1i} + \beta_{3i}}{(1-p)} + \frac{15(3-\nu_0)}{2(7-5\nu_0)(1-p)} \frac{p}{(1-p)} \right]^{-\frac{1}{3}},
\]

\[
\frac{v_{e13}}{E_{e1}} = \frac{v_{e31}}{E_{e3}} = \frac{v_0}{E_0} \left[ 1 + \frac{3(1-\nu_i)(1+5\nu_0)}{2\nu_0(7-5\nu_0)} \frac{p}{(1-p)} \right],
\]

where \( x \rightarrow 1, z \rightarrow 2, y \rightarrow 3 \), \( p = \frac{1}{V} \sum_{k=1}^{3} \left( a_k^2 b_k N_k \right) \), \( \beta = \frac{1}{V} \sum_{k=1}^{3} N_k \left( a_k^2 n + b_k m + c_k l \right) \), \( V \) is the total volume, \( N_k \) is the number of the \( k \)-th type of voids in the volume \( V \), \( a_k \) and \( b_k \) are lengths of the major and minor axes of elliptical voids, \( n, m \) and \( l \) are unit normals to these axes, \( E_0, G_0 \) and \( v_0 \) are the Young’s modulus, shear modulus and Poisson’s ratio of the undamaged material (assumed to be isotropic), respectively.
The effective thermal conductivities, for 3D transversely isotropic case ($k_{ex}$ and $k_{ez}$ in transverse direction, $k_{ey}$ in spray direction), of ceramic coatings are expressed as [11, 139, 182]

$$k_{ex} = k_{ez} = k_0 \left[1 + \frac{8 \beta_{11}}{3(1-\rho)} + \frac{3}{2} \frac{1}{(1-\rho)}\right]^{-1},$$  

$$k_{ey} = k_0 \left[1 + \frac{8 \beta_{33}}{3(1-\rho)} + \frac{3}{2} \frac{1}{(1-\rho)}\right]^{-1},$$  

where $k_0$ is the thermal conductivity of the undamaged material.

### 5.4 Results and Discussion

In terms of the proposed micromechanical models in above sections, numerical simulations were conducted to investigate the effect of microstructure on the effective properties of the alumina coating (see Table 4-1). A flow chart of the simulations is shown in Fig. 5-5.

![Flow chart of microstructural simulation of ceramic coating](image)
100 random RVEs were constructed to obtain statistical results for the effective properties. The RVEs had average porosity 1.8%, the uniform dimension 80 µm, and microvoids in each of them had the distribution shown in Fig. 5-4. The Poisson's ratio was set as $\nu = 0.27$ (also used in computations below). The density of each type of microvoids in each RVE may vary in terms of the Monte-Carlo simulation while the total density of the microvoids was uniform 0.01 µm$^2$. The computed effective properties of these RVEs are shown in Fig. 5-6 and their average values and standard deviations are shown in Table 5-4.
Figure 5-6 Normalized effective properties of alumina coating: (a) Young's modulus; (b) Poisson's ratio; (c) thermal conductivity.
Table 5-4 Average values of normalized effective properties of alumina coating with porosity 1.8%, RVE size 80 μm.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Direction (x direction)</th>
<th>Spray direction (y direction)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young's modulus $E_{ave}$</td>
<td>0.965±0.004</td>
<td>0.727±0.028</td>
</tr>
<tr>
<td>Poisson's ratio</td>
<td>0.262±0.000</td>
<td>0.215±0.003</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>0.974±0.003</td>
<td>0.843±0.018</td>
</tr>
</tbody>
</table>

A general trend revealed by this figure and table is that the properties in both directions decrease – as compared to the material without microvoids – due to the effect of microdefects, while the values in the spray direction have a larger reduction than those in the transverse direction. Elastic and thermal properties in the spray direction reduce by about 27% and 16%, respectively. This implies that microvoids have a somewhat higher effect on elastic properties. These results also indicate that the effective transverse modulus exhibits only relatively small variations. In contrast, fluctuations of the modulus in the spray direction are significantly more pronounced. This indicates that the modulus in the spray direction is more sensitive to details of the coating microstructure, which in real coatings can be explained by specificity of their porous lamellar coating microstructures containing elongated and flat splats in the plane of deposition.

These results are compared to those obtained from the nanoindentation tests (see Section 4.1.3, Fig. 4-10 and Table 4-3) and bending tests described in Chapter 4. In the nanoindentation tests, the Young's modulus of alumina coating is 140±35 GPa for the case of contact force 50 mN and indentation depth 555±92 nm. For such a depth, it is assumed that the effect of microvoids can be neglected and the value 140 GPa can be considered as the Young's modulus of the undamaged material $E_0$. Thus, the Young's modulus in the spray direction, calculated with the help of the suggested simulation scheme, is $140 \times 0.727 = 102$ GPa, which agrees well with the value 104±15 GPa obtained from the nanoindentation for the case of larger indentation depth 2195±481 nm. The calculated value of the modulus in the transverse direction is $140 \times 0.965 = 135$ GPa, which also agrees with the results from the three-point bending tests, 128±21 GPa (see Table 4-5 and Fig. 4-22).
Effect of RYE dimensions

In order to use the obtained effective properties as the homogenized data in continuum damage models, understanding of the effect of RYE dimensions is necessary. As it has been reviewed in Chapter 2, the dimensions of RYE should be large enough to include the effect of microvoids or microcracks but, at the same time, they also should be small enough for the macroscopic stress and strain state to be considered as homogeneous or only with a small inhomogeneity.

Thus, the effect of RVE dimensions as well as their minimum size is studied to obtain a consistent level of the moduli. Numerical computations of the effective Young's moduli are performed for five different dimensions of elements: 50, 80, 125, 200 and 250 μm. For each dimension, 100 random RVEs are generated, all having average porosity 1.8% and various distributions of microvoids. It can be seen from Fig. 5-6(a) that there is a scatter of the values for the case of dimension 80 μm and the standard deviation is 0.028, while the maximum relative scatter (\(\frac{\text{Max}(|E_i - E_{\text{ave}}|)}{E_{\text{ave}}}, i = 1, 2, 3, \ldots, 100\)) is 12.1%.

The results for other dimensions are shown in Table 5-5. The calculations with the varying RVE dimensions indicate that the average moduli of the RVEs with different dimensions are similar, while a decrease in the dimensions results in a considerable increase in the scatter of the values, especially, in the spray direction. These results suggest that the homogenization assumption in continuum models can not be used for the case of smaller RVE dimension, e.g., less than 80 μm when the porosity level is 1.8%.

Table 5-5 Average values of normalized effective Young's moduli of alumina coating with porosity 1.8% for different RVE dimensions.

<table>
<thead>
<tr>
<th>Dimension (μm)</th>
<th>Transverse direction (x direction)</th>
<th>Spray direction (y direction)</th>
<th>Relative scatter in y direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>0.964±0.008</td>
<td>0.724±0.055</td>
<td>16.7%</td>
</tr>
<tr>
<td>80</td>
<td>0.965±0.004</td>
<td>0.727±0.028</td>
<td>12.1%</td>
</tr>
<tr>
<td>125</td>
<td>0.965±0.003</td>
<td>0.723±0.020</td>
<td>8.2%</td>
</tr>
<tr>
<td>200</td>
<td>0.964±0.002</td>
<td>0.720±0.012</td>
<td>4.1%</td>
</tr>
<tr>
<td>250</td>
<td>0.964±0.001</td>
<td>0.718±0.010</td>
<td>3.1%</td>
</tr>
</tbody>
</table>
• **Effect of porosity**

The porosity level does not only affect the effective properties of ceramic coatings but also has a significant effect on the scatter of these values. Figure 5-7 shows variation of the average relative Young’s modulus in the transverse \((E_x/E_0)\), spray \((E_y/E_0)\) direction and the orthotropy ratio of the two moduli \((E_y/E_x)\) as well as the maximum relative error of \(E_y/E_0\) with the increase of the porosity level from 0.1% to 9.5%. Generally, the modulus in the transverse direction is larger than that in the spray direction, and the moduli in both directions decrease as porosity increases. However, the decrease is more evident in the spray direction than that in the transverse one. The orthotropy ratio decreases as well but the difference between the ratios turns smaller with the increase of porosity. These results are similar to those obtained by other researchers [10]. On the other hand, the relative scatter increases from 3.6% to 21.9% with the increase of the porosity. This means that there is a larger scatter in the material’s properties for the higher porosity of elements, and the minimum RVE dimension for lower porosity may not be applied for its higher values.

![Figure 5-7 Effect of porosity on effective properties of alumina coating.](image-url)
Effect of size of microvoids

In Fig. 5-6, the scatter in the effective properties is induced by the variation of the microvoids density and porosity during the Monte-Carlo simulations. For a given porosity level, it is possible to study directly the effect of size of microvoids on the effective properties. In the case of spherical voids, each type of microvoids has uniform aspect ratio, i.e., $\frac{b_k}{a_k} = 1$. From the definition of the porosity $p$ and the hole density tensor $\beta$ in Eqs. (5-9) to (5-12), it can be deduced that variation in the size of each type of microvoids in the coating (e.g., $a_k \rightarrow n a_k$) leads to a corresponding variation in the number of microvoids of this type ($N_k \rightarrow N_k/n$), while the parameter $\beta$ does not change. This means that variation in the size of microvoids in a RVE does not affect the material’s properties for the RVE under the condition that the porosity level and the spherical shape ($\frac{b_k}{a_k} = 1$) of each microvoid are given, neglecting mutual reaction between the microvoids.

5.5 Summary and Conclusion

- Microstructures of ceramic coatings are modelled based on the data obtained by the image analysis technique for optical micrographs of coatings.
- Effective properties such as the Young’s modulus, Poisson’s ratio and thermal conductivity are calculated in terms of the proposed microstructure modelling scheme. The data from simulations agree well with those obtained by nanoindentation and bending tests.
- The effect of microstructure on the material properties is investigated quantitatively and qualitatively in terms of statistical analysis. Microvoids affect the coating’s properties both in the spray and transverse directions, while the effects on the values in the former direction are more evident. With the increase in porosity, the properties reduce apparently. For the case of 1.8% porosity, the Young’s moduli of alumina coatings in the transverse and spray direction reduce by about 3.5% and 17.3%, respectively.
• With a decrease in RVE dimension, the scatter of the effective properties increases, especially in the spray direction. For the case of 1.8% porosity, the scatter rises from 3.1% to 16.7% with the dimension decreasing from 250 μm to 50 μm.
Chapter 6. Theory and Analysis of Damage and Fracture Evolution in Ceramic Coatings

6.1 Overview

This chapter proposes a novel theory of damage and fracture evolution in ceramic coatings. Firstly, basic assumptions are given and a multi-scalar of damage variable is defined for the case of multi-axial loading states. Subsequently, a constitutive relationship is proposed. Then, damage evolution and critical states of materials for the multi-axial loading states are analysed. Based on the suggested constitutive relationship, a framework of multiscale analysis for damage and fracture in ceramic coatings is proposed. These methods serve as a basis for numerical simulations described in the following chapters.

6.2 Basic Assumptions and Constitutive Relationship

In this chapter, undamaged (virgin) materials are assumed to be homogeneous at the macroscale. Defects, formed in the brittle material due to loading, are assumed to be penny-shaped microcracks or mesocracks. It is also assumed that only elastic damage occurs in the material, i.e., there is no plastic flux, permanent deformation, or any other energy dissipation mechanisms. Considering that the principal directions of stress and strain tensors coincide with the principal directions of damage, the problem could be considered in the principal coordinate system. Thus, the stress-strain relationship of an element with damage configuration in the principal direction can be expressed as

\[
\varepsilon = [C(D)] \cdot \sigma, \tag{6-1}
\]
Where $[\sigma]$, $[\varepsilon]$ and $[C(D)]$ are stress, strain and compliance matrices, respectively. The major principal strain $[\varepsilon]$ can be divided into three parts corresponding to the major principal stress $[\sigma]$ as following relation:

$$
\begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{bmatrix} = [C(D)] \begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3
\end{bmatrix} = [C(D)] \begin{bmatrix}
\sigma_1 \\
0 \\
0
\end{bmatrix} + \begin{bmatrix}
\sigma_2 \\
0 \\
0
\end{bmatrix}
$$

$$
= \varepsilon' + \varepsilon'' + \varepsilon'''
$$

In terms of strain equivalence principle and the damage definition (see Eqs. (2-1) and (2-2)), it can be obtained that: $\varepsilon' = \frac{\sigma_1}{E_1 (1-D)}$, $\varepsilon'' = -\frac{\nu_i D_j \sigma_j}{E_2 (1-D)}$ and $\varepsilon''' = -\frac{\nu_i D_j \sigma_j}{E_3 (1-D)}$, where $D_i$ ($i \neq j$) can be considered as effect of damage $D_i$ in the principal direction $i$ on the Poisson’s ratio. Similarly, $\varepsilon''$ and $\varepsilon'''$ can be obtained.

It is assumed that the undamaged material is isotropic and has the Young’s modulus $E_1 = E_2 = E_3 = E_0$ and Poisson’s ratio $\nu_0$. In the principal directions, the following relation holds:

$$
\begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{bmatrix} = \frac{1}{E_0} \begin{bmatrix}
1 & -\nu_0 D_{21} & -\nu_0 D_{31} \\
\nu_0 D_{21} & 1 & -\nu_0 D_{23} \\
\nu_0 D_{31} & \nu_0 D_{32} & 1
\end{bmatrix} \begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3
\end{bmatrix}
$$

$$
(6-3)
$$

or

$$
\begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3
\end{bmatrix} = [S(D)] \begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{bmatrix} = \begin{bmatrix}
1-D_1 & 1-D_2 & 1-D_3 \\
\nu_0 \frac{1-D_1}{D_{21}} & 1-D_2 & \nu_0 \frac{1-D_3}{D_{32}} \\
\nu_0 \frac{1-D_1}{D_{31}} & \nu_0 \frac{1-D_2}{D_{32}} & 1-D_3
\end{bmatrix} \begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{bmatrix}
$$

$$
(6-4)
$$

where $[S(D)]$ is a stiffness matrix.
6.3 Effective Damage and Its Evolution

6.3.1 One-Dimensional Damage Presentation and Evolution

For a uniaxial loading, from Eq. (6-3), there are

\[
\begin{bmatrix}
\sigma \\
\varepsilon_x \\
\varepsilon_y
\end{bmatrix}
= \frac{1}{E_0}
\begin{bmatrix}
1 - \nu_y D_1 & -\nu_y D_{21} & -\nu_y D_{31} \\
-\nu_y D_{21} & 1 - \nu_x D_2 & -\nu_x D_{12} \\
-\nu_y D_{31} & -\nu_x D_{12} & 1 - \nu_x D_3
\end{bmatrix}
\begin{bmatrix}
\sigma_1 \\
0 \\
0
\end{bmatrix}
= \frac{1}{E_0}
\begin{bmatrix}
\sigma_1 / (1 - D_1) \\
-\nu_y D_{12} \sigma_1 / (1 - D_2) \\
-\nu_x D_{12} \sigma_1 / (1 - D_3)
\end{bmatrix}
= \frac{1}{E_0}
\begin{bmatrix}
\sigma_1 \\
-\nu_y D_{12} \sigma_1 \\
-\nu_x D_{12} \sigma_1
\end{bmatrix}
\begin{bmatrix}
1 - D_1 \\
1 - D_2 \\
1 - D_3
\end{bmatrix}
\begin{bmatrix}
\sigma_1 \\
0 \\
0
\end{bmatrix}.
\]  

(6-5)

For uniaxial tensile loading of brittle elastic materials, the thermodynamic analysis [27, 85] provides the following form of the damage evolution law:

\[
D = D_0 \exp \left( \frac{W_{\text{ref}}}{W^*} \right), \quad D_0 = 1 - \frac{E^*}{E_0},
\]  

(6-6)

where the initial damage \( D_0 \) reflects the effect of the material’s initial state, \( E^* \) is an initial level of the effective Young’s modulus. The parameter \( W^* \) in the evolution law characterizes the material’s intake of energy at damage and is further denoted as damage energy absorption capacity of the material. Temperature-dependent values of \( W^* \) in a broad temperature interval have been determined from the experiments on bulk alumina, with initial damage \( D_0 \) being directly related to porosity. Strain energy of the undamaged material, undergoing the same loading history, is expressed as \( W_{\text{ref}} = \frac{1}{2} E_0 \varepsilon^2 \), \( \varepsilon \) is the major principal strain.

Failure of a material’s element is linked to the condition of the stress \( \sigma_1 \) reaching its maximum value \( \sigma_m \) and

\[
\frac{d\sigma_1}{d\varepsilon} \bigg|_{\sigma_m} = 0.
\]  

(6-7)

From the Eqs. (6-5) to (6-7), the maximum value \( D_m \) of the damage variable \( D \) can be determined as

\[
D_m \exp \left( \frac{D_m - 1}{2D_m} \right) = D_0.
\]  

(6-8)
6.3.2 Two-Dimensional Damage Presentation and Evolution

In principal directions for a 2-D case, the stress-strain relationship for plane stress condition is

\[
\begin{pmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{pmatrix} = \left[ C(D) \right] \begin{pmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3
\end{pmatrix} = \begin{pmatrix}
C_{11} & C_{12} & C_{13} \\
C_{12} & C_{22} & C_{23} \\
C_{13} & C_{23} & C_{33}
\end{pmatrix} \begin{pmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3
\end{pmatrix}
\]

\[
= \frac{1}{E_v} \begin{pmatrix}
\frac{1}{1-D_1} & -\nu_0 D_{31} & -\nu_0 D_{31} \\
-\nu_0 D_{21} & \frac{1}{1-D_2} & -\nu_0 D_{21} \\
-\nu_0 D_{31} & -\nu_0 D_{21} & \frac{1}{1-D_3}
\end{pmatrix} \begin{pmatrix}
\sigma_1 \\
\sigma_2 \\
0
\end{pmatrix},
\]

(6-9)

where the damage components \(D_i (i = 1, 2, 3)\) is considered to be induced by the major principal strain \(\varepsilon_i\). Then, Eq. (6-9) can be simplified as

\[
\begin{pmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3
\end{pmatrix} = \begin{pmatrix}
C_{11} & C_{12} \\
C_{21} & C_{22}
\end{pmatrix} \begin{pmatrix}
\sigma_1 \\
\sigma_2
\end{pmatrix} = \frac{1}{E_v} \begin{pmatrix}
\frac{1}{1-D_1} & -\nu_0 D_{31} \\
-\nu_0 D_{21} & \frac{1}{1-D_2} \\
\end{pmatrix} \begin{pmatrix}
\frac{1}{1-D_1} & -\nu_0 D_{31} \\
\nu_0 D_{21} & \frac{1}{1-D_2}
\end{pmatrix} \begin{pmatrix}
\sigma_1 \\
\sigma_2
\end{pmatrix}.
\]

(6-10)

In order to reduce the number of independent elastic constants, it is assumed that the Saint-Venant's relations hold exactly, i.e., \(\frac{1}{C_{12}} = C_{11} + C_{12} - 2C_{12}\), where \(G_{12}\) is the shear modulus. In terms of strain equivalence and the thermodynamic analysis [27], the damage components are defined as

\[
D_i = D_0 \exp \left( \frac{\sigma_i W_i}{W^*} \right),
\]

(6-11)

where \(W_i = \frac{1}{2} E_i \varepsilon_i^2\), \(D_0 = 1 - \frac{E_i^*}{E_0}\), \(E_i^*\) are the effective Young's moduli in the principal directions, \(c_i = \begin{cases} 1 & \text{when } \varepsilon_i \geq 0 \text{ (tension)} \\ -1 & \text{when } \varepsilon_i < 0 \text{ (compression)} \end{cases}\)

Considering symmetry and positiveness of the compliance matrix \([C(D)]\) in Eq. (6-9), for an orthotropic case:
Chapter 6. Theory and Analysis of Damage and Fracture Evolution in Ceramic Coatings

\[
\begin{align*}
\frac{1}{(1-D_i)(1-D_j)} & \geq \frac{\nu_2^2 D_{12} D_{21}}{(1-D_i)(1-D_j)}, \\
\frac{D_{12}}{1-D_i} &= \frac{D_{21}}{1-D_j},
\end{align*}
\] 

(6-12)

from which it can be deduced that \(\frac{D_j \nu_2}{1-D_i} \leq \frac{1}{\sqrt{(1-D_i)(1-D_j)}}\). Then it can be assumed that

\[D_j = (1-D_i) \delta_i \quad (i \neq j).\]

(6-13)

Comparing Eqs. (6-9) and (6-12) with Eq. (5-13) in Chapter 5, it can be obtained that

\[\delta_0 = \left[1 + \frac{3(1-\nu_0)(1+5\nu_0)}{2\nu_0 (7-5\nu_0)} \frac{p}{(1-p)}\right].\]

Thus, setting \(\nu = \nu_0 \delta_0\), Eq. (6-9) is transformed as

\[
\begin{pmatrix}
\epsilon_1 \\
\epsilon_2
\end{pmatrix} = \frac{1}{E_0} \begin{pmatrix}
\frac{1}{1-D_i} & -\nu(1-D_i) \\
\nu(1-D_i) & \frac{1}{1-D_j}
\end{pmatrix} \begin{pmatrix}
\sigma_1 \\
\sigma_2
\end{pmatrix}
\] 

(6-14)

or

\[
\begin{pmatrix}
\sigma_1 \\
\sigma_2
\end{pmatrix} = \frac{E_0}{1-\nu^2 (1-D_i)(1-D_j)} \begin{pmatrix}
1-D_i & \nu(1-D_i)(1-D_j) \\
\nu(1-D_i)(1-D_j) & 1-D_j
\end{pmatrix} \begin{pmatrix}
\epsilon_1 \\
\epsilon_2
\end{pmatrix}.
\]

(6-15)

The major principal stress is expressed as

\[\sigma_1 = \frac{E_0 (1-D_i)}{1-\nu^2 (1-D_i)(1-D_j)} \left[\epsilon_1 + \nu \epsilon_2 (1-D_j)\right].\]

(6-16)

Now the following conditions are considered:

1) When \(D_{01} = D_{02} = 0\), the element will never crack;

2) When \(D_{01} = 0\) and \(D_{02} \neq 0\), Eq. (6-16) can be transformed as

\[\sigma_1 = \frac{E_0}{1-\nu^2 (1-D_j)} \left[\epsilon_1 + \nu \epsilon_2 (1-D_j)\right].\]

(6-17)

However, \(\frac{\partial \sigma_1}{\partial \epsilon_1} \bigg|_m = \frac{E_0}{1-\nu^2 (1-D_j)} \neq 0\). This means that the element will never crack either.

3) When \(D_{01} \neq 0\) and \(D_{02} = 0\), Eq. (6-16) can be transformed as
\[ \frac{\partial \sigma_1}{\partial \varepsilon_1} \bigg|_{m} = \frac{E_0 (1 - D_1)}{1 - \nu^2 (1 - D_1)} - \frac{E_0 \left[ \epsilon_1 + \nu \varepsilon_2 \right] D_1 \frac{E_0 \varepsilon_1}{W^*} \varepsilon_1}{\left[ 1 - \nu^2 (1 - D_1) \right]^2} \]

\[ = \frac{E_0 (1 - D_1)}{1 - \nu^2 (1 - D_1)} \left[ (1 - D_1) - \frac{\left[ \epsilon_1 + \nu \varepsilon_2 \right] D_1 \frac{E_0 \varepsilon_1}{W^*} \varepsilon_1}{1 - \nu^2 (1 - D_1)} \right] = 0, \quad (6-18) \]

\[ \frac{\partial \sigma_1}{\partial \varepsilon_2} \bigg|_{m} = \frac{E_0 (1 - D_1) \nu}{1 - \nu^2 (1 - D_1)} = 0. \quad (6-19) \]

It can be considered that the element will crack when the damage measure \( D_1 = D_m = 1 \).

4) When \( D_{11} \neq 0 \) and \( D_{22} \neq 0 \), the major principal stress reaches its maximum value \( \sigma_1 = \sigma_m \), when \( D_1 = D_m \) and \( \frac{\partial \sigma_1}{\partial \varepsilon_1} = \frac{\partial \sigma_1}{\partial \varepsilon_2} = 0 \), i.e.,

\[ \frac{\partial \sigma_1}{\partial \varepsilon_1} \bigg|_{m} = \frac{E_0 (1 - D_1)}{1 - \nu^2 (1 - D_1)(1 - D_2)} - \frac{E_0 \left[ \epsilon_1 + \nu \varepsilon_2 \right] (1 - D_1) \frac{E_0 \varepsilon_1}{W^*} \varepsilon_1}{\left[ 1 - \nu^2 (1 - D_1)(1 - D_2) \right]^2} \]

\[ = \frac{E_0 (1 - D_1)}{1 - \nu^2 (1 - D_1)(1 - D_2)} \left[ (1 - D_1) - \frac{\left[ \epsilon_1 + \nu \varepsilon_2 \right] (1 - D_1) \frac{E_0 \varepsilon_1}{W^*} \varepsilon_1}{1 - \nu^2 (1 - D_1)(1 - D_2)} \right] = 0, \quad (6-20) \]

\[ \frac{\partial \sigma_1}{\partial \varepsilon_2} \bigg|_{m} = \frac{E_0 (1 - D_1) \nu}{1 - \nu^2 (1 - D_1)(1 - D_2)} \left[ \nu (1 - D_2) - \nu D_2 \frac{E_0 \varepsilon_2}{W^*} \varepsilon_2 \right] \]

\[ - \left[ \epsilon_1 + \nu \varepsilon_2 \right] \frac{E_0 \nu^2 (1 - D_1)^2 D_2 \frac{E_0 \varepsilon_2}{W^*} \varepsilon_2}{\left[ 1 - \nu^2 (1 - D_1)(1 - D_2) \right]^2} = 0. \quad (6-21) \]

It can be obtained from Eq. (6-11) that

\[ c_1 \frac{E_0 \varepsilon_1}{W^*} = 2 \ln \left( \frac{D_1}{D_0} \right), \quad (6-22) \]

\[ \frac{E_0 \varepsilon_1 \varepsilon_2}{W^*} = 2 c_1 \varepsilon_2 \sqrt{\ln \left( \frac{D_1}{D_{10}} \right) \ln \left( \frac{D_2}{D_{20}} \right)}, \quad (6-23) \]

Then, Eqs. (6-20) and (6-21) can be transformed as
Chapter 6. Theory and Analysis of Damage and Fracture Evolution in Ceramic Coatings

\[
(1-D) \frac{\left[ \varepsilon_1 + \nu \varepsilon_2 (1-D_2) \right] D_1 F_1 \frac{E_1 \varepsilon_1}{W_1}}{1-\nu^2 (1-D)(1-D_2)} = (1-D_m) - 2D_m \frac{\ln \left( \frac{D_m}{D_{01}} \right) + \nu (1-D) c_2 \sqrt{\ln \left( \frac{D_m}{D_{01}} \right) \ln \left( \frac{D_2}{D_{02}} \right)} }{1 - \nu^2 (1-D_m)(1-D_2)} = 0, \tag{6-24}
\]

\[
(1-D) \left\{ (1-D_2) - D_2 \frac{E_2 \varepsilon_2}{W_2} c_2 \right\} = (1-D) \frac{D_2 c_2}{1-\nu^2 (1-D)(1-D_2)} \frac{E_1 \varepsilon_1}{W_1} + \nu (1-D_2) \frac{E_2 \varepsilon_2}{W_2} \tag{6-25}
\]

One of the roots of the set of Eqs. (6-24) and (6-25) is \( D_m = 1 \) and another solution is:

\[
\begin{align*}
F_1 (D_m, D_2) &= (1-D_m) \left[ 1 - \nu^2 (1-D_m)(1-D_2) \right] - \\
2D_m \ln \left( \frac{D_m}{D_{01}} \right) + \nu (1-D) c_2 \sqrt{\ln \left( \frac{D_m}{D_{01}} \right) \ln \left( \frac{D_2}{D_{02}} \right)} = 0, \tag{6-26}
\end{align*}
\]

\[
\begin{align*}
F_2 (D_m, D_2) &= (1-D_2) \left[ 1 - \nu^2 (1-D_m)(1-D_2) \right] - \\
2D_2 \ln \left( \frac{D_2}{D_{02}} \right) + \nu (1-D_m) c_2 \sqrt{\ln \left( \frac{D_m}{D_{01}} \right) \ln \left( \frac{D_2}{D_{02}} \right)} = 0
\end{align*}
\]

These equations can be solved by the Newton-Raphson method using the following relation:

\[
\begin{bmatrix}
D_m^{(i+1)} \\
D_2^{(i+1)}
\end{bmatrix} = \begin{bmatrix}
D_m^{(i)} \\
D_2^{(i)}
\end{bmatrix} - \begin{bmatrix} J_1 \end{bmatrix}^{-1} \begin{bmatrix} F_1' \end{bmatrix}, \tag{6-27}
\]

where the Jacobian matrix \( J_1 \) has the following components:

\[
\begin{bmatrix}
\frac{\partial F_1}{\partial D_m} & \frac{\partial F_1}{\partial D_2} \\
\frac{\partial F_2}{\partial D_m} & \frac{\partial F_2}{\partial D_2}
\end{bmatrix}
\]
\[
\frac{\partial F_1}{\partial D_m} = 2 \nu^2 (1 - D_m)(1 - D_1) - 2 \ln \left( \frac{D_m}{D_0} \right) - 3 - 2(1-D_m) c_2 \ln \left( \frac{D_m}{D_0} \right) - \frac{1}{2} \ln \left( \frac{D_m}{D_0} \right)
\]

\[
\frac{\partial F_2}{\partial D_2} = 2 \nu^2 (1 - D_m)(1 - D_2) - 2 \ln \left( \frac{D_m}{D_0} \right) - 3 - 2(1-D_m) c_2 \ln \left( \frac{D_m}{D_0} \right) - \frac{1}{2} \ln \left( \frac{D_m}{D_0} \right)
\]

\[
\frac{\partial F_1}{\partial D_2} = (1 - D_m)^2 \nu^2 + D_m \nu c_2 \ln \left( \frac{D_m}{D_0} \right) - \frac{1 - D_2}{D_2 \ln \left( \frac{D_m}{D_0} \right)}
\]

\[
\frac{\partial F_2}{\partial D_m} = (1 - D_2)^2 \nu^2 + D_2 \nu c_2 \ln \left( \frac{D_m}{D_0} \right) - \frac{1 - D_m}{D_m \ln \left( \frac{D_m}{D_0} \right)}
\]

Comparing Eq. (6-26) with Eq. (6-8), it is obvious that the value \( D_m \) is only dependent on the initial damage measure \( D_0 \) for a one-dimensional case, while for a two-dimensional case, it is also related to the Poisson’s ratio of the material. Results of the numerical solution of Eq. (6-26) using Eq. (6-27) are given in Fig. 6-1. It can be seen from the figure that the maximum damage value \( D_m \) in the major principal direction is mainly dependent on its corresponding initial measure \( D_{01} \), while the effect of the initial measure \( D_{02} \) in minor direction on the value \( D_m \) could be neglected.
6.4 Presentation of Element's Failure – Initiation of Fracture

The major principal stress-strain curve described by Eq. (6-16) reaches its maximum for the damage value \( D_I = D_m \), which can be calculated from the Eq. (6-27). For brittle materials, this corresponds to a local failure event with damage instantly transiting from the value \( D_m \) to the nominal value \( D = 1 \), corresponding to the complete failure of an element.

To describe the element’s failure, a few methods are generally used in finite element analysis: 1) to cancel the element and free the boundary adjacent to neighbouring elements; 2) to assume the Young’s modulus of the element in the direction perpendicular to the crack, to become a very small number; 3) to insert a cohesive element in the element; 4) to use the average mechanical properties for all elements around the element, including that of the failed one.

In this chapter, the element’s failure is linked to generation of a crack crossing the element (Fig. 6-2). This is accounted for in the model by modification of the element’s
stiffness parameters considering almost total loss of stiffness along the direction perpendicular to the crack plane, which, in its turn, is assumed to be perpendicular to the direction of the maximum principal stress. In the Local Coordinate System (LCS) \( x' o' y' \), the modified stiffness tensor of the element with a crack is expressed in the form:

\[
\begin{bmatrix}
\sigma_1 \\
\sigma_2
\end{bmatrix} = \frac{E}{1 - \nu^2} \begin{bmatrix}
\delta_{11} & \nu \delta_{11} (1 - D_2) \\
\nu \delta_{11} (1 - D_2) & (1 - D_2)
\end{bmatrix} \begin{bmatrix}
\epsilon_1 \\
\epsilon_2
\end{bmatrix},
\]

(6-28)

where \( \delta_{11} \) (\( \delta_{11} << 1 \)) is considerably less than its original magnitude before crack initiation (1-\(D_{1m}\)).

Thus, the events of local failure, introduced into the model in terms of cracks in respective elements, could change the type of anisotropy of coating. Oriented normally to the principal strain direction, which not always coincide with the anisotropy axis, they can result in a transition from initially local isotropy or orthotropy (in the Global Coordinate System (GCS) \( x oy \)) to the induced fully anisotropic situation (Fig.6-1).

### 6.5 Framework of Multiscale Analysis in Ceramic Coatings

It is known from the review in Chapter 3 that stress level in ceramic coatings may vary significantly through their thickness (generally from 100 \( \mu m \) to 1000 \( \mu m \)). This requires that element dimension should be small enough, e.g., less than 80 \( \mu m \), to capture the stress/strain gradient in the coatings in finite element simulations. However, real microstructures of the coatings at that length scale often demonstrate significant
heterogeneity, due to random distributions of microvoids, making the periodicity assumption in general multiscale models (see Section 2.5) inadequate. This drawback is further enhanced in cases of non-uniform processes of damage and fracture in coatings under complex loading conditions discussed in Chapter 3.

Thus, in this section, a framework of two-scale (macro and meso) damage and fracture evolution in ceramic coatings is proposed in terms of the finite element mesh superposition method. The macroscopic finite element analysis is firstly conducted for a global region $\Omega^G$, and the homogenized material model is used to reflect microscopic heterogeneity. Local regions $\Omega^L$ are then analysed by a set of mesoscopic finite element analyses, the results of which are superimposed onto the macroscopic elements. Properties of each mesoscopic element are determined by its local heterogeneity such as microvoids, microcracks etc. Results of the macroscopic analysis are used as boundary conditions (BCs) for mesoscopic analyses for local areas. These BCs may be of the displacement or force type. For the former case, the applied boundary displacements are interpolated from the macroscopic mesh solution. For the latter case, the internal forces or stresses obtained from the macroscopic calculation are converted to nodal forces on the mesoscopic meshes. An illustration of the framework of the macro-meso scale analysis is shown in Fig. 6-3.
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6.6 Summary

- Constitutive equations with consideration of damage and fracture evolution in heterogeneous brittle materials are proposed.
- Failure of an element is represented on the basis of continuum damage mechanics.
- A framework of the multiscale analysis for damage and fracture in ceramic coatings is proposed in terms of the finite element mesh superposition method.
Chapter 7. Modelling Damage and Fracture Evolution in Ceramic Coatings under Mechanical Loading

Development of damage and fracture in heterogeneous ceramic coatings under three-point bending is studied in this chapter using finite element analysis, both at the macroscale and mesoscale, accounting for a statistical distribution of material’s strength. The coatings are assumed to have a brittle local behaviour and initial damage is distributed randomly through test specimens. The analysis also considers that there exists a length scale which is characteristic for growing damage, depending on the fracture processes induced. A simulation procedure to evaluate damage development through test specimens is implemented and the influence of the scatter of the fracture stress distributions is analysed. By means of these simulations and their comparison to experimental results, the energy absorption capacity $W^*$ described in Chapter 6 is determined.

7.1 Model Background

Research on evaluating the lifetime and thermomechanical behaviour of high performance materials in various components, e.g., turbine blades, which are made of superalloys with ceramic thermal barrier coatings (TBCs), has gained increasing interest in recent years. Studies of crack initiation and propagation in TBCs are an important area of research aimed at evaluation of the lifetime of superalloys with TBC in various applications. Multiple studies have been made about the failure mechanism for TBCs under thermal loading while only a few studies investigated the failure mechanism under mechanical loading from the points of view of experimental or analytical methods. However, those TBC-related components are subjected not only to thermal loadings but
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also to high mechanical stresses induced by tensile or bending loading. This requires that the coating must be able to resist cracking or spalling induced by mechanical stresses when it plays its protective role under thermal loading. Thus, numerical simulations of damage and fracture evolution in ceramic coatings under three-point bending are conducted in this chapter and compared to the results from the bending tests in Chapter 4.

7.2 Model Description

An illustration of an alumina-aluminium specimen under three-point bending is shown in Fig. 7-1. The specimen’s dimensions (see Fig. 4-3) are $L \times (H_1 + H_2) \times W = 37 \text{ mm} \times (0.25+5.25) \text{ mm} \times 3.5 \text{ mm}$. The span $S = 27 \text{ mm}$ and the thickness of the alumina coating and substrate is 0.25 mm and 5.25 mm, respectively. The specimen is modelled by quadrilateral finite elements and plane stress condition is assumed during the finite element analyses, which are implemented based on the finite element package PATRAN/NASTRAN 2001R3, Microsoft Windows 2000 and a PC (1.5 GHz).

The total number of elements is 6346 and the number of nodes is 7035. The coating is discretized into 1002 finite elements (see Fig. 7-2) of a uniform size 0.083 mm × 0.11 mm (considered as a macroscale). Each element is considered as homogeneous and has the material properties shown in Table 5-4, i.e., the Young’s modulus $E_0$ is 140 GPa and the homogenized compliance tensor of the alumina coating is

![Figure 7-1 Illustration of an alumina-aluminium specimen under three-point bending.](image-url)
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\[ C_{1.8\%} = \frac{1}{E_o} \begin{bmatrix} 1 & 0.265 & 0 \\ 0.965 & 0.965 & 0 \\ 0.723 & 0 & 1 \\ 0.83 \end{bmatrix} \]

Figure 7-2 Finite element meshing and boundary conditions of the specimen under three-point bending.

It has been known from the results of three-point bending tests (see Table 4-5) that the critical force characterising the onset of macroscopic cracking is between 458 N and 493 N for specimens with these dimensions. In this study, an average force value 470 N is considered as the level of the critical force applied for specimens in three-point bending tests.

Various definitions of damage have been used to describe the response of different materials to external loading. One of the CDM approaches to brittle materials, suggested in [27] and extended in Chapter 6 to non-uniaxial stress states, is employed to describe damage evolution in porous alumina coatings. Initiation and growth of local microcracks linked to local failures affect damage evolution due to changes in elastic anisotropy at the level of elements and subsequent modifications of stress and strain fields. A flow chart of the simulation for the global region (macroscale) of the coating under bending is shown in Fig. 7-3. The finite element code NASTRAN is expanded to include the constitutive relationship for a media with damage defined as a material subroutine.
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Geometry and mesh generation
Input of initial material properties

Modelling microstructure of a RVE

Calculating homogenized effective properties and initial damage distribution

Construct constitutive relationship of damaged material

Load/Boundary condition

Finite Element solution

Extract principal stress, strain and shear angle etc. from results

Macroscopic damage analysis

\[ \frac{|D_i' - D_i'^{-1}|}{D_i'} < 1\% \]

\( N \)

\( Y \)

Extract nodal displacements and forces at boundary between global and local regions of coating

Mesoscopic damage analysis (see Fig. 7-7)

Coordinate transformation

Modify compliance tensor in LCS

Figure 7-3 A flow chart of macroscopic damage and fracture analysis for global region of a coating under external loading.
7.3 Results of Simulations and Discussion

7.3.1 Analyses for Stress and Damage Distribution at Macroscale

A distribution of the major and minor principal stresses and of the shear angle (the direction of major principal stress) in a central region (14.8 mm × 0.25 mm) around the centre of the coating (x = 0) with porosity 1.8% under three-point bending is shown in Figs. 7-4 and 7-5. In other regions of the coating, the major principal stresses induced by the bending are relatively small so that they are neglected in the figures. The stress values in Fig. 7-4 are normalised by the maximum stress $\sigma_m = 363$ MPa, attained at the central element of the top surface of the coating. Values of the coordinate x is also normalised with the length value 14.8 mm of the central region and represented as $R_x$. 

![Graph showing stress distribution](image-url)
The results indicate that both minor stresses and the shear angle of elements around the coating's centre in Fig. 7-4 are close to zero. This means that the loading state of the central elements can be considered as uniaxial tension. When the damage value of the
central element of the top surface of the coating reaches its maximum value $D_m$, the major principal stress of the element is considered to be the critical stress $\sigma_m$. From Eqs. (6-5) to (6-7), the energy absorption capacity $W'$ can be determined as

$$W' = \frac{\sigma_m^2}{2E_0 (1-D_m)^2 \ln \left( \frac{D_m}{D_0} \right)} = 420.3 \text{ kJ/m}^3,$$

where the critical stress $\sigma_m = 363$ MPa, the Young's modulus $E_0 = 140$ GPa, the initial damage $D_0 = 1 - 0.965 = 0.035$ (see Table 5-4) and its corresponding maximum damage $D_m = 0.219$ (see Eq. (6-8)).

The iterative macroscopic damage analysis is conducted in terms of the procedure shown in Fig. 7-3. The damage distribution in the major principal direction of the alumina coating under bending before and after the iteration is shown in Fig. 7-5. It can be seen from the figure that there are differences between Fig. 7-5 (a) and (b) due to the account for damage evolution induced by external loading. Variation of the shear angle of each element leads to variation in the distribution of initial damage in the major principal direction (see Eq. (6-10)) although each angle is very small in the region of stress concentration. Consequently, the damage distribution under loading also varies in different elements thus leading to inhomogeneity in the coatings. The discontinuities in the damage distribution in Fig. 7-5(b) locate at about 0.89 mm from the coating centre. These positions could be the subsequent positions where failures occur after the first crack is initiated in the coating centre ($x = 0$) during loading. This phenomenon is very similar to the results from the three-point bending tests in Chapter 4, in which it is found that the distance between the first and the second cracks is about 1 mm.

Apparently, damage evolution induced by external loading affects elastic properties in each element and vice versa; variations of the properties lead to changes in the damage field in the coating and its inhomogeneity. These phenomena become more evident in mesoscopic analysis due to introduction of more inhomogeneous properties of the initial damage in the coating.
Figure 7-5 Damage distribution in the major principal direction in the coating under three-point bending, before (a) and after (b) the iterative damage analysis, as well as the fringes (c) of the damage field.
7.3.2 Analysis of Stress, Damage and Fracture Evolution at Mesoscale

In order to study further the effect of microstructure, which induces larger local inhomogeneity on damage and fracture evolution in ceramic coatings, mesoscopic damage analysis for a local region (2.0 mm x 0.25 mm) around the centre of the coating (see Fig. 7-1) was conducted. This region includes the positions where cracks induced by the loading may be initiated in the macroscopic analysis of last sub-section 7.3.1. It was discretized into 720 elements and 730 nodes and each of them has uniform size 27.7 µm x 27.7 µm (considered as mesoscale). Properties of each element are determined by the microstructural simulation, discussed in Section 5.2.4. At the boundary between this region and the global region, the nodal forces and displacement restriction, interpolated from the results of the macroscopic solution described in the last sub-section, are applied (see Fig. 7-6). A flow chart of the mesoscopic analysis is shown in Fig. 7-7.
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Figure 7-6 Nodal forces and displacements applied to the boundary between the local and global region. (*Rx* and *Ry* are normalized coordinates with the length value 2 mm and 0.25 mm, respectively).
Chapter 7. Modelling Damage and Fracture Evolution in Ceramic Coatings under Mechanical Loading

Mesh generation and microstructure modelling of the local region

Determine properties of each mesoscopic element in the local region of the coating

Construct constitutive relationship of the damaged material

Load/Boundary condition (see Fig. 7-3)

Finite Element solution

Coordinate transformation

Modify compliance tensor in LCS

Extract principal stress, strain and shear angle etc. from results

Damage analysis including modelling crack initiation and evolution for each element $i$

$\frac{|D_i^j - D_i^{j-1}|}{D_i^j} < 1\%$

Figure 7-7 A flow chart of mesoscopic damage and fracture analysis for a local region in the coating under three-point bending.
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The initial damage distribution corresponding to the effective material properties linked to microstructures and element dimensions of the local region was determined for all elements based on the computational results from Chapter 5. Figure 7-8 shows such an initial random damage distribution in the major principal direction of the coating and its major principal stress distribution at the region just before the coating fails. Due to inhomogeneity of the local region, the stress distribution is non-symmetric and closely related to the local states, although the boundary and load conditions from the macroscopic analysis is symmetric.

Figure 7-8 Distributions of (a) initial damage and (b) stresses (MPa) in the major principal direction at the local region in the coating (just before failure of the coating, porosity 1.8%) under three-point bending.

Once an element in the coating fails, it instantly leads to damage evolution and further failure of the "weaker" elements around it. Figure 7-9 shows such a damage and fracture process in the coating under three-point bending. In order to better visualise the fracture process, only failed elements are shown in the figures. The failure starts from an
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element (Fig. 7-9(a)), which is at a middle place of the local region and the damage state $D_1$ of the element is larger than its damage limit value $D_{ml}$ (see Eqs. (6-9) to (6-23)), and propagates into the coating due to coalescence of other locally failed elements at each iterative step (Fig. 7-9(b) to (g)) until it reaches the coating/substrate interface (Fig. 7-9(g)). It can be seen from the figures that cracks in the coating are initiated in one or several places (Figs. 7-9(b) to (d)), at which the stresses concentrate and the elements usually have larger initial damage (Fig. 7-8). These cracks may diverge or coalesce (see Fig. 7-9(g)) during their propagation and they are not formed symmetrically on the both sides of the central axis, due to inhomogeneity of the local region shown in Fig. 7-8(a).
Moreover, crack paths are irregular and depend on the local damage states of the elements in front of the crack tips, where stresses concentrate. This can be seen from the stresses distribution of the region shown in Fig. 7-10(a) corresponding to the damage states in Fig. 7-9(a). This means that the results from the theory of damage mechanics corresponds to that of fracture mechanics. Furthermore, the damage evolution and stresses distribution resulting from the proposed damage theory indicate that cracks induced by the loading initiate at and propagate to the places where: 1) the element has a larger initial damage value; 2) stresses concentrate; 3) the damage level of the element is larger than its limit value $D_{ml}$. In other words, failures do not necessarily occur in places with the highest stress concentration. Thus, failure of each element, on the one hand, induces further failure of other elements. On the other hand, it also releases energy so that the stresses in the coating decrease and gradually reach a stable state Fig. 7-10(b).
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Figure 7-10 (b)
Figure 7-10 Distributions of major principal stresses (MPa) in the local region of the coating when the first crack initiates (a) and stresses reach stability (b).

Figure 7-11 shows another failure process in the alumina coating under the same loading conditions and different random seed for its initial random damage distribution (i.e., another statistical realisation, see Fig. 7-12). For this case, the first crack is also initiated from the element at the middle of the top surface of the coating, but here it directly propagates to the interface (Figs. 7-11 (a) and (b)) where it turns and starts propagating along the interface (Figs. 7-11 (c) and (d)). This crack is accompanied by another crack, which is initiated in an initially "weaker" element but it has an inclined path (Figs. 7-11 (c) and (d)). Similar crack trajectories have been observed in the three-point bending tests (compare Figs. 7-9 and 7-11 with Fig. 4-25).

These results indicate that damage and fracture evolution in ceramic coatings under loading is significantly affected by the local material properties due to the random distribution of microvoids in coatings. However, results from the simulations, using not only the above two random seeds but also many other seeds, also indicate that there are always two main macrocracks induced by this loading: one macrocrack occurs at the middle of the coating and another one is initiated in a "weaker" position at the top surface of the coating about 0.5 mm to 0.9 mm from the middle crack. This result agrees with the test results (see Fig. 4-24) and can be explained by the macroscopic damage distribution in Fig. 7-5(b), which shows that there are local concentrations of damage due to anisotropy and heterogeneity of the material properties of coatings.
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Figure 7-11 Another statistical realisation of damage and fracture processes in an alumina coating (random seed 8929) under three-point bending.

Figure 7-12 Initial damage distribution (random seed 8929) in an alumina coating.
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7.4 Summary

- A multi-scale method is used to analyse the three-point bending problem for a coated specimen at room temperature. Macroscopic and microscopic stresses in the coating under loading are evaluated quantitatively. The stress gradient in the coating under three-point bending leads to a development of damage which is relatively localised and stable, and fracture stresses measured in the bending tests correspond to a significant progression of the damage.

- Failure mechanisms in coatings under three-point bending are investigated qualitatively and quantitatively with the account for their microstructure. Especially, a crack propagation path is described vividly in terms of the damage theory proposed in Chapter 6. The orientation and shape of this path is determined by the local properties of the coatings. Thus, an explicit account for arbitrary microstructural morphologies and damage/fracture patterns is provided. The results indicate that the proposed method can explain the crack patterns and predict the average distance between the neighbouring cracks observed in the three-point bending tests.

- The studies in this chapter are restricted to a two-dimension analysis, which does not fully correspond to the actual damage processes induced. However, the results obtained show clearly the capacity of this analysis to describe the damage development in heterogeneous materials. Extension of these studies into three dimensions may lead to more diffuse damage and fracture evolution.
Chapter 8. Thermally Induced Damage and Fracture Evolution in Ceramic Coatings

In this chapter, damage and fracture evolution in alumina coatings under transient thermal loading is examined in terms of a finite element model. Random microstructures within each element of discretized ceramic coatings are accounted for in terms of statistical distributions of microvoids in the element. Processes of failure evolution at the microscopic level are introduced into mesoscopic considerations using the framework of continuum damage mechanics (CDM) and a damage parameter as a measure of the materials deterioration. The main feature of this study is direct investigations of the effect of microstructure on damage, as well as analyses of fracture initiation and evolution in alumina coatings by means of numerical simulations. The study is focused on exploring microstructural effects on the thermal shock behaviour of coatings. In the simulations, specimens with various levels and parameters of porosity of alumina coatings are subjected to the high-heat flux to study the effect of microstructure on their response.

8.1 Model Background

A considerable mismatch of coefficients of thermal expansion for ceramic coatings and metallic substrates results in generation of stresses in such systems, even under purely thermal loading in the absence of mechanical loads. Transient thermal stresses induced by thermal shock associated, for instance, with fuel ignition in an engine or a turbine are superimposed onto the mismatch stresses. The combined stresses can cause initiation and propagation of cracks and eventually lead to local failures in coatings. The damage and fracture development due to thermal stresses has been one of the principal problems encountered with ceramic thermal barrier coatings (TBCs), which play an increasing role in various industrial applications.

Various models have been developed to describe the final spallation failure in ceramic coatings, while the damage evolution process leading to such spallation at some
critical stage has been insufficiently understood. This can be naturally explained by the complex microstructural morphology of ceramic coatings, which greatly influences the initiation and evolution of damage and fracture in them. However, appropriate experimental schemes for observation and measurement of microstructural damage and fracture evolution are still to be developed. Thus, modelling tools using various general approaches combined with the experimental results are the most suitable methods to account for the effect of random microstructures of real materials.

It has been reviewed in Chapter 3 that coatings in numerical simulations are often either directly discretized into elements, neglecting the effect of microstructure, or they are simply considered as a single layer of elements with a uniform stress field, equally biaxial in the plane of the coating/substrate. However, in the case of incorporation of effective properties, obtained for brittle materials by various models, RVE dimensions in the simulations must be much larger than a microstructural length scale. Moreover, the element size in finite element macroscopic simulations should be at least as large as the RVE dimensions in order to obtain representative results. Thus, many microstructural details have to be smeared out although they could have a significant effect on damage and fracture evolution in coatings. These drawbacks are further enhanced in studies of non-uniform processes of damage and fracture in coatings under complex loading conditions. Thus, more adequate models should be suggested.

8.2 Model Description

Numerical simulations of damage and fracture evolution in alumina coatings under transient thermal loading are carried out using finite element method based on MSC-PATRAN/NASTRAN. The damage model and the scheme to introduce microstructure into the simulations, which are discussed in Chapter 6, are implemented as user subroutines. The specimen used in the simulations is an alumina coating bonded to a metal substrate. An illustration of such a specimen under thermal shock is shown in Fig. 8-1. The specimen’s length is \( L = 30 \text{ mm} \), thickness of the alumina coating \( H_1 = 0.25 \text{ mm} \), thickness of the substrate \( H_2 = 5.25 \text{ mm} \), the specimen’s width \( W = 3 \text{ mm} \), and the support span \( S = 22 \text{ mm} \). The bond between the substrate and coating is considered to be of negligible thickness.
8.2.1 Governing Equations

Due to the complexity of the transient thermal loading and the crack morphology, the thermal shock problem is solved as a quasi-steady-state uncoupled thermoelastic (plane stress) problem. The governing equations for this problem are well-known and can be described as

\[
\begin{align*}
\{\varepsilon_{i1}\} & = \{\varepsilon_{i1} + \alpha \Delta T\} \\
\{\varepsilon_{i2}\} & = \{\varepsilon_{i2} + \alpha \Delta T\}, \tag{8-1}
\end{align*}
\]

where \(\varepsilon_i\) and \(\varepsilon'_i\) \((i=1, 2)\) are total strains and the mismatch strains induced by the thermal stresses; \(\alpha\) is the coefficient of thermal expansion. Damage and fracture evolution of the alumina coating under loading is presented by Eq. (6-10) using strains \(\varepsilon'_i\). In Eq. (8-1), \(\Delta T\) is the temperature difference between the current and initial temperature levels; the temperature field is determined by solution of the following transient heat transfer equations:

\[
\frac{\partial}{\partial x} \left( k_x \frac{\partial T}{\partial x} \right) + \frac{\partial}{\partial y} \left( k_y \frac{\partial T}{\partial y} \right) = \left( \rho c_p \right) \frac{\partial T}{\partial t}, \quad (i=x, y), \tag{8-2}
\]

where \(T\) is temperature, \(t\) is time, \(\rho\) is density, \(c_p\) is the specific heat, \(x\) and \(y\) are the coordinates. The thermal conductivities \(k_i\) of the coating with porosity 1.8% is \(k_1 = 0.974k_0\).
and $k_2=0.843k_0$ (see Table 5-4), $k_0$ being the thermal conductivities of undamaged materials. Solution of the above equations is completed using MSC-NASTRAN.

To avoid inaccurate results or unstable solutions, the proper choice of an initial time increment is required. An adequate magnitude of the initial time step depends on a number of factors, including the spatial size of the element mesh and thermal diffusivity of the material. The selection criterion is suggested by NASTRAN as:

$$\Delta t_0 \approx \frac{1}{10} \cdot (\Delta x)^2 \cdot \frac{\rho \cdot c_p}{k_0},$$

where, $\Delta t_0$ is the initial time step, $\Delta x$ is the mesh size. In this chapter, the two values are $0.083 \text{ mm}$ and $2.98 \times 10^{-4} \text{ s}$, respectively, and thus 12,000 time steps are necessary for the computation running until the maximum temperature in the substrate is close to its melting point $550^\circ\text{C}$.

In the simulations, the coating is modeled as a two-dimensional system consisting of two layers made of ceramic and the metal substrate as shown in Fig. 8-1. The visco-plasticity caused by the material sintering and creep at high temperature is neglected due to high temperature stability of the coating. Quadratic four-node elements are created in the simulations. The substrate (aluminium) is discretized into 4607 nodes and 4320 elements. The coating is discretized into 1084 nodes and 810 finite elements, which are described as three layers: top, middle and interface layers (see Fig. 8-2), with a uniform size $0.083 \text{ mm} \times 0.11 \text{ mm}$ (considered as macroscale). Each element in the coating is considered as homogeneous, and its initial material properties are based on the data in Table 5-4, i.e., the homogenized compliance tensor of the alumina coating (porosity 1.8%) is

$$C_{1.8\%}^H = \frac{1}{E_0} \begin{bmatrix} 1 & 0.265 & 0 \\ 0.965 & 0.965 & 0 \\ 0 & 0.727 & 0 \\ 0.83 & 0 & 1 \end{bmatrix},$$

and the initial damage values in $x$ and $y$ directions are $D_{0x} = 0.035$ and $D_{0y} = 0.277$. The large difference between the two components of the initial damage is linked to the specific character of the manufacturing process of ceramic coating. This character affects the coating’s microstructures (see Chapter 5) and determines anisotropy in the coating’s behaviour and their high sensitivity to the load orientation. During the computation, the
properties of each element depend on its local damage state that is discussed in the following sub-section.

![Finite element discretization of the alumina coating and substrate.](image)

8.2.2 Material Properties

An alumina-aluminium specimen — the aluminium alloy substrate bonded with an alumina coating — is used in the simulations. Aluminium alloy is considered as isotropic and have properties shown in Table 8-1.

Table 8-1: Material properties of the substrate [183].

<table>
<thead>
<tr>
<th></th>
<th>Aluminium alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s modulus (GPa)</td>
<td>71</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.35</td>
</tr>
<tr>
<td>Coefficient of linear thermal expansion (\times 10^{-6}/\degree{C})</td>
<td>24</td>
</tr>
<tr>
<td>Thermal conductivity (W/m/K)</td>
<td>190</td>
</tr>
<tr>
<td>Specific heat (J/kg/K)</td>
<td>880</td>
</tr>
<tr>
<td>Density of solid (kg/m³)</td>
<td>2700</td>
</tr>
<tr>
<td>Melting point (ºC)</td>
<td>550–660</td>
</tr>
</tbody>
</table>
Chapter 8. Thermally Induced Damage and Fracture Evolution in Ceramic Coatings

It has been known in Chapter 7 that the energy absorption capacity and the Young's modulus of the alumina coating are 420 kJ/m³ and 140 GPa, respectively, at room temperature (20°C). It is assumed that they are proportional to that of bulk alumina (see Table 3-2) at the same temperature. Thus, nominal mechanical properties of alumina coating used in this Chapter are determined and shown in Table 8-2. The Poisson's ratio of alumina is taken as 0.27.

Table 8-2 Mechanical properties of alumina coating.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Young's modulus (GPa)</th>
<th>Energy absorption capacity (kJ/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>140.0</td>
<td>420</td>
</tr>
<tr>
<td>400</td>
<td>137.2</td>
<td>378.5</td>
</tr>
<tr>
<td>750</td>
<td>132.5</td>
<td>353.1</td>
</tr>
<tr>
<td>1000</td>
<td>128.0</td>
<td>336.3</td>
</tr>
<tr>
<td>1500</td>
<td>115.9</td>
<td>310.0</td>
</tr>
</tbody>
</table>

Thermal properties of the alumina coating used in the simulations are shown in Table 8-3. Thermal conductivity in the table is determined from comparison of numerical results with experimental ones from Fig. 4-43. Other material properties are taken from literature [141]. At this stage the effect of microstructure on these properties is neglected, though this may affect accuracy of the results.

Table 8-3 Thermal properties of alumina.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Coefficient of linear thermal expansion (x10^6/\text{K})</th>
<th>Thermal conductivity (W/m/K)</th>
<th>Specific Heat (J/kg/K)</th>
<th>Density of solid (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>4.1</td>
<td>6.6</td>
<td>755.</td>
<td>3984</td>
</tr>
<tr>
<td>500</td>
<td>7.1</td>
<td>11.4</td>
<td>1165.</td>
<td>3943</td>
</tr>
<tr>
<td>1000</td>
<td>8.1</td>
<td>10.2</td>
<td>1255.</td>
<td>3891</td>
</tr>
<tr>
<td>1200</td>
<td>8.3</td>
<td>9.6</td>
<td>1285.</td>
<td>3868</td>
</tr>
<tr>
<td>1400</td>
<td>8.5</td>
<td>9.1</td>
<td>1315.</td>
<td>3845</td>
</tr>
<tr>
<td>1500</td>
<td>8.6</td>
<td>9.0</td>
<td>1330.</td>
<td>3834</td>
</tr>
</tbody>
</table>
8.2.3 Load and Boundary Conditions

Displacement constraints are added at the central node of the specimen’s bottom both in x and y directions (see Fig. 8-1), and at the symmetry axis (y axis) in x direction. Convective (to ambient) thermal boundary conditions are assumed at the surface, two edges and bottom of the specimen. The coefficient of convection used in the simulations is $10^{-5}$ W/mm$^2$/K. Radiation of each specimen to space is neglected. A laser-induced high heat flux is applied to the top surface of the coating, with the peak at the centre of the surface, to reproduce the laser-induced thermal shock tests of Chapter 4. The heat flux $q(x)$ can be described as a Gaussian distribution by the following equation

$$q(x) = q_{\text{max}} \exp \left( -2 \left( \frac{x}{r} \right)^2 \right),$$

(8-5)

where $x$ is the distance along the coating surface from the centre, $r$ is the spot radius containing 86.6% of the laser power (0.5 kW in this Chapter), and $q_{\text{max}}$ is the maximum heat flux. It has been known from the thermal shock tests (Section 4.3) that the diameter of the laser spot area is 12 mm, i.e., the spot radius $r = 6$ mm. It is defined that the laser power

$$P_L = 2\pi \int_0^r q(x)dx.$$  

(8-6)

Then, the maximum heat flux $q_{\text{max}}$ can be calculated as

$$q_{\text{max}} = \frac{P_L}{2\pi \int_0^r \exp \left( -2 \left( \frac{x}{r} \right)^2 \right)dx} \approx \frac{P_L}{1.358r^2}.$$  

(8-7)

8.2.4 Flow Chart of the Simulations

A general flow chart of the simulations is shown in Fig. 8-3. It starts with the data input for the initial state. Material parameters, specimen dimensions and initial effective properties of materials are among the input data resulting from the analyses, based on the account for microstructure of the coating and described in Chapters 5 and 7. Next, a two-dimensional transient heat transfer analysis is performed, resulting in transient temperature distributions, which is used to determine corresponding deformations and thermal stresses everywhere in the model for each time step. Then, structural analysis for plane-stress conditions is performed to determine the damage and fracture evolution in
the coating. In the analysis at each time step, an iterative computation runs until the convergence conditions \( \left| \frac{D_i^{j+1} - D_i^j}{D_i^j} \right| \leq \varepsilon \) are met for all elements, where \( D_i^j \) is the damage value of the \( j \)-th element at the \( i \)-th iterative step, \( k \) is a time step and \( \varepsilon \) is a small positive value (\( \varepsilon = 10^{-2} \) in these calculations).

Figure 8-3 A flow chart of the thermal shock simulation.
8.3 Results and Discussion

8.3.1 Temperature Distribution and Evolution

Numerical simulations are performed for the maximum temperature at the coating surface (alumina-aluminium, porosity of alumina 1.8%) rising from initial room temperature 20°C to 700°C, in the central node (x = 0) of the coating top surface (see Fig. 8-4). During the heating time, the temperature difference between the central node of the top surface and that of the interface is from 150°C to 190°C. This difference is larger at the initial heating time. Figures 8-5 and 8-6 demonstrate the temperature distribution (at 3.6 s, Fig. 8-5) in the coating and the temperature evolution (Fig. 8-6) at the top surface of the coating along x for different time steps. Due to the symmetry of the boundary conditions and the geometry about the centre of the specimen, only the right half of the specimen is analysed in this section. Values of the coordinate x is normalised with the value 30 mm of full specimen length and denoted as Rx in Figures.

![Figure 8-4 Temperature evolution at the central nodes (x = 0) of the top surface of the coating and the interface between the coating and the substrate.](image)
Figure 8-5 Temperature distributions in the coating $t = 3.6$ s.

Figure 8-6 Temperature evolution at top surface of the alumina coating during transient thermal loading.
8.3.2 Stress-Strain Analysis of Alumina Coating under Thermal Loading

Using these temperature fields of the alumina coating, as well as that of the substrate, as thermal laoding for each time step, a quasi-static structural analysis is performed. Figure 8-7 shows distributions of the major principal stress (at 3.6 s, Fig. 8-7) in the coating during the thermal loading. At this time, the maximum temperature in the coating is 692°C, while in the substrate, it is 546°C, which is close to the lower limit (550°C) of the melting point of the substrate. It can be seen from Fig. 8-7 that compressive stresses exist at the most part of the coating due to material properties (e.g., coefficient of thermal expansion etc.) and temperature mismatch between the elements or between the coating and the substrate. However, at the interface layer of the coating connected to the substrate, tensile stresses develop and reach their maximum value at the edge ($Rx = 0.5$). Figure 8-8 shows shear angles of the stresses at the interface elements and demonstrates that the major principal stress has a direction perpendicular to the interface for most interface elements. In other words, delamination could be the major failure mode of the coating under thermal loading.

![Figure 8-7 Major principal stresses distribution in the alumina coating under transient thermal loading at time 3.6 s.](image-url)
Chapter 8. Thermally Induced Damage and Fracture Evolution in Ceramic Coatings

Figure 8-8 Shear angle of the major principal stresses at the elements of alumina coating.

Figure 8-9 shows evolution of the major stresses in the interface layer during the transient thermal loading. It has been known that the thermal stresses are induced by the mismatch in material properties and temperature difference (and gradients) for elements in the coating and substrate. As seen from Fig. 8-9, with the increase in the heating time, these stresses increase and reach maximum values at some time (1.4 s in this case), then they decrease. This process is affected by the following factors. Firstly, mismatch of coefficients of thermal expansion between the coating and the substrate leads to tensile stresses in the coating at a given temperature $\Delta T$. Secondly, higher temperature in the coating than that in the substrate induces compressive stresses in the coating, especially at the initial stage of heating. Next, the generated stresses/strains induce damage evolution that, in its turn, results in deterioration of the elastic moduli of the coating, while the damage evolution inversely can reduce the stresses. This combination of the factors leads to the phenomenon that the stresses in the coating have a limited value during the heating process.
8.3.3 Damage Distribution and Evolution of Alumina Coating under Transient Thermal Loading

It has been shown in the Section 8.2.1 that specific microstructure of the plasma sprayed ceramic coating leads to its anisotropy (see Eq. (8-4)) so that local magnitudes and orientations of the major principal stresses (see Figs. 8-7 and 8-8) are rather complex in the coating under the thermal shock. Accordingly, there exist large differences between the initial local levels of damage components (Fig. 8-10(b)) for the elements, especially between those in the middle layer and in other layers, due to the variation in these orientations of stresses. On the basis of these initial damage levels, the damage state at each element in the coating evolves under the thermal loading and reaches its specific state (see Figs. 8-10(a) and 8-11). It can be seen from these figures that the maximum value of the damage is in the area, situated at some distance from the edge: $Rx = 0.4-0.45$ (Figs. 8-10(a) and 8-11), while the corresponding thermal tensile stress has the maximum values at the interface element in the edge of the specimen ($Rx = 0.5$) at each time step (see Fig. 8-9). This means that failure, generally delamination, may easily
initiate at that place. This phenomenon has been observed in the laser-induced thermal shock tests (see Figs. 4-40 and 4-45) in Chapter 4 (though the coating there has different properties from those in this simulation). As was discussed above about the stresses evolution, the damage value reaches a limit at the time 1.4 s and then decreases. No failure occurs in elements under the studied loading conditions for material with low porosity <2%. This result also agrees with the experimental observations in Chapter 4.

![Graph](a)
Figure 8-10 Distributions of local principal damage level (a) in the alumina coating under transient thermal loading at time 3.6 s and the corresponding initial damage distribution (b) in major principal direction.

Figure 8-11 Damage evolution at interface elements of the alumina coating under transient thermal loading.
8.3.4 Effect of Porosity and Failure of a Coating

The effects of porosity on the loading behaviour of the alumina coating under the laser-induced thermal shock were investigated by numerical simulations. It can be seen from Fig. 8-12 that the temperature difference between the central nodes at the interface and top surface of alumina coating steadily increases with the increase of the porosity. Obviously, a coating with larger porosity has larger ability to protect the substrate from high-temperature loading. However, the increase of the porosity of a coating leads to larger damage levels in the coating so that failure can start in it. Figure 8-13 indicates that for the alumina coating studied in this chapter, a crack occurs at the weakest place ($R_x = 0.37-0.48$) described in the last sub-section when the porosity rises from 1.8% to 4%. A similar microcrack in the thermal shock tests can be seen in Fig. 4-45. As it has been described in Section 7.3.2, the microcrack initiates at an element that has the weakest local properties in this area. Then, it propagates along the interface and at last leads to delamination or spallation of the coating.

Figure 8-12 Effect of porosity (1.8% to 6%) on temperature difference between the central nodes at interface and top surface of alumina coating.
8.4 Summary

- Damage distribution and fracture evolution in alumina coatings under the laser-induced thermal shock are investigated by numerical simulations. The numerical results are compared to the experimental results in Chapter 4 quantitatively and qualitatively.
- During the thermal loading, compressive stresses exist in most parts of the coating. Tensile stresses generally concentrate in the interface elements of the coating near to the edge of the specimen. The direction of the major principal stresses of most elements is perpendicular to the interface.
- Failures of the coating induced by the thermal loading generally occur at the interface layer of the coating, near to but not directly at the edge of the specimen.
- Effects of porosity of coatings on their behaviour under thermal loading are examined. Higher porosity can increase temperature difference between the coating top surface and the substrate, i.e., improve its thermal protection properties. But such coatings are more prone to damage accumulation that can eventually lead to initiation of macroscopic fracture in form of delamination/spallation.
Chapter 9 Conclusions and Further Work

This chapter concludes the research carried out in this thesis by summarising the major contributions made in the study of microstructural effects on damage and fracture evolution in plasma sprayed ceramic coatings, and suggesting some directions for future research.

9.1 Concluding Remarks

Damage and fracture evolution of thermally plasma sprayed alumina coatings and the effect of microstructure on their properties have been investigated in this thesis by means of literature survey, experiments and numerical simulations. Various experiments have been carried out in order to determine some material properties, to study damage and fracture phenomena and to characterise microstructures of coatings. The experiments involved optical image analysis, nano-indentation, three-point bending, the laser-induced thermal shock and infrared thermography. The experimental results have been used to validate the developed two-dimensional finite element models for damage and fracture analysis of thermally plasma sprayed alumina coatings. The numerical models allow for predictions of the stress-strain and damage distributions as well as of the possible positions where failures may occur in the coating under mechanical or thermal loading. Most importantly, the proposed models adequately reflect experimental results. Following the list of objectives set in Introduction of Chapter 1, the PhD research has brought several specific conclusions described below.

- Literature reviews indicate that, up to date, there is no general agreement regarding the definition of damage variables due to the complex nature of damage in real materials. But such a definition is necessary in CDM so that it can properly reflect the effect of presence and evolution of a large number of randomly distributed microcracks of irregular shapes in porous brittle materials.
• Approaches of FM have acquired success in many engineering areas. They can be used in cases with explicitly given geometry and location of one or more macrocracks. However, in ceramic materials, there exist a number of natural flaws such as inclusions, pores, damaged grain boundaries etc. It is too difficult mathematically to apply the FM approaches to analyse effect of these natural flaws, especially, in cases with failures in structures that have no visible initial cracks. This necessitates studies of the evolution of internal microscopic damage before the formation of the visible macroscopic defect. Thus, the growth of the macrocrack through a solid with a heterogeneous microstructure may be studied using FM approaches, while continuum damage accumulation prior to the macrocrack initiation may be determined using CDM approaches. The relation between the two approaches is actually a question of different characteristic sizes of microcracks and macrocracks.

• Results from either literature survey or the experiments in this thesis indicate that there exist large differences in properties of thermally plasma sprayed ceramic coating and the comparable bulk ceramics.

• Both thickness and porosity of coatings have significant effects on loading behaviour under thermal loading. Thermal resistance of the coatings increases with the increase in their thickness or the porosity level. However, thicker coatings easily spall during heating.

• Microvoids affect properties of the plasma sprayed coatings both in the spray and transverse directions, with the effect on the values in the spray direction being more pronounced. With the increase in porosity, the elastic properties apparently reduce. For the case of 1.8% porosity, the Young’s moduli of the plasma sprayed alumina coatings in the transverse and spray direction reduces by about 3.5% and 17.3%, respectively, compared to their nominal values. With a decrease in dimensions of the RVE, the scatter of the effective properties increases, especially in the spray direction. For the case of 1.8% porosity, the scatter rises from 3.1% to 16.7% with the dimension decreasing from 250 μm to 50 μm.

• Both the experimental observation and the numerical simulation in this thesis indicate that cracks induced by three-point bending in the specimens with alumina coating are not formed symmetrically on the both sides of the central axis, due to local variations in microstructure and, consequently, material properties. However, there are generally two main macrocracks induced by this loading: one macrocrack occurs at
the middle of the coating and another one is initiated in a “weaker” position at the top surface of the coating about 0.5 mm to 0.9 mm from the middle crack. The crack paths are irregular and may deflect, depending on the local microstructure. This suggests that good design of microstructure of the coatings may improve their crack resistance.

- During the thermal loading of the coating, compressive stresses exist in its most part. Tensile stresses generally concentrate on the interface elements of the coating near to the side of the specimen. The direction of the stresses of most elements is perpendicular to the interface. Failures in the coating induced due to thermal loading generally occur at the interface layer of the coating, close to (but not exactly at) the specimen’s edge, although stresses usually concentrate there. Higher porosity can increase temperature difference between the coating top surface and the substrate. But durability of the coating decline with the increase in the porosity level.

- A good agreement between the experimental and numerical results obtained in this project indicates that the extended damage evolution theory can explain the crack patterns, predict the average distance between the neighbouring cracks and thus provide explanation of failure mechanism for the coatings under loading.

### 9.2 Recommendation for Future Research

Damage and fracture evolution in porous brittle materials, especially in ceramic coatings, has been an issue due to the material’s complex microstructure, which can not be neglected in analysis for coatings. The work carried out and reported in this thesis represents an attempt to develop the much-needed techniques to support the analysis of damage/fracture processes in ceramic coatings. While some initial progress has been made by this research, much still needs to be done. The following are some possible future work:

- In this thesis, the damage and fracture analyses for the coatings are implemented using a two-dimensional variant of the damage theory and a 2D finite element formulation. In order to account for practical loading states of materials, it would be of interest to extend the current algorithm to three-dimensional ones. For this purpose, different/upgraded software and hardware with a higher computational capacity is necessary.
• In this thesis, only microstructures of plasma sprayed ceramic coatings are modelled. It is necessary to examine applicability of the proposed methods in modelling other type of coating, e.g., PVD coatings etc.

• The shape of microvoids in coatings is assumed to be elliptical in this thesis. In further studies, some other shapes such as those shown in Fig. 3-1 can be introduced.

• It is assumed in this thesis that only brittle damage exists in ceramic specimens during loading. However, other types of damage such as creep damage could be also initiated in the materials, especially in the case of their subcritical states. Thus, introduction of creep damage as well as its combination with brittle damage and other types of damage would be also of interest.

• When an element fails, its damage variable $D$ is assumed to be a number close to 1 in this thesis. To determine the value of residual stiffness of locally failed areas accurately, introduction of cohesive zone models into the current damage theory may help.

• Thermal softening of both the coating and the substrate at higher temperature loading can be included into the constitutive equations, which would probably influence deformation mechanisms and improve the overall accuracy of the FE model.
References


References


References


References


