FRACTURE OF FIBRE-REINFORCED CERAMIC MATRIX COMPOSITES UNDER CONDITIONS OF THERMAL SHOCK

A THESIS SUBMITTED TO THE UNIVERSITY OF SURREY FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

By
Christos Theodorou Kastritseas

May 2005
ABSTRACT

The behaviour of fibre-reinforced ceramic matrix composites (CMCs) under conditions of thermal shock was investigated in this study. A series of water-quench tests were carried out on samples of cross-ply and plain-weave (PW) woven Nicalon/CAS (calcium aluminosilicate) composites. Damage modes due to thermal shock were characterised by examining polished surfaces of the quenched materials using optical and scanning electron microscopy. In addition, the effect of thermal shock on the mechanical properties of the woven composite was assessed under tensile and flexural loading. Predictions of the critical temperature change (ΔTc) for the onset of thermal shock fracture in both unidirectional (UD) and 2-D CMCs (cross-ply and woven) were made.

In terms of the critical temperature change, the thermal shock resistance of surfaces with (0°/90°)3s, (90°/0°)3s and woven configurations was found to be comparable (ΔTc≈400±50°C) with that of UD Nicalon/CAS of similar thickness - the effect of ply architecture was minor. By contrast, surfaces with configurations of much smaller thickness (i.e. (0°/90°)s, (90°/0°)s) exhibited significantly higher thermal shock resistance (by >100°C) and much smaller crack densities with increasing severity of shock. Damage, unless temperature-induced microstructural changes occurred, was in the form of matrix cracking that left the fibres unaffected, and originated in the central plies of each CMC. Although on application of more severe shocks cracking extended to the outer plies, crack density always exhibited a gradient across the material thickness and was higher towards the central region. All phenomena could be understood in terms of the interaction of temperature gradients of adjacent material surfaces.

The orientation and the extent of matrix cracking depended on the type of ply (i.e. longitudinal, transverse or, in the woven CMC, matrix-rich layer-'ply'). Longitudinal and matrix plies contained matrix cracks perpendicular to the horizontal (length) direction and exhibited 'composite' behaviour by virtue of a surface stress transfer mechanism - on application of more severe shocks, cracks increased in number but remained surface features. Matrix cracks in transverse plies ran parallel to the horizontal, and, although always small in number, increased significantly in length and depth at higher temperature differentials. Thus, transverse plies exhibited behaviour similar to monolithic ceramics and particulate CMCs.

Thermal shock caused a small and gradual reduction in the mechanical properties of PW woven Nicalon/CAS, the onset of which occurred at higher temperature differentials than the onset of thermal shock damage. It was shown that the reduction in properties was associated with the propagation under load of the shock-induced matrix cracks in transverse plies. Thus, transverse plies were determined to be the weaker elements of the 2-D materials as they contained cracks that extended significantly in length and depth and affected mechanical properties. The extent of such cracks, as well as of other types of thermal shock damage, was found to be smaller for the woven CMC, perhaps due to the undulating nature of its microstructure.

The onset of thermal shock fracture in UD and 2-D Nicalon/CAS CMCs was analysed by considering the anisotropic nature of the applied stress field as well as the presence of residual thermal stresses. A strength-based criterion, combined with a model for the effect of the biaxial nature of shock-induced stresses on the effective value of the interfacial shear stress, enabled satisfactory predictions to be made of the thermal shock resistance of the surface of UD Nicalon/CAS that contained longitudinal fibres, as well as of the central longitudinal plies of faces with (90°/0°)3s, (90°/90°)3s, and woven configurations. The heat transfer conditions during fracture were also determined. A fracture mechanics-based criterion combined with a modified analytical result from the literature was used to treat the situation of the transverse (end) face of UD Nicalon/CAS, as well as for the central transverse plies of faces with (0°/90°)3s, (0°/90°)3s, and woven configurations. The success of this approach depended on accurate knowledge of two material parameters, i.e. the relevant fracture toughness and the critical dimension beyond which thermal shock resistance becomes independent of material dimensions. Based on sufficient knowledge of the critical dimension, a method was also devised that allowed the effect of material dimensions to be incorporated into predictions of the thermal shock resistance.
To my father,
Acknowledgements

The work described in this thesis was performed in the School of Engineering of the University of Surrey between October 2001 and September 2004 with financial support from the Engineering & Physical Sciences Research Council of the UK and from the 'Alexander S. Onassis' Public Benefit Foundation.

The guidance, help, and patience of my supervisors, Professor Paul Smith and Dr. Julie Yeomans, are gratefully acknowledged. I would like to thank them particularly for allowing me the freedom to pursue my ideas, and for their support and understanding through some very difficult times.

In addition, thanks should go to Mr. Mike Parker, Mr. Peter Haynes, Dr. Hall Belmonte, Dr. Brian Le Page, Professor Panos Tsakiropoulos and Mrs. Shirley Hankers for providing help at various stages of this project.

Sharing the office and the laboratory (as well as a swimming pool in Rhodes...) with Mr. Nick Ludford has been a pleasure. His help with specimen preparation and microscopy are particularly appreciated.

I must also extend my thanks to my other office-mates, present and past: Miss Wantanee Buggakupta, Mr. Shreeram Thozhur, Mr. Alex Katsanos, as well as Mr. John Brown, Dr. Aidan Cole-Baker and Dr. Ben Crunkhorn.

This work, together with the whole of my undergraduate and graduate studies, would not have been possible without the continuous personal and, at times, financial support of my parents to whom I am grateful.

Last, but not least, I would like to thank Irene for accompanying me throughout this experience. She has been always there for me when it mattered.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Chapter No. and Title</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Introduction</td>
<td></td>
</tr>
<tr>
<td>1.1. PROJECT BACKGROUND</td>
<td>2</td>
</tr>
<tr>
<td>1.2. PROJECT AIMS AND THESIS OUTLINE</td>
<td>7</td>
</tr>
<tr>
<td>2. Literature Review</td>
<td></td>
</tr>
<tr>
<td>2.1. INTRODUCTION</td>
<td>10</td>
</tr>
<tr>
<td>2.2. THERMAL SHOCK OF BRITTLE MATERIALS: THE THERMAL SHOCK-INDUCED STRESS FIELD</td>
<td>11</td>
</tr>
<tr>
<td>2.3. EXPERIMENTAL METHODS</td>
<td></td>
</tr>
<tr>
<td>2.3.1. Introduction</td>
<td>20</td>
</tr>
<tr>
<td>2.3.2. Thermal Shock Simulation Methods</td>
<td>20</td>
</tr>
<tr>
<td>2.3.3. Assessment of Thermal Shock Damage</td>
<td>22</td>
</tr>
<tr>
<td>2.4. THERMAL SHOCK OF MONOLITHIC CERAMICS</td>
<td>25</td>
</tr>
<tr>
<td>2.5. THERMAL SHOCK OF PARTICLE- AND WHISKER-REINFORCED CMCS</td>
<td>30</td>
</tr>
<tr>
<td>2.6. THERMAL SHOCK OF FIBRE-REINFORCED CMCS</td>
<td>35</td>
</tr>
<tr>
<td>2.6.1. Introduction</td>
<td>35</td>
</tr>
<tr>
<td>2.6.2. Thermal Shock Damage and its Effect on Mechanical and Thermal Properties</td>
<td></td>
</tr>
<tr>
<td>2.6.2.1. Unidirectional (UD) CMCs</td>
<td>36</td>
</tr>
<tr>
<td>2.6.2.2. Cross-ply CMCs</td>
<td>39</td>
</tr>
<tr>
<td>2.6.2.3. 2-D and 3-D Woven CMCs</td>
<td>40</td>
</tr>
<tr>
<td>2.6.3. Studies of the Interface</td>
<td>45</td>
</tr>
<tr>
<td>2.7. THEORETICAL CONSIDERATIONS</td>
<td>46</td>
</tr>
<tr>
<td>2.8 CONCLUDING REMARKS</td>
<td>50</td>
</tr>
</tbody>
</table>
3. The Onset of Thermal Shock Fracture in UD CMCs

3.1. INTRODUCTION

3.2. THE ONSET OF PERPENDICULAR MATRIX CRACKING UNDER CONDITIONS OF THERMAL SHOCK

3.2.1. Derivation of the Predictive Model

3.2.1.1. The Condition for Cracking due to Thermal Shock
3.2.1.2. The Applied Stress Field
3.2.1.2.1. Thermal Shock-Induced Stresses
3.2.1.2.2. The Residual Stress Field
3.2.1.2.3. The Matrix Cracking Stress
3.2.1.3. Application of the Critical Condition For Fracture due to Thermal Shock
3.2.1.4. Comparison with Experimental Results
3.2.1.5. Discussion

3.2.2. Modelling Changes in Interfacial Shear Stress due to Thermal Shock

3.2.2.1. Introduction
3.2.2.2. Model Derivation
3.2.2.3. Application of the modified Coulomb-type Model
3.2.2.4. Discussion

3.2.3. Determination of the stress reduction factor ‘A’

3.2.4. The present approach as a predictive model for ΔTc

3.2.5. Discussion

3.3. FRACTURE ON THE END FACES OF UD NICALON/CAS UNDER CONDITIONS OF THERMAL SHOCK

3.3.1. Introduction

3.3.2. The Onset of Cracking

3.3.2.1. Strength-based Approach
3.3.2.1.1. The Critical Condition for Cracking due to Thermal Shock
3.3.2.1.2. The Thermal Shock-Induced Stress Field
3.3.2.1.3. Application of the Critical Condition for Cracking due to Thermal Shock
3.3.2.1.4. Discussion

3.3.2.2. Fracture Mechanics-based Approach
3.3.2.2.1. The Critical Condition For Cracking due to Thermal Shock
3.3.2.2.2. The Thermal Shock-Induced Stress Intensity Factor
3.3.2.2.3. The Fracture Toughness of the Transverse Face of a UD CMC
3.3.2.2.4. Application of the Critical Condition for Cracking due to Thermal Shock
3.3.2.2.5. Correlation with Experimental Results
3.3.2.2.6. Discussion
3.3.3. The Morphology of Cracks on End Faces of UD CMCs
   3.3.3.1. Analysis
   3.3.3.2. Discussion

3.4. CONCLUDING REMARKS

4. Materials and Experimental Techniques

4.1. MATERIALS

4.2. EXPERIMENTAL PROCEDURE
   4.2.1. Introduction
   4.2.2. Sample Preparation
   4.2.3. Thermal Shock Simulation Procedure
   4.2.4. Mechanical Testing
      4.2.4.1. Tensile Testing
      4.2.4.2. Flexure Testing
   4.2.5. Damage Observation

5. Thermal Shock Damage on Cross-Ply CMCs

5.1. INTRODUCTION

5.2. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED SIMPLE CROSS-PLY NICALON/CAS LAMINATES
   5.2.1. The (0°/90°)_{s} Laminate
      5.2.1.1. Critical Quenching Temperature Difference (ΔT_{c})
      5.2.1.2. Damage Modes due to Thermal Shock
         5.2.1.2.1. Nomenclature
         5.2.1.2.2. General
         5.2.1.2.3. Horizontal Matrix Cracks
         5.2.1.2.4. Perpendicular Matrix Cracks
      5.2.1.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT)
         5.2.1.3.1. General
         5.2.1.3.2. Description of Thermal Shock Damage Development
         5.2.1.3.3. Quantification of Thermal Shock Damage Development
   5.2.2. The (90°/0°)_{s} Laminate
      5.2.2.1. Critical Quenching Temperature Difference (ΔT_{c})
      5.2.2.2. Damage Modes due to Thermal Shock
         5.2.2.2.1. Nomenclature
         5.2.2.2.2. General
         5.2.2.2.3. Perpendicular Matrix Cracks
         5.2.2.2.4. Horizontal Matrix Cracks
5.2.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT)  
5.2.2.3.1. General 118  
5.2.2.3.2. Description of Thermal Shock Damage Development 119  
5.2.2.3.3. Quantification of Thermal Shock Damage Development 120

5.2.3. Summary 122

5.3. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED MULTI-LAYER CROSS-PLY NICALON/CAS LAMINATES 123  
5.3.1. The (0°/90°)3s Laminate 123  
5.3.1.1. Critical Quenching Temperature Difference (ΔTc) 123  
5.3.1.2. Damage Modes due to Thermal Shock 123  
5.3.1.2.1. Nomenclature 123  
5.3.1.2.2. General 123  
5.3.1.2.3. Horizontal Matrix Cracks 123  
5.3.1.2.4. Perpendicular Matrix Cracks 124  
5.3.1.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT) 125  
5.3.1.3.1. General 125  
5.3.1.3.2. Description of Thermal Shock Damage Development 128  
5.3.1.3.3. Quantification of Thermal Shock Damage Development 130

5.3.2. The (90°/0°)3s Laminate 133  
5.3.2.1. Critical Quenching Temperature Difference (ΔTc) 133  
5.3.2.2. Damage Modes due to Thermal Shock 133  
5.3.2.2.1. Nomenclature 133  
5.3.2.2.2. General 134  
5.3.2.2.3. Horizontal Matrix Cracks 134  
5.3.2.2.4. Perpendicular Matrix Cracks 134  
5.3.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT) 134  
5.3.2.3.1. General 134  
5.3.2.3.2. Description of Thermal Shock Damage Development 136  
5.3.2.3.3. Quantification of Thermal Shock Damage Development 139

5.3.3. Summary 141

5.4. DISCUSSION 143

5.5. CONCLUDING REMARKS 147
6. Thermal Shock Behaviour of Woven CMCs

6.1. INTRODUCTION 149

6.2. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED PW WOVEN NICALON/CAS 150
   6.2.1 Critical Quenching Temperature Difference ($\Delta T_c$) 150
   6.2.2. Damage Modes due to Thermal Shock 150
      6.2.2.1. Nomenclature 150
      6.2.2.2. General 151
      6.2.2.3. Perpendicular Matrix Cracks 151
      6.2.2.4. Horizontal Matrix Cracks 153
      6.2.2.5. Fibre Failures 154
   6.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential ($\Delta T$) 154
      6.2.3.1. General 154
      6.2.3.2. Description of Thermal Shock Damage Development 156
      6.2.3.3. Quantification of Thermal Shock Damage Development 159
   6.2.4. Discussion 161

6.3. MECHANICAL TESTING OF THERMALLY-SHOCKED PW WOVEN NICALON/CAS 165
   6.3.1. Introduction 165
   6.3.2. Tensile Testing 165
   6.3.3. Flexural Testing 170
   6.3.4. Discussion 174

6.4. CORRELATION OF CHANGES IN MECHANICAL PROPERTIES DUE TO THERMAL SHOCK WITH OBSERVED THERMAL SHOCK DAMAGE 176

6.5. CONCLUDING REMARKS 179

7. The Onset of Thermal Shock Fracture in 2-D CMCs

7.1. INTRODUCTION 181

7.2. THE CRITICAL CONDITIONS FOR CRACKING DUE TO THERMAL SHOCK 182
   7.2.1. Longitudinal ($0^\circ$) Plies 182
   7.2.2. Transverse ($90^\circ$) Plies 182
Chapter 1:

Introduction
1.1. PROJECT BACKGROUND

Ceramic matrix composites (CMCs) reinforced with ceramic fibres of various architectures have been identified as key material systems for improving the thrust-to-weight ratio of high-performance aircraft engines. It has been shown that even a small increase in the efficiency of gas turbines could save significant amounts of fuel costs while sharply reducing the output of environmental pollutants (Meetham and Van de Voorde 2000). Efficiency gains can be achieved by enhancing the working pressure ratio of the compressor, by increasing the temperature of the gases coming out of the combustion chamber and entering the turbine, as well as by reducing the weight of the engine.

Today, materials requirements are covered by the use of advanced nickel- and cobalt-based superalloys. However, the safe operation of these materials is only achieved by the application of suitable thermal barrier coatings (TBCs) on the surfaces of hot-section components and by active turbine blade cooling with air drawn from the compressor. Clearly, these materials have reached the limits of their development while the employment of suitable protection methods is expensive and detrimental to the efficiency of the machine (cooling air drawn from compressor reduces efficiency).

The requirements for the next generation of gas turbines in terms of refractoriness and high temperature stability are more than covered by another group of engineering materials, namely advanced ceramics. However, monolithic engineering ceramics are characterised by their propensity to fail in a brittle, catastrophic manner and show, from a designer’s point of view, an
 unacceptable variation in strength. Therefore, although there are continuing attempts to improve the suitability of monolithic ceramics for gas turbine applications, focus has shifted to an emerging class of ceramic materials, fibre-reinforced ceramic matrix composites (CMCs). The matrix is either a glass (e.g. borosilicate glass), a glass ceramic (e.g. aluminosilicate), or a ceramic material (e.g. SiC, Al₂O₃), although only the latter category satisfies the high temperature requirement (Harris 2000). The most widely used ceramic fibres are non-oxide SiC-based ones coming under the trade names Nicalon™, Tyranno™, and Sylramic™, as well as oxide-based ones such as the Nextel™ family (Ichikawa and Ishikawa 2000).

Fibre-reinforced CMCs not only exhibit the favourable high-temperature properties of their monolithic counterparts but are also tough, i.e. they are damage-tolerant, and show less variability in strength. This is achieved mainly through the incorporation of strong fibres, much stronger than the matrix, and effectively-engineered weak fibre-matrix interfaces. Under load, the weaker matrix cracks first and the advancing matrix crack is deflected at the fibre-matrix interface that debonds ahead of it. The intact fibres then bridge the crack opening and prevent catastrophic failure. The weak interface also activates a number of processes in the crack wake such as fibre sliding and fibre pull-out that consume energy and contribute to the increased toughness (Figure 1.1(a)) (Evans 1990). A typical stress-strain curve of a fibre-reinforced CMC (Figure 1.1(b)) shows non-linear behaviour after matrix cracking has occurred and the material continues to support load until the fibres fail.
Other advantages of CMCs compared with metals and alloys include their lower density and their lower thermal expansion coefficients. The combination of properties has the potential to lead to many important benefits for gas turbine engines such as reduction/elimination of cooling requirements, simpler component design, reduced weight of support structure, improved fuel efficiency, reduced emissions, higher blade frequencies, reduced blade clearances, longer service life, and higher thrust (DiCarlo 2001).

Extensive research programmes were performed in the USA, Europe and Japan during the 1980s and 1990s, targeted towards the production of successful, cost-effective, CMC systems to be used in rotating, as well as static, components of future jet engines. Potential gas turbine applications (Figure 1.2), apart from turbine vanes and blades, include shrouds, combustor liners, turbine discs, exhaust flaps, exhaust acoustic liners as well as inter-turbine transition ducts, fasteners, frame holders, and heat seals (Ohnabe et al. 1999).
In the course of these programmes, attention shifted in the 1990s from glass- and glass ceramic-matrix systems that have limited high temperature capabilities to ‘true’ ceramic composites, i.e. ceramic matrices reinforced with ceramic fibres that fulfil the high temperature requirements (Harris 2000). More specifically, attention has focused on SiC-matrix systems reinforced with woven fabrics of SiC-based fibres with appropriate interface coatings to ensure ‘tough’ behaviour, produced mainly by the CVI (chemical vapour infiltration) process (Lara-Curzio 2000). CMCs with woven reinforcement offer more balanced properties compared with other fibre architectures, combined with ease of handling and good drapability (Tan et al. 1997).

Considerable research has been undertaken on the room-temperature properties of these materials. Their elevated temperature properties have also been studied extensively since the requirements imposed involve operation at very high temperatures for tens of thousands of hours. However, little information seems to exist as far as the effect of single or repeated transient temperature gradients on their behaviour is concerned. This is of great importance
since, according to Bast (1993), an emergency shut-down of a gas turbine could lead to a
temperature decrease of more than 800°C within one second, a situation expected to occur about
100 times during the lifetime of modern gas turbine engines.

The performance of fibre-reinforced CMCs under thermal shock conditions is also of great
interest to the nuclear industry, where SiC/SiC composites are currently considered for
application as insulator and structural materials in fusion reactors due to the low neutron
activation for SiC and their good mechanical properties at high temperatures (Naslain 2004).
Thermal shocks are inflicted on the materials in this application during reactor start-up and shut-
down cycles as well as under plasma discharge conditions.

A limited number of articles concerned with the thermal shock behaviour of CMCs have
appeared in recent years in the open literature. Although it is generally acknowledged that fibre-
reinforced CMCs possess excellent thermal shock and thermal cycling resistance, especially
when compared with monolithic ceramics, due to their inherent damage tolerance, published
studies have shown that thermal transients affect their properties in a negative manner and could
have a detrimental effect on their long-term service life (Wang and Singh 1994). Therefore, it is
of great interest not only to study the response of CMCs under such thermal environments but
also to try to understand the underlying mechanisms of property degradation and to model their
effect on CMC behaviour.
1.2. PROJECT AIMS AND THESIS OUTLINE

The aim of this project is to develop a theoretical framework that will advance significantly current understanding of the thermal shock behaviour of fibre-reinforced CMCs. Using the results of Blissett (1995) in unidirectional CMCs as the starting point, experimental and analytical work is extended to CMCs containing 2-D fibre reinforcement. This involves identification and quantification of the main damage mechanisms as a function of the applied thermal shock, assessment of the effect of thermal shock on the residual properties of the material, and prediction of the onset of fracture in these materials under conditions of thermal shock.

The method to achieve the above aim incorporates a series of thermal shock tests, careful microscopic investigations of the thermally-treated material to reveal shock-related damage, a series of mechanical tests of thermally-shocked samples to assess mechanical property change, as well as closed-form analytical modelling using strength-based and fracture mechanics-based failure criteria.

The thesis begins with a thorough review of the thermal shock behaviour of CMCs (Chapter 2). This is performed with reference to the behaviour of monolithic ceramics, and includes both particulate- and fibre-reinforced systems. Fundamental theoretical concepts are also presented together with an overview of experimental techniques usually employed to study the behaviour of ceramics and CMCs under such conditions.
The theoretical basis for analytical prediction of the thermal shock resistance of fibre-reinforced CMCs is presented in Chapter 3. Models that can describe the different cracking features in UD CMCs are developed and are correlated with experimental results from the open literature.

Basic information about the materials used and the experimental techniques employed in this study are given in Chapter 4. Subsequently, the behaviour of cross-ply and woven CMCs under conditions of thermal shock is studied in depth in Chapters 5 and 6 respectively. Comprehensive descriptions of thermal shock damage and its accumulation are included and, in the case of the woven materials, are supplemented by detailed assessment of post-shock mechanical properties. This leads to Chapter 7, where the models developed in Chapter 3 for the UD material are extended and applied to 2-D CMCs (cross-ply and woven).

Finally, Chapter 8 summarises the main findings of this investigation and presents proposals for future work.
Chapter 2:

Literature Review
2.1. INTRODUCTION

The aim of this chapter is to present an overview of the performance of ceramic matrix composites (CMCs) under conditions of thermal shock, i.e. when they are subjected to sudden changes in temperature during either heating or cooling. The description of the thermal shock behaviour of CMCs is given with reference to the thermal shock resistance of monolithic ceramic materials. Monolithic ceramics have greater thermal shock sensitivity than metals and can even suffer catastrophic failure due to thermal shock because of an unfavourable ratio of stiffness and thermal expansion to strength and thermal diffusivity, and their limited plastic deformation.

The structure of the chapter is as follows: the stress field developed in a thermally-shocked component is described in section 2 and maximum stresses are identified and quantified. Section 3 contains an overview of the experimental methods used to simulate thermal shock conditions in the laboratory and the means utilised to assess its impact on ceramics and CMCs. The behaviour of monolithic ceramics is described in section 4 with reference to the main mechanical and thermal properties that affect it. In addition, methods to model this behaviour are presented. Section 5 concentrates on the thermal shock behaviour of particle- and whisker-reinforced CMCs while section 6 contains an extensive review of damage modes sustained in fibre-reinforced CMCs due to thermal shock and their effect on properties, the role of the interface and attempts to analyse and model the situation.

It should be noted that this review concentrates on thermal shock (i.e. a single thermal cycle) and no attempt is made to incorporate and describe the effects of cyclic thermal loading (cyclic thermal shock, thermal shock fatigue etc.) on the behaviour of CMCs.
2.2. THERMAL SHOCK OF BRITTLE MATERIALS: THE THERMAL

SHOCK-INDUCED STRESS FIELD

When a body is subjected to a rapid temperature change such that non-linear temperature gradients appear, stresses arise due to differential expansion of each volume element at a different temperature. The temperature at each point changes with time at a rate dependent on the coefficient of surface heat transfer (HTC) between the medium of different temperature and the body, the shape of the body, and its thermal conductivity. High HTCs, large dimensions, and low thermal conductivities result in large temperature gradients and, thus, large stresses. This leads to the establishment of a dimensionless parameter, the ‘Biot modulus’, for the description of the heat transfer condition (Kreith 1986):

\[
Bi = \frac{l h}{k}
\]  

[2.1]

where \( l \) is a characteristic material dimension (e.g. the half-thickness of a plate), \( h \) is the HTC between the body and the medium, and \( k \) is the thermal conductivity of the body. The larger the value of \( Bi \), the larger is the rate of heat transfer between a medium of different temperature and the body.

The sudden temperature change (\( \Delta T \)) that generates non-linear temperature gradients in a body and, as a consequence, thermal stresses is termed ‘thermal shock’. If \( \Delta T \) is positive (i.e. the temperature change is downward) the material is subjected to a ‘cold’ shock whereas if \( \Delta T \) is negative the material is subjected to a ‘hot’ shock. The term refers to a single thermal cycle (\( N=1 \)) in contrast to terms such as ‘thermal cycling’, ‘cyclic thermal shock’, and ‘thermal fatigue’ that apply to multiple thermal cycles (\( N>1 \)).
For the calculation of the thermal shock-induced stresses, we consider the plate of Fig. 2.1 of Young’s modulus $E$, Poisson’s ratio $v$, and with coefficient of thermal expansion (CTE) $\alpha$, initially held at temperature $T_i$.

![Fig. 2.1. Schematic of a plate of thickness $2H$ subjected to thermal shock.](image)

If the top and bottom surfaces of the plate come into sudden contact with a medium of lower temperature $T_\infty$ they will cool and try to contract. However, the inner part of the plate initially remains at a higher temperature which hinders the contraction of the outer surfaces giving rise to tensile surface stresses balanced by a distribution of compressive stresses at the interior. By contrast, if the surfaces come into contact with a medium of higher temperature $T_\infty$, they will try to expand. As the interior will be at a lower temperature, it will constrain the expansion of the surfaces, thus giving rise to compressive surface stresses balanced by a distribution of tensile stresses at the interior.

If perfect heat transfer between the surfaces and the medium is assumed (i.e. if $Bi \to \infty$) the surface immediately adopts the new temperature while the interior of the plate remains at $T_i$. Following Munz and Fett (1999), this case corresponds to having a plate that can expand freely
in the z-direction with suppressed expansion in the x- and y-directions. In the absence of displacement restrictions, the plate would expand along the x- and y- directions by thermal strains of:

\[ \varepsilon_x = \alpha(T_a - T_i) \quad [2.2] \]
\[ \varepsilon_y = \alpha(T_a - T_i) \quad [2.3] \]

Since thermal expansion in both directions is completely suppressed, elastic strains are created that compensate the thermal strains, i.e.

\[ \varepsilon_{el,x} + \varepsilon_{th,x} = 0 \quad [2.4] \]
\[ \varepsilon_{el,y} + \varepsilon_{th,y} = 0 \quad [2.5] \]

From equations [2.2], [2.3], [2.4] and [2.5] we have:

\[ \varepsilon_{el,x} = -\varepsilon_{th,x} = -\alpha(T_a - T_i) = \alpha(T_i - T_a) = \alpha \Delta T \quad [2.6] \]
\[ \varepsilon_{el,y} = -\varepsilon_{th,y} = -\alpha(T_a - T_i) = \alpha(T_i - T_a) = \alpha \Delta T \quad [2.7] \]

The elastic strains cause ‘thermal stresses’ along the x- and y- axes and can be written as:

\[ \varepsilon_{el,x} = \frac{\sigma_{TS}^x}{E} - \frac{\nu \sigma_{TS}^y}{E} \quad [2.8] \]
\[ \varepsilon_{el,y} = \frac{\sigma_{TS}^y}{E} - \frac{\nu \sigma_{TS}^x}{E} \quad [2.9] \]
By substituting [2.6] and [2.7] in [2.8] and [2.9] respectively and solving first for $\sigma_x^{TS}$ and then for $\sigma_y^{TS}$ we can obtain the thermal shock-induced stresses along the x- and y-axes as:

$$\sigma_x^{TS} = \sigma_y^{TS} = \frac{E\alpha\Delta T}{1-\nu}$$

Equation [2.10] shows that thermal shock induces a bi-axial stress field, the maximum value of which depends on the elastic properties of the material and the imposed temperature differential.

However, if the rate of heat transfer is not infinite the thermal shock-induced stresses will gradually build up and after some time reach a peak value that will be a fraction of the value given by [2.10]. The solution requires transient stress analysis such as those of Cheng (1951) and Manson (1966) with the assumption of the plate of Fig. 2.1 being infinite. Following Lu and Fleck (1998), the plate is initially held at temperature $T_0$ and at time $t=0$ its top and bottom faces (at $z=\pm H$) are suddenly exposed to a convective medium of $T_\infty$. The surface heat flow is assumed to satisfy:

$$k_z \frac{\partial T}{\partial z} = \mp h(T_\infty - T), \text{ at } z = \pm H$$

where $k_z$ is the thermal conductivity in the z-direction and $T(z,t)$ the temperature of the material. The plate is assumed to be a uniform, linear thermo-elastic solid and is analysed under the constraint that it is free to expand with vanishing axial force

$$\int_{-H}^{H} \sigma_x^{TS} \, dz = \int_{-H}^{H} \sigma_y^{TS} \, dz = 0$$

[2.12]
and vanishing normal stress in the through-thickness direction, i.e. \( \sigma_z = 0 \). The transient thermal shock-induced stresses, \( \sigma_x(z,t) = \sigma_y(z,t) \), associated with the temperature distribution \( T(z,t) \) are then given by:

\[
\sigma_x^{TS}(z,t) = \sigma_y^{TS}(z,t) = -\frac{E\alpha(T-T_i)}{1-\nu} + \frac{E\alpha}{(1-\nu)2H} \int_{-H}^{H} (T-T_i) \, dz \quad [2.13]
\]

To obtain the temperature distribution \( T(z,t) \) the heat flow in the through-thickness direction needs to be considered. This is governed by:

\[
\frac{\partial^2 T}{\partial z^2} = \frac{1}{k_z} \frac{\partial T}{\partial t}, \quad |z| \leq H \quad [2.14]
\]

This equation is solved with heat transfer boundary condition [2.11] by a standard separation-of-variables technique, to give:

\[
\frac{T_{(x,t)} - T_i}{T_i - T_\infty} = -1 + 2 \sum_{n=1}^{\infty} \exp\left(-\beta_n^2 \frac{k_z t}{H^2}\right) \times \frac{\sin \beta_n \cos(\beta_n z/H)}{\beta_n + \sin \beta_n \cos \beta_n} \quad [2.15]
\]

where \( \beta_n \) are the roots of \( \beta_n \tan \beta_n = Bi \). The thermal shock-induced stresses are obtained from [2.13] and [2.15], and are written in non-dimensional form as:

\[
\frac{-TS}{\sigma_x} = \frac{-TS}{\sigma_y} = \frac{\sigma_x^{TS}(z,t)}{E\alpha(1-\nu)^{-1}(T_i - T_\infty)} = \frac{\sigma_y^{TS}(z,t)}{E\alpha(1-\nu)^{-1}(T_i - T_\infty)}
\]

\[
= \frac{T_{(x,t)} - T_i}{T_i - T_\infty} \frac{1}{2H} \int_{-H}^{H} \frac{T_{(x,t)} - T_i}{T_i - T_\infty} \, dz
\]
\[ = 2 \sum_{n=1}^{\infty} \exp \left( -\beta_n^2 \frac{k_0 t}{H^2} \right) \frac{\sin \beta_n}{\beta_n + \sin \beta_n \cos \beta_n} \times \left\{ \cos \left( \frac{\beta_n z}{H} \right) - \frac{\sin \beta_n}{\beta_n} \right\} \]  

[2.16]

The evolution of dimensionless stresses is then plotted against dimensionless time \((\tilde{t} = k_0 t / H^2)\) at selected locations \((z/H)\) through the thickness of the plate and for various values of the Biot modulus. An example of such a plot is given in Fig. 2.2.

![Fig. 2.2. The evolution of dimensionless thermal shock-induced stress with dimensionless time at various locations through the plate thickness for \(Bi=10\) (after Lu and Fleck 1998).](image)

The plots show that under cold shock and for all values of \(Bi\), the maximum tensile stress is achieved at the surfaces while the maximum compressive stress is achieved at the centre of the plate. The opposite is true for hot shock conditions. The maximum tensile stress, \(\sigma_{max}^{TS}\), achieved at the surface during cold shock and at the centre during hot shock are then plotted against \(1/Bi\), as shown in Fig. 2.3.
Fig. 2.3. The maximum tensile thermal shock-induced stress achieved at the surface in cold shock and in the centre of the plate in hot shock as a function of $1/Bi$. Also shown are curve fits described by equations [2.17] and [2.18].

(after Lu and Fleck 1998)

It can be seen that $\sigma_{\text{max}}^{-\text{TS}}$ increases with increasing $Bi$ for both cold and hot shock whereas the maximum tensile stress developed at the surface during cold shock is always much higher than the peak tensile stress developed at the centre of the plate during hot shock. This observation, combined with the fact that brittle materials usually contain a distribution of surface flaws, means that cold shock is a much more dangerous condition for a brittle material.

The maximum surface stress in the infinite plate under cold shock is adequately described by the formula:

$$\sigma_{\text{max}}^{-\text{TS}} (\pm H, t^*) = \left( 1.5 + \frac{3.25}{Bi} - 0.5e^{-\frac{16}{Bi}} \right)^{-1}$$  \hspace{1cm} [2.17]

where $t^*$ is the time taken for this value to be reached. The maximum tensile stress at the centre of the plate for hot shock is given by:
\[
\frac{-\sigma^{TS}}{\sigma_{\text{max}}} (0, t^*) = \frac{0.3085}{1 + (2/Bi)}
\]  

[2.18]

Equation [2.17] can be written through [2.10] as:

\[
\sigma^{TS}_{\text{max}} = \frac{E\alpha\Delta T}{1 - \nu} \left(1.5 + \frac{3.25}{Bi} - 0.5e^{\frac{16}{Bi}}\right)^{-1}
\]  

[2.17]

and subsequently as:

\[
\sigma^{TS}_{\text{max}} = A \frac{E\alpha\Delta T}{1 - \nu}
\]  

[2.18]

In [2.18], the parameter 'A' is termed the 'stress reduction factor', given by:

\[
A = \frac{1}{1.5 + \frac{3.25}{Bi} - 0.5e^{\frac{16}{Bi}}} = \frac{1}{f(Bi)}
\]  

[2.19]

Equation [2.18] is the classic formula used to characterise thermal shock-induced stresses at the surfaces of brittle components during cold shock. The function \(f(Bi)\) can be written more generally as:

\[
f(Bi) = a + \frac{b}{Bi} - ce^{\frac{d}{Bi}}
\]  

[2.20]

where the values of \(a, b, c, d\) depend on the shape of the component and are determined by using analyses similar to the one presented above for an infinite plate. For example, it was shown that
for an infinite plate $a=1.5$, $b=3.25$, $c=0.5$ and $d=-16$ whereas for an infinite rod $a=1.5$, $b=4.67$, $c=0.5$, $d=-51$ (Manson 1966).

The value of $f(Bi)$ (and consequently $A$) depends on the Biot modulus, i.e. on the HTC, thermal conductivity and material dimensions. For severe shocks $Bi$ becomes very large, so $f(Bi)$ becomes $f(Bi) \approx a$, which leads to the maximum value of the stress reduction factor being given by $A \approx 1/a$ (Wang and Singh 1994).

Experimental evidence suggested that there is a critical value of the characteristic specimen dimension, $l_c$, above which $Bi$ (and consequently $A$ and the value of the shock-induced stress) becomes independent of material dimensions (Wang and Singh, 1994). Becher and Warwick (1993) showed graphically that this value may be approximated by:

$$l_c \approx \frac{b}{a} \frac{k}{h}$$

For example, for an infinite rod, the critical dimension is given by $l_c \approx 3.1k/h$. 
2.3. EXPERIMENTAL METHODS

2.3.1. Introduction

This section aims to present briefly the experimental methods used to evaluate the performance of ceramics and CMCs under conditions of thermal shock. Reference is made to techniques used to impose the actual thermal shock condition as well as the destructive and non-destructive methods employed to assess damage morphologies and changes in residual properties.

2.3.2. Thermal Shock Simulation Methods

The methods used to simulate thermal shock can be classified into two categories, depending on the sign of the temperature differential to which the material is exposed: (1) Quench Tests, when the material is subjected to a sudden temperature decrease ($\Delta T<0$), or (2) Fast Heating Methods, if a sudden increase in temperature ($\Delta T>0$) is involved.

In a quench test, the specimen is heated to a pre-determined temperature in a furnace and is held at that temperature for a certain period of time (~10-20 min) to allow for the furnace and specimen temperatures to reach equilibrium. A sudden temperature decrease is then brought about by bringing the heated specimen into contact with a cooling agent. The difference in temperatures between the specimen and the cooling agent is defined as the ‘quenching temperature difference’. The process is repeated for different furnace temperatures until the temperature at which fracture and/or property degradation is just initiated can be determined.
The difference in temperature between this and the medium is the ‘critical quenching temperature difference’, $\Delta T_c$.

Methods to cause rapid temperature decrease of the heated specimen include immersing the specimen into a quenching medium, subjecting it to a flow of high velocity cold air (Faber et al. 1981) or water (Absi and Glandus 2004), and contacting the specimen with a cold metal rod (Rogers and Emery 1992). The most popular method has been the first, while the most commonly used quenching medium is room-temperature water (Wang and Singh 1994). Other quenching media include boiling water (Becher 1981, Tiegs and Becher 1987), room-temperature air (Boccaccini et al. 1998 and 1999), glycerine oil (Ishitsuka et al. 1989, Uribe and Baudin 2003), silicone oil (Evans et al. 1975, Konsztowicz 1990 and 1993, Tancret and Osterstock 1997), ethylene glycol (Thompson and Rawlings 1991), methyl alcohol (Ishitsuka et al. 1989), liquid nitrogen (Lee et al. 1993, Tancret and Osterstock 1997), liquid metals (Henceke et al. 1984), pre-heated salt (Soboyejo et al. 2001), or fluidized beds (Morrel 1993, Schneibiel et al. 1998).

The advantages of the quench test include its simplicity and the well-defined temperature difference between sample and cooling agent. However, a major drawback is that the value of the HTC is often difficult to assess, especially for quenching into water where $h$ is affected by different boiling phenomena (Kreith 1986). In addition, the HTC is not a constant for a certain quenching medium, as it changes with specimen temperature and is affected by the surface finish of the specimen (Becher et al. 1980, Becher 1981, Becher and Warwick 1993). Quenching into media other than water results in significantly lower values of $h$ (Lee et al. 1993). For these reasons, quenching experiments are suitable for comparing materials but not for measuring absolute values (Pompe et al. 1993, Morrel 1993).
In a fast heating test, usually the central area of a specimen is quickly heated up by a heating source. Heating sources used include plasma jets, lasers, energetic electron beams, hot gas jets, arc discharges and hydrogen-oxygen flames (Pompe et al. 1993, Schneider and Petzow 1993). The fast heating test causes a different stress distribution in the specimen to the quench test, which results in the activation of a different population of flaws. The thermal shock resistance of a material can be evaluated by measuring the critical temperature difference for crack initiation (i.e. as in the quench test), by measuring the critical power of the heating source for failure, or by measuring the temperature gradient as a function of time and calculating the corresponding energy input and stress intensity factor (Wang and Singh 1994). Generally, the thermal shock induced by a heating source is considered to be much less severe than that imparted during a quench test, especially when room-temperature water is the quenching medium (Case 2002).

2.3.3. Assessment of Thermal Shock Damage

The impact of thermal shock on the properties of a ceramic or a CMC is assessed by means of destructive and non-destructive testing methods. Flexural or tensile (mainly for CMCs) tests of suitably-sized thermally-shocked specimens are usually employed to measure retained mechanical properties as a function of the temperature difference. The temperature differential for which a significant drop in property values is observed is the \( \Delta T_c \). For monolithic ceramics and particle- or whisker-reinforced CMCs the property under investigation is usually strength, whereas in fibre-reinforced CMCs a drop in Young’s modulus is usually a better indication of the onset of damage.
Alternative approaches, termed ‘indentation thermal shock tests’, with pre-cracks of known sizes have been used by several authors to assess thermal shock damage in monolithic ceramics. Knoop (Hasselmann et al. 1978, Faber et al. 1981) or Vickers (Gong et al. 1992, Osterstock 1993, Andersson and Rowcliffe 1996, Tancret and Osterstock 1997, Collin and Rowcliffe 1999 and 2000, Lee et al. 2002) indentations were made on rectangular bars, which were then heated to pre-determined temperatures and quenched into water. Crack extensions from the indentations were measured as a function of quench temperature differential, and the critical temperature for spontaneous crack growth (failure) was determined for the material. Fracture mechanics analyses, which took into account measured resistance-curve (R-curve) functions, were then used to account for the data trends.

In addition, it has been shown (Boccaccini et al. 2001, Chlup et al. 2001) that the chevron-notched specimen flexural technique (CN-technique) can be a reliable method to assess fracture properties (fracture toughness, work of fracture) in thermally-shocked brittle matrix composites reinforced by brittle fibres.

As an alternative to destructive methods, various non-destructive techniques have been employed to assess damage caused by thermal shock. These include the determination of the post-shock Young’s modulus using ultrasonics or through the identification of the mechanical resonant frequencies of the material (Carter et al. 1988, Lee and Case 1989 and 1990, Wang and Singh 1994, Wang et al. 1994 and 1996, Boccaccini et al. 1997 and 1998), the monitoring of the change in the spectra of ultrasonic pulses passed through a specimen before and after thermal shock (Thompson and Rawlings 1991), the acoustic emission technique (Evans et al. 1975, Konsztowicz 1990 and 1993, Rogers and Emery 1992), the measurement of the change in specific damping capacity ($Q^{-1}$) (Lee and Case 1989 and 1990, Boccaccini et al. 1997, 1998,
1999), and the measurement of thermal diffusivity before and after the shock using the ‘flash diffusivity’ method (Ellingson 1995, Graham et al. 2003).

In addition, optical microscopy (e.g. reflected light microscopy) and scanning electron microscopy have been used extensively for direct observation of crack patterns on suitably-polished surfaces of thermally-shocked samples. More recently, a thermal shock testing technique has been developed (Wereszczak et al. 1999) that uses a high-resolution, high-temperature infrared camera to capture the surface temperature distribution of a test specimen at fracture.
2.4. THERMAL SHOCK OF MONOLITHIC CERAMICS

The behaviour of ceramic materials under conditions of thermal shock is characterised by a number of parameters (figures-of-merit) that concentrate on either the initiation of cracking due to thermal shock or the resistance of a material to crack propagation during thermal shock (Kingery 1955, Hasselman 1970, 1978 and 1985). The first parameter is derived by considering [2.10] and assuming that fracture occurs when the thermally-induced stress, $\sigma^{TS}$, becomes equal to the strength of the material, $\sigma_t$. By solving [2.10] for $\Delta T (=\Delta T_c)$ we obtain the 'maximum allowable quenching temperature difference' for the onset of cracking under severe thermal shock conditions, i.e. conditions that approximate perfect heat transfer between the material surface and the quenching medium (e.g. in water quench), as:

$$ R = \frac{\sigma_t(1-\nu)}{E\alpha} $$

[2.22]

[2.22] shows that, for high resistance to crack initiation, high strengths combined with low stiffness and CTE are required. Under mild thermal shock conditions (e.g. in a boiling water quench) the thermal conductivity also becomes important and [2.22] is modified to give:

$$ R' = \frac{\sigma_t(1-\nu)k}{E\alpha} $$

[2.23]

The resistance to crack propagation is characterised by the following parameter:

$$ R'' = \frac{EG}{\sigma_t^2(1-\nu)} $$

[2.24]
where $G$ is the surface fracture energy. [2.23] shows that for better resistance to crack propagation high values of stiffness and toughness are required combined with low strengths.

Different parameters impose different requirements on ceramic materials depending on whether fracture resistance or crack propagation resistance is of prime importance. The values of the above parameters for a range of ceramic materials are presented in Table 2.1, where the property-dependence of thermal shock behaviour can be observed.

Table 2.1. Values of the thermal shock resistance parameters $R$, $R'$, $R''$ for a range of ceramic materials (after Munz and Fett 1999)

<table>
<thead>
<tr>
<th>Material</th>
<th>$R_0$ (K)</th>
<th>$R$ ($kW m^{-2}$)</th>
<th>$R''$ (mm)</th>
<th>$R'$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$</td>
<td>73</td>
<td>2.19</td>
<td>0.23</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>46</td>
<td>3.9</td>
<td>0.28</td>
<td></td>
</tr>
<tr>
<td>ZrO$_2$</td>
<td>324</td>
<td>2.7</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>SiC</td>
<td>206</td>
<td>66</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>Si$_3$N$_4$</td>
<td>495</td>
<td>75</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>BeO</td>
<td>342</td>
<td>20</td>
<td>0.10</td>
<td></td>
</tr>
<tr>
<td>Al$_2$TiO$_5$</td>
<td>47</td>
<td>36</td>
<td>0.71</td>
<td></td>
</tr>
</tbody>
</table>

*HPSN: Hot-pressed silicon nitride  
**RBSN: Reaction-bonded silicon nitride

A variety of other parameters have also been proposed that characterise the thermal shock behaviour of brittle materials under a range of different conditions, and these can be found in reviews such as that of Wang and Singh (1994).

Alternative analyses, aiming to combine the two different approaches, have been performed using fracture mechanics concepts. Hasselman (1969) considered a brittle solid that contained circular, uniformly-distributed Griffith microcracks. Crack instability due to thermal shock was assumed to take place by the simultaneous radial propagation of $N$ cracks of radius $l$ in a unit volume. Hasselman proposed that the driving force for crack propagation is derived from the elastic energy stored in the body at the instant of fracture. The total energy per unit volume of a body is the sum of the elastic energy and the fracture energy of the cracks, i.e.:
\[ W_t = \frac{3(L\Delta T)^2 E}{2(1-2\nu)} \left( 1 + \frac{16(1-\nu^2)NL^3}{9(1-2\nu)} \right)^{-1} + 2\pi NL^2 \gamma \]  

[2.25]

Cracks are unstable between those limits for which:

\[ \frac{dW_t}{dL} = 0 \]  

[2.26]

Combining [2.25] and [2.26] we get the critical quenching temperature difference as:

\[ \Delta T_c = \sqrt{\frac{\pi G(1-2\nu)^2}{2E\alpha^2(1-\nu^2)L}} + \sqrt{\frac{128\pi G(1-\nu^2)N^2L^5}{81E\alpha^2}} \]  

[2.27]

The analysis showed that a material containing short cracks, much smaller than a characteristic length \( L_m \), would propagate in an unstable manner at \( \Delta T_c \) due to the released elastic energy being converted into kinetic energy, towards a final crack length \( L_f \) and cause a drastic reduction in strength. For initially longer cracks, i.e. \( L > L_m \), or for short cracks that have reached \( L_f \), quenching at higher values of \( \Delta T \) causes propagation in a stable, quasi-static manner and the material shows a gradual strength decrease above the \( \Delta T_c \) associated with that particular crack size. In addition, it was shown that thermal shock resistance increased with increasing initial microcrack density.

Evans (1975), Evans and Charles (1977), and Emery (1980) performed more refined fracture mechanics studies regarding the onset and arrest conditions, Bahr et al. (1988) and Pompe (1993) extended this work and considered the propagation of multiple cracks, while Swain (1990) found that materials showing non-linear deformation and R-curve behaviour have a better
resistance to thermal shock. More specifically, the behaviour of a crack in the thermal shock-induced stress field was deduced from the dependence of the crack length on the stress intensity factor. Unstable propagation of a flaw in a brittle material under conditions of thermal shock was assumed to occur when the following criteria were satisfied:

\[ K > K_t, \frac{dK}{dL} > \frac{dK_t}{dL} \]  \hspace{1cm} [2.28]

where \( K \) is the thermal stress intensity factor, \( K_t \) the material fracture toughness \( (K_t) \) or the crack length-dependent critical stress intensity factor \( (K_t) \) for materials that exhibit \( R \)-curve behaviour, and \( a \) is the crack length. In the case where:

\[ K > K_t, \frac{dK}{dL} < \frac{dK_t}{dL} \]  \hspace{1cm} [2.29]

the flaw will propagate in a stable manner. Thus, by superimposing the \( K_t \)-curve of a material onto curves that describe the \( K \)-curve behaviour generated for a given thermal shock treatment, conditions of crack propagation and arrest were predicted. Such analyses verified Hasselman’s findings but were able to define onset and arrest conditions with better accuracy.

The analyses were also found to correlate well with experimental findings. High-performance engineering ceramics usually have high stiffnesses combined with low values of toughness. Due to careful processing conditions the number and size of flaws they contain are limited ensuring high strengths. The extent of the temperature differentials that they can sustain without cracking is then mainly dictated by the values of CTE and, to a lesser degree, by the values of thermal conductivity. Materials with lower CTE and high thermal conductivities can sustain higher \( \Delta T \)s.
However, all such materials suffer large and abrupt losses of strength at $\Delta T_c$ as crack propagation occurs in an unstable fashion.

By contrast, refractory or porous ceramic materials usually have low stiffnesses and contain a lot of large flaws or pores. These materials do not show a definite $\Delta T_c$ but exhibit a gradual reduction in strength starting at low $\Delta T$s. Examples of both types of materials are given in Fig. 2.4.

Fig. 2.4. The thermal shock behaviour of (a) monolithic alumina, and (b) porous SiC at different sintering temperatures (after Munz and Fett 1999).
2.5. THERMAL SHOCK OF PARTICLE- AND WHISKER-REINFORCED CMCS

The attraction in reinforcing ceramic matrices with particles or whiskers is that, with appropriate microstructural design and property tailoring, materials with property combinations not possible in monolithic ceramics can be obtained. In addition, the materials remain effectively isotropic and can be manufactured by well-established techniques already in use for the manufacture of monolithic ceramics (Hansson and Warren 2000).

It is the capability of property tailoring that gives particle- and whisker-reinforced CMCs the edge over monolithic ceramics under conditions of thermal shock. By choosing carefully the properties of the reinforcement, reductions in Young's modulus and CTE combined with increases in thermal conductivity compared with the unreinforced matrix material can be realised. Strict microstructural control during processing can result in fully dense, finely-grained materials with good adhesion between reinforcement and matrix that ensure high strengths. In this way, high critical temperature differentials for crack initiation can be achieved. In addition, the presence of the reinforcement results in the introduction of a number of energy-dissipating mechanisms such as crack deflection, crack bridging etc., which significantly improve toughness and damage tolerance. Thus, better resistance to crack propagation compared with monolithic ceramics is also possible. The result is a material that can sustain higher values of ΔT and, in addition, retain a higher percentage of its initial strength at ΔT > ΔT_c compared with its monolithic equivalent.

A number of experimental studies support the above analysis. Aghajanian et al. (1989) reported that the ΔT_c of low porosity alumina-matrix CMCs reinforced with aluminium particles increased compared with unreinforced alumina, while porous CMCs with the same constituents
behaved as refractory ceramics, i.e. displayed a low, but not definite, \( \Delta T_c \) and gradual reduction in strength with increasing \( \Delta T \). Similar refractory-type behaviour was observed by Aldridge and Yeomans (1999) in the case of a sintered alumina-matrix composite reinforced with 20 vol.% iron particles that contained increased levels of porosity. However, a similar hot-pressed CMC with low porosity exhibited much higher \( \Delta T_c \) and higher strength retention at \( \Delta T_c \) compared with monolithic alumina, Fig. 2.5). Zin and Batra (1999) showed theoretically that crack bridging by metal particles resulted in a significant reduction of the thermal shock-induced stress intensity factor.

Bannister and Swain (1990) and Swain (1991) investigated the thermal shock behaviour of ZrO\(_2\)-particle-reinforced Al\(_2\)O\(_3\) and AlN- and BN- particle reinforced TiB\(_2\) and reported higher thermal shock resistance compared with the respective monoliths as well as no significant reduction in post-shock flexural strength. This was attributed to the materials exhibiting \( R \)-curve behaviour due to the formation of microcracks around the reinforcing phases.

![Fig. 2.5. The thermal shock behaviour of hot-pressed alumina, hot-pressed and sintered alumina reinforced with iron particles (after Aldridge and Yeomans 1999).](image-url)
Wang et al. (2001) found improved resistance to thermal shock (by ~70°C) of a 6 vol.% tungsten carbide particle reinforced alumina compared with the unreinforced material, which was consistent with higher toughness, reductions in Young’s modulus and CTE, as well as the strong bonding of the reinforcement particles to the matrix (so that they did not act as strength-reducing flaws). Similar reasons were put forward by Uribe and Baudin (2003) to explain the increased thermal shock resistance of an alumina-matrix CMC reinforced with 10 vol.% aluminium titanate particles, and also by Nieto et al. (2004), who observed increased resistance to crack initiation and stable crack propagation in an alumina containing 10 vol.% sub-micron-sized AlN particles. In the case of alumina/silicon carbide-particle nanocomposites, Maensiri and Roberts (2002) observed superior resistance to thermal shock compared with the matrix material. However, since no changes in thermal or mechanical properties could be identified, the improvement was associated with a change in crack path (intergranular in alumina, transgranular in the nanocomposite).

Similar observations have been made regarding the thermal shock behaviour of whisker-reinforced CMCs. Tiegs and Becher (1987) reported no decrease in flexural strength following quenches into boiling water for a 20 vol.% SiC whisker-reinforced Al₂O₃. The authors noted that a small increase in thermal conductivity and a slight decrease in CTE of the composite, compared with the matrix material, could not account for the extent of the improvement in thermal shock resistance and attributed it to the interaction of microcracks with the reinforcement (i.e. crack arrest, deflection, etc.), which resulted in increased toughness. Similar conclusions were drawn by Collin and Rowcliffe (2001) who tested the same material using the indentation-quench method. Pettersson and Johnson (2003) identified a clear correlation between increased toughness and pronounced R-curve behaviour with improved thermal shock resistance to explain the behaviour of alumina reinforced with Ti (C, N) whiskers. Zhao et al. (2002)
investigated the thermal shock resistance of β-sialon matrix composites reinforced with titanium carbonitride whiskers and noticed that the addition of whiskers had no influence on the matrix microstructure, but their presence improved both the hardness and the fracture toughness of the CMCs. No unstable crack extension occurred in the composites for ΔT = 90-700°C, but above 700°C performance deteriorated as a result of severe oxidation of the whiskers.

Studies have also shown that, since the amount of reinforcement added affects all mechanical and thermal properties, there is an optimum volume fraction of particle or whisker reinforcement that should be added to the matrix material to ensure superior resistance to thermal shock (Becher 1981, Jia et al. 1996, Sbaizer and Pezzotti 2003, Pettersson and Johnson 2003). The shape of the reinforcement also plays an important role in determining behaviour under thermal shock. Sbaizer and Pezzotti (2003) showed that the use of coarse and elongated particles resulted in better CMC performance compared with the use of fine-grained particles.

The importance of careful tailoring of the constituents to achieve improved thermal shock resistance is highlighted by the study of Jia et al. (1996). The incorporation of SiC whiskers in Si₃N₄ resulted in the CMC having a lower ΔT_c than monolithic Si₃N₄, as the CMC had a slightly lower thermal conductivity but a much larger CTE compared with the unreinforced matrix. However, stable crack growth occurred in the CMC in contrast to unstable crack growth in the monolithic material, which was attributed to the presence of the reinforcement. It was concluded that unreinforced Si₃N₄ is more suitable for use under mild thermal shock conditions, where the objective is to avoid fracture, while the CMC should be used under severe thermal shock conditions, where initiation of cracking is unavoidable and resistance to crack propagation and post-shock strength retention become important.
The behaviour of particle- and whisker-reinforced CMCs under conditions of thermal shock can be modelled successfully using the fracture mechanics methods outlined in the previous paragraph (e.g. Aldridge and Ycomans 2001) while the thermal shock parameters (figures-of-merit) can also be useful for initial material comparison.
2.6. THERMAL SHOCK OF FIBRE-REINFORCED CMCS

2.6.1. Introduction

Although particle- and whisker-reinforced CMCs can exhibit better thermal shock behaviour compared with monolithic ceramics, generally they still show a step decrease in their strength at $\Delta T_c$. A combination of the properties of high-performance engineering ceramics with high $\Delta T_c$ and gradual strength reduction above $\Delta T_c$ (i.e. refractory-type behaviour) can only be realised with the incorporation of continuous ceramic fibres into ceramic matrices.

With optimum selection of fibres and matrices, favourable residual stress conditions can be established in the matrix, which lead to increased $\Delta T_c$. Above $\Delta T_c$, matrix cracks appear but the presence of crack-deflecting fibre-matrix interfaces ensures minimal effect on mechanical properties as the fibres remain largely unaffected. As damage is also confined mostly to the surface of the materials, changes in mechanical and thermal properties are more readily identified by means other than mechanical testing.

In the following paragraphs an overview of damage due to thermal shock and its effect on the mechanical properties of CMCs with different fibre architectures is provided for a number of different reinforcement architectures. Subsequently, the effect of thermal shock on interfacial properties is discussed, followed by a description of attempts to analyse and model the thermal shock behaviour of these materials.
2.6.2. Thermal Shock Damage and its Effect on Mechanical and Thermal Properties

2.6.2.1. Unidirectional (UD) CMCs

Bhatt and Phillips (1990) reported that thermal shock reduced the flexural mechanical properties of a UD composite comprising SiC-fibres in a reaction-bonded Si₃N₄ matrix but that it did not affect its tensile properties (Young’s modulus, ultimate strength, matrix cracking stress). It was suggested that the loss in flexural strength was caused by the loss of inter-ply integrity of the composite after matrix fracture and the failure mode changing from a tensile fracture to delamination driven by shear stress.

Matrix cracking due to thermal shock and its effect on the flexural properties of UD Nicalon™ fibre-reinforced composites with borosilicate glass (Pyrex™) and lithium aluminosilicate (LAS) matrices was described by Kagawa et al. (1989 and 1993). Damage was confined to the surface (two to three fibre diameters deep) and independent of ΔT. The Pyrex™-matrix system exhibited multiple matrix cracking perpendicular to the fibre axis at ΔTₜ=600°C, which coincided with a notable decrease in Young’s modulus and flexure strength. The decrease in E was attributed to matrix crack formation on the specimen surface, but the reduction in flexure strength was explained as a change in failure mode to interlaminar shear failure, caused by a reduction in interfacial shear strength due to thermal shock.

In the LAS-matrix system matrix cracks parallel to the fibre axis were mainly observed at ΔTₜ=800°C, accompanied by a reduction in Young’s modulus, although flexure strength seemed to remain unaffected by thermal shock treatment. This was attributed to the difference in the direction of matrix crack propagation in the two composites due to the formation of α-
spodumene-silica solution in the LAS matrix during thermal shock, which could have acted as a source of microcracking because of thermal expansion mismatch.

Multiple matrix cracking perpendicular to the fibre axis was also reported by Blissett et al. (1997) for a UD Nicalon™/CAS (calcium aluminosilicate) (Fig. 2.6).

![Image of multiple matrix cracking](image_url)

**Fig. 2.6.** Multiple matrix cracking perpendicular to the fibre axis due to thermal shock in UD Nicalon™/CAS (after Blissett et al. 1997)

The density of these cracks increased with increasing ΔT but showed a reduction for ΔT>800°C, which seemed to be consistent with the formation of strong silica bridging between the matrix and the fibres.

In addition to matrix cracking perpendicular to the fibre axis, matrix cracks also occurred parallel to the mid-plane of the laminate. These cracks were first seen on the end faces of the composite at ΔT_c=400°C (Fig. 2.7).
The depth the cracks penetrated into the matrix increased and their path geometries changed with increasing ΔT. These effects were attributed to the interaction of increasing applied thermal stresses with simultaneous reductions in the interfacial shear strength due to oxidation of carbon.

![Image](image.jpg)

**Fig. 2.7.** (a) Photomicrograph, and (b) schematic of matrix cracking on end face of UD Nicalon\textsuperscript{TM}/CAS (after Blissett et al. 1997).

Similar damage modes, termed ‘thermal debond cracks’ were observed by Graham \textit{et al.} (2003) on the end face of a thermally-shocked UD Nicalon\textsuperscript{TM}/LAS II composite. The authors highlighted the presence of high tensile radial stresses across the fibre-matrix interface, which favoured the appearance of such cracks, and noted that they tended to run through fibre-rich regions where these stresses are highest. Reductions in thermal diffusivity due to thermal debond crack formation were measured. The appearance of such damage modes may also be responsible for the change in failure mode under flexure reported for other systems (Bhatt and Phillips 1990, Kagawa \textit{et al.} 1993).
Blissett et al. (1998) reported that thermal shock effects on the residual flexural properties of the Nicalon™/CAS were more evident at intermediate temperature differentials, i.e. $\Delta T=450-600^\circ C$ and this was attributed to the observed matrix cracking.

2.6.2.2. Cross-ply CMCs

Blissett (1995) studied cross-ply Nicalon™/CAS laminates of two different lay-ups and reported that thermal shock damage within individual plies was similar to that seen in UD specimens of the same material (Blissett et al. 1997).

Initial damage in a $(0^\circ/90^\circ)_s$ laminate was sustained at $\Delta T_c=400^\circ C$ in the eight central $90^\circ$ plies, and consisted of a single thermal debond crack similar to the ones observed on the end faces of UD Nicalon™/CAS (Fig. 2.8).

![Fig. 2.8. Photomicrograph of thermal debond crack in the eight central $90^\circ$ plies of a $(0^\circ/90^\circ)_s$ Nicalon™/CAS laminate (Blissett 1995).](image)
As this damage mode is not observed under monotonic tensile or fatigue loading applied along the axis of the longitudinal fibres in the 0° plies (e.g. Pryce and Smith 1992), its appearance is indicative of the bi-axial nature of the thermal shock-induced stress field. Short cracks just crossing the interface between plies as a result of thermal shock treatment were also reported. It was noted that both damage modes became more pronounced at higher values of ΔT.

The second laminate, (0°/90°)_{3s}, exhibited only slightly different cracking features, attributed to the difference in the stacking sequences of the laminates. A major thermal debond crack appeared at ΔT_{c} =350°C and was confined to the two central 90° plies. Similar cracks were observed in some of the adjacent 90° plies but were less pronounced. At ΔT=400°C matrix cracks perpendicular to the longitudinal fibres appeared in the 0° plies. For higher values of ΔT, debond cracks were observed in most of the other 90° plies while the perpendicular matrix cracks were seen crossing to the adjacent 90° plies before being arrested by the horizontally-running debond cracks. However, the outer plies and the thinner 0° plies seemed to remain intact up to ΔT=720°C. Flexure strength and Young's modulus were found to decrease with increasing ΔT (Blissett et al. 1998).

2.6.2.3. 2-D and 3-D Woven CMCs

CMCs with 2-D woven fibre reinforcements have been found to possess higher resistance to thermal shock than unidirectional or cross-ply CMCs of the same constituents (Nicalon™ fibres and SiC matrices) and prepared by the same method (Chemical Vapour Infiltration-CVI) (Wang et al. 1997).
Only a slight drop in the flexural strength of a woven Nicalon™/Al₂O₃ composite was observed by Fareed et al. (1990) after quenching through ΔT=1000°C and 1200°C. This was attributed to the effectively engineered weak fibre/matrix interface.

Lamicq et al. (1986) reported that the bending strength of water-quenched woven SiC/SiC (CVI) specimens decreased slightly in the quench range ΔT=300-750°C, and then remained unchanged up to ΔT=1200°C. The composite also seemed to exhibit a steep R-curve behaviour.

Wang et al. (1994 and 1996) reported on the thermal shock behaviour of 2-D woven Nicalon™/SiC CMCs manufactured by CVI and polymer impregnation and pyrolysis (PIP), as well as that of a Nextel™-312/SiC (CVI) composite system. The Nextel™/SiC (CVI) system failed in post-quench flexure tests by fracture through the 2-D fibre planes and showed different critical temperature differentials for the onset of decrease in each of its macroscopic properties. Reduction in ultimate strength, σ_u, began at ΔT_c(σ_u)=400°C, matrix cracking stress, σ_mc, started to decrease at ΔT_c(σ_mc)=600°C, while the work of fracture (WOF) decreased continuously as ΔT increased. Reductions in thermal diffusivity with increasing values of ΔT were also reported for this system by Ellingson (1995).

The properties of the Nicalon™/SiC (PIP) system followed a similar pattern (ΔT_c(σ_u)=400°C, ΔT_c(σ_mc)=500°C), though this system failed through an interlaminar shear failure process (delamination) and the property reduction saturated at ΔT=600°C. The Nicalon™/SiC (CVI) system failed by fracture through fibre planes but its properties (σ_u, σ_mc, WOF) had the same critical temperature difference, ΔT_c=700°C. The pre- and post-quench stress-displacement curves for this material can be seen in Fig. 2.9.
However, measurement of the Young’s modulus of this system before and after quenching by means of a dynamic mechanical resonance technique showed the onset of decrease at \( \Delta T_c(E) = 400^\circ C \), i.e. significantly lower than the \( \Delta T_c \) of the other properties.

![Fig. 2.9. Effect of increasing \( \Delta T \) on stress-displacement curves of Nicalon\textsuperscript{TM}/SiC (CVI) -solid line corresponds to unshocked sample (after Wang et al. 1996).](image)

An assessment of the thermal shock damage of woven Nicalon\textsuperscript{TM}/SiC (CVI) composite specimens was performed by Webb et al. (1996), the results being confirmed subsequently by Kagawa (1997). It was noted that the many pores and irregularities in the matrix inherent to this particular composite geometry provide stress concentrations that amplify the thermal loading and create preferential sites for crack formation. For this reason, CVI-SiC composites exhibit lower \( \Delta T_c \) for the onset of cracking than monolithic SiC (Kagawa 1997). Three types of thermal shock-induced damage on the material surface were reported: (1) Matrix cracks that originated from the corners of uninfiltreated pores in regions outside fibre bundles. These cracks appeared at \( \Delta T=250^\circ C \) and did not penetrate deeply into the fibre bundles, though the penetration depth
increased with increasing $\Delta T$; (2) Matrix cracks between fibres within a fibre bundle. These occurred at $\Delta T=1000^\circ C$ and were similar to thermal shock damage observed by Kagawa et al. (1993) in UD CMCs; and (3) Degradation of the fibre-matrix interface and removal of fibres. This type of damage appeared at $\Delta T=600^\circ C$ but was attributed to both thermal shock and/or oxidation effects. In addition, matrix cracks that severed ligaments between cloths were seen at $\Delta T \geq 600^\circ C$ in the interior of thermally-shocked specimens (Figure 2.10).

![Fig. 2.10. Thermal shock damage to interior of Nicalon™/SiC (CVI) at $\Delta T=1000^\circ C$. (after Webb et al. 1996)](image)

The mechanism of formation of these cracks is not clear as thermal shock loading induces mainly high stresses at or near the surface. Webb et al. (1996) reported that further increases in $\Delta T$ increased the severity of all types of thermal shock damage.

Correlation of these observations with property measurements by Wang et al. (1996), led to the postulation that surface matrix cracks that appear at low $\Delta T (=250^\circ C)$ are not strength-controlling but they are responsible for the reduction in Young’s modulus observed at
$\Delta T_c(E) = 400^\circ C$. On the other hand, the interior cracks that severed links between fibre cloths at $\Delta T \geq 600^\circ C$ seem to affect the strength of the composite, which decreases after $\Delta T_c = 700^\circ C$. Such behaviour was summarised by Boccaccini (1998) in the graph of Fig. 2.11. It has to be noted that the behaviour of $E$ is also typical of some thermal properties of CMCs (Ellingson 1995, Graham et al. 2003). Note that there is no abrupt change in any property above $\Delta T_c$. However, if the strength of the fibre-matrix interface is strong, fibre-reinforced CMCs revert to behaviour typical of monolithic ceramics (Twitty et al. 1995).

![Fig. 2.11. Schematic diagram showing the variation of fracture strength ($\sigma$), Young’s modulus($E$), internal friction ($Q^{-1}$), and microcracking density ($\varepsilon$) with increasing shock severity. (after Boccaccini 1998)](image.png)

The thermal shock behaviour of a 3-D carbon fibre-reinforced SiC-matrix CMC manufactured by CVI was assessed using the air-quench method by Yin et al. (2002). Damage consisted of matrix cracks that induced a reduction in Young's modulus, strength, and work of fracture for $\Delta T > 700^\circ C$. 
2.6.3. Studies of the Interface

It appears that the strength of the fibre-matrix bond in thermally-shocked CMCs remains unaffected unless high temperature oxidation processes are involved. Boccaccini et al. (1999) did not identify any significant changes in the properties of the fibre-matrix interface of SiC/borosilicate glass composites as a result of thermal shock, while Chawla et al. (2001) observed only a slight decrease in the interfacial shear stress of a thermally-shocked Nicalon™-fibre SiC-whisker BMAS (barium magnesium aluminosilicate)-matrix hybrid composite. If heating and soaking at temperatures harmful to the integrity of the interface are involved prior to quenching, degradation due to oxidation processes occurs (Blissett et al. 1997 and 1998). In this case, changes in properties are explained as a combination of both oxidation and thermal shock (Graham et al. 2003). The oxidation of the carbon interface in Nicalon™-reinforced glass ceramic-matrix CMCs leads to cracks in the matrix causing fibre failure due to the resulting strong interfacial bond (Blissett et al. 1997).
2.7. THEORETICAL CONSIDERATIONS

Only a few studies have appeared in the literature regarding the analysis and modelling of the thermal shock behaviour of fibre-reinforced CMCs.

Wang and Chou (1991) studied numerically the 3-D transient thermal stress in angle-ply laminated composites caused by sudden changes in the thermal boundary conditions. The study showed that $\Delta T_c$ would be reduced if the fibre volume fraction, $V_f$, the CTE or the Young’s modulus of the composite increased, while it would increase with increasing thermal conductivity. By contrast, Boccaccini (1998) showed that increasing $V_f$ increased the $\Delta T$ for the onset of matrix cracking in glass and glass-ceramic matrix composites. Wang and Chou (1991) also demonstrated that the change in CTE had the biggest effect on $\Delta T_c$ while the change in thermal conductivity had the least influence. In addition, as the fibre orientation angle deviates from 45° towards 90° or 0° the interlaminar normal stress decreased while the in-plane thermal stress transverse to the fibre direction increased. This resulted in the initial failure mechanism changing from delamination to matrix micro-cracking.

Wang et al. (1996) performed a 1-D qualitative analysis using the stresses generated due to thermal shock and the residual stresses associated with the thermal expansion mismatch between the fibres and the matrix. The analysis showed that if $(\text{CTE})_f > (\text{CTE})_m$ then the matrix is under tension only in the radial direction and possible matrix cracking will be circumferential, while the fibre is under tension in all directions (longitudinal, radial, and circumferential), which may promote fibre damage. If $(\text{CTE})_f < (\text{CTE})_m$, then the matrix is under tension in both longitudinal and circumferential directions; hence, radial and normal-to-fibre matrix cracking will be possible. Moreover, the fibre is under compression in all three directions, so fibre damage will be
limited. Debonding would also be possible in both cases. As a confirmation, they applied their analysis to the results of Kagawa et al. (1993). In the Nicalon\textsuperscript{TM}/Pyrex\textsuperscript{TM} composite, where \((\text{CTE})_f < (\text{CTE})_m\), the matrix is under a tensile stress in the longitudinal direction, which dictates that cracks will be perpendicular to the fibre axis, as observed in the experiment. Conversely, in the LAS-matrix composite \((\text{CTE})_f > (\text{CTE})_m\), i.e. the matrix is under tension in the radial direction, which results in cracks parallel to the fibre.

Particular interest has been paid to the analytical prediction of the \(\Delta T_c\) for the onset of matrix cracking. Blissett et al. (1997) and Boccaccini (1998) considered the residual stresses present in the composite due to thermal expansion mismatch between fibre and matrix which, when superimposed to the applied thermal stresses, could lead to matrix cracking. Their approach was based on the assumption that the stress that produces matrix cracking would be the same whether applied mechanically or thermally. Hence, the matrix cracking stress \((\sigma_{mu})\) was equated with the critical thermal shock-induced stress \((\sigma^\text{T S})\), which is the thermal stress required to produce matrix cracking, taking also into account the effect of residual stress \((\sigma_r)\), i.e.

\[
\sigma_{mu} = \sigma^\text{T S} + \sigma_r \tag{2.30}
\]

Following [2.18], the critical thermal shock-induced stress is given as:

\[
\sigma^\text{T S} = \frac{AE\alpha\Delta T_c}{1-\nu} \tag{2.31}
\]

For Blissett et al. (1997), \(E, \alpha, \) and \(\nu\) are matrix properties whereas Boccaccini (1998) defines ‘effective’ values calculated using the rule of mixtures.
Blissett et al. (1997) used the concentric cylinder model of Powell et al. (1993) to obtain residual stresses whereas Boccaccini (1998) utilised the results of a simple force balance in 1-D performed by Wang et al. (1996), which gives the residual thermal stresses in the matrix along the axial direction as:

\[
\sigma_{\text{matrix}}^{r} = \frac{E_{m} \Delta \alpha \Delta T}{1 + \frac{E_{m} (1 - \gamma)}{E_{f} V_{f}}} \]

[2.32]

Different models were also used to obtain the matrix cracking stress \((\sigma_{\text{mu}})\) with Blissett et al. (1997) using the classic Aveston et al. (1971) (ACK) analysis and Boccaccini (1998) using the model of Pagano and Kim (Pagano and Kim 1994), which gives \(\sigma_{\text{mu}}\) as:

\[
\sigma_{\text{mu}} = \frac{K_{IC}}{2 \sqrt{\frac{r + s}{\pi}}} \]

[2.33]

where \(K_{IC}\) is the fracture toughness of the matrix, \(r\) is the fibre radius, and \(s\) is the fibre spacing.

The model assumes that there is no interaction between cracks, which Boccaccini (1998) explains as a plausible assumption in the early stages of thermal shock damage.

By solving the resulting expressions for \(\Delta T_{c}\), the values of the critical temperature differentials are obtained. These are given as:
\[
\Delta T_c = \frac{1 - \nu}{AE_m\alpha_m} \left[ \frac{6\Gamma_m E_f E_m V_f^2}{E_f r V_m} \right]^{\frac{1}{3}} - \sigma_r \quad (\text{Blissett et al. 1997}) \quad [2.34]
\]

\[
\Delta T_c = \frac{1 - \nu}{AE_c\alpha_c} \left[ \frac{K_{Ic,m}}{2\sqrt{(r + s)/\pi}} - \frac{E_m\Delta\alpha\Delta T}{E_m(1 - V_f) + E_f V_f} \right] \quad (\text{Boccaccini 1998}) \quad [2.35]
\]

In [2.34], \( \Gamma_m \) is the matrix fracture energy, \( \tau \) is the interfacial shear strength, and \( E_1 \) is the axial modulus of the composite.

Although the two approaches are very similar, the value of \( \Delta T_c \) in Boccaccini’s model does not depend on the interfacial shear stress \( \tau \), as a result of the model chosen for the value of matrix cracking stress. Blissett et al. (1997) suggested that their method was valid for the UD material providing that some key parameters (interfacial shear stress, matrix fracture energy) were determined independently.
2.8 CONCLUDING REMARKS

This chapter has reviewed the performance of CMCs under conditions of thermal shock. It has been shown that CMCs exhibit superior resistance to thermal shock, compared with their monolithic counterparts, as catastrophic failure can always be avoided. Resistance to higher temperature differentials and property retention after the onset of thermal shock cracking (especially in fibre-reinforced CMCs) can be realised, provided that the mechanical and thermal properties of CMCs are optimised by careful choice of their constituents.

The behaviour of particle- and whisker-reinforced CMCs can be adequately described by using and adapting the models and methodology developed for monolithic ceramics. By contrast, analysis and modelling of the performance of fibre-reinforced CMCs is a subject still in its infancy that requires further attention. The situation is very complex due to the variety of damage mechanisms developed in these materials (especially 2-D CMCs) and is further complicated due to their anisotropic character, the scarcity of experimental results, and the variety of manufacturing methods that result in materials with different design philosophies.
Chapter 3:

The Onset of Thermal Shock Fracture in UD CMCs
3.1. INTRODUCTION

The experimental findings reviewed in the previous chapter showed that ceramic matrix composites reinforced with unidirectional fibres exhibit two distinct fracture phenomena after being exposed to thermal shock loading.

More specifically, multiple matrix cracking perpendicular to the fibre direction was reported for the faces of the composite that contained longitudinal fibres, while matrix cracks with a characteristic morphology were identified in the central region of the end faces. Blissett et al. (1997) and Boccaccini (1998) have made some progress regarding prediction of the onset of multiple matrix cracking due to thermal shock. However, no explanation exists for the onset and appearance of the cracks seen traversing the end faces of UD CMCs.

The aim of the theoretical work included in this chapter is to provide a satisfactory explanation for all the observed phenomena. The first part is an attempt to predict the onset of multiple matrix cracking. The work of the abovementioned authors is the starting point. In the second part cracking on the end face is tackled. Initially, a simple model for the prediction of the critical temperature differential is developed. Then, a number of theoretical concepts are put forward in order to explain the morphology of the crack pattern.
3.2. THE ONSET OF PERPENDICULAR MATRIX CRACKING UNDER CONDITIONS OF THERMAL SHOCK

3.2.1. Derivation of the Predictive Model

3.2.1.1. The Condition for Cracking due to Thermal Shock

The approach of Blissett et al. (1997) and Boccaccini (1998) is followed and it is postulated that multiple matrix cracking perpendicular to the fibres occurs when the thermally-induced stresses along the fibre direction become equal to the stress required to cause matrix fracture. Thus, the thermal stresses in the matrix can be equated with the uniaxial matrix strength, i.e.:

$$\sigma_{1,M}^{th} = \sigma_{mu}$$ \hspace{1cm} [3.1]

where $\sigma_{1,M}^{th}$ describes the thermally-induced stresses in the matrix along the direction of the fibres. The thermally-induced stresses may comprise, apart from thermal shock-induced stresses, residual thermal stresses usually present in CMCs due to differences in the coefficient of thermal expansion between the matrix and the reinforcing fibres. Thus

$$\sigma_{1,M}^{th} = \sigma_{1,M}^{TS} + \sigma_{1,M}^{RES}$$ \hspace{1cm} [3.2]

where $\sigma_{1,M}^{TS}$ is the axial thermal shock-induced stress in the matrix. Accordingly, the critical condition for the onset of fracture [3.1] becomes through [3.2]:

$$\sigma_{1,M}^{TS} + \sigma_{1,M}^{RES} = \sigma_{mu}$$ \hspace{1cm} [3.3]
The parameters included in [3.3] need to be described analytically before the equation can be applied. This is the theme of the following paragraphs.

3.2.1.2. The Applied Stress Field

3.2.1.2.1. Thermal Shock-Induced Stresses

To obtain the shock-induced stress in the matrix, $\sigma_{1,M}^{TS}$, the full thermo-elastic stress field developed at the surface of the material during thermal shock needs to be characterised. Such an approach would require complex three-dimensional transient stress analysis similar to that performed by Wang and Chou (1985 and 1986). However, a simplified approach can be followed if only the maximum values of the induced stresses are taken into account.

Consider the surface of a rectangular plate of UD CMC initially at temperature $T_1$ (Fig. 3.1). The composite consists of a matrix of volume fraction $V_m$ with properties $E_m$, $\alpha_m$, $v_m$, which contains parallel fibres of volume fraction $V_f$ with properties $E_f$, $\alpha_f$, $v_f$.

![Fig. 3.1. A UD composite (fibres aligned along 1-direction) subjected to thermal shock. Thermal shock-induced stresses are also shown.](image)

If the material is rapidly cooled from $T_1$ to $T_o$ and perfect heat transfer between the plate and the cooling medium is assumed, the surface immediate adopts the temperature $T_o$ while the other parts of the plate remain at $T_1$. This case corresponds to having a plate that can freely expand in
the 3-direction (i.e. perpendicular to the plane of Figure 1), with suppressed expansion in the 1- and 2-directions. In the absence of displacement restrictions, the plate would expand along the 1- and 2- directions by thermal strains of:

\[ \varepsilon_{th,1} = \alpha_1(T_0 - T_1) \]  \[3.4\]

\[ \varepsilon_{th,2} = \alpha_2(T_0 - T_1) \]  \[3.5\]

The CTEs along the principal material directions are given by:

\[ \alpha_1 = \frac{E_m \alpha_m V_m + E_f \alpha_f V_f}{E_m V_m + E_f V_f} \]  \[3.6\]

\[ \alpha_2 = (1 + \nu_m)\alpha_m V_m + (1 + \nu_f)\alpha_f V_f - \alpha_1 \nu_{12} \]  \[3.7\]

In addition, the major Poisson’s ratio is given by:

\[ \nu_{12} = \nu_f V_f + \nu_m V_m \]  \[3.8\]

Since thermal expansion in both directions is completely suppressed, elastic strains are created that compensate the thermal strains, i.e.

\[ \varepsilon_{el,1} + \varepsilon_{th,1} = 0 \]  \[3.9\]

\[ \varepsilon_{el,2} + \varepsilon_{th,2} = 0 \]  \[3.10\]

From equations [3.4], [3.5], [3.9] and [3.10] we have:
\( \varepsilon_{el,1} = -\varepsilon_{sh,1} = -\alpha_1(T_o - T_i) = \alpha_1(T_i - T_o) = \alpha_1 \Delta T \) \[3.11\]

\( \varepsilon_{el,2} = -\varepsilon_{sh,2} = -\alpha_2(T_o - T_i) = \alpha_2(T_i - T_o) = \alpha_2 \Delta T \) \[3.12\]

The elastic strains cause ‘thermal stresses’ along the principal axes of the material and can be written as:

\[
\varepsilon_{el,1} = \frac{\sigma_{TS}^1}{E_1} - \frac{\nu_{21} \sigma_{TS}^2}{E_2}
\]

\[3.13\]

\[
\varepsilon_{el,2} = \frac{\sigma_{TS}^2}{E_2} - \frac{\nu_{12} \sigma_{TS}^3}{E_1}
\]

\[3.14\]

The Young’s moduli along the principal material directions and the minor Poisson’s ratio are taken as:

\[
E_1 = E_m V_m + E_f (1 - V_m)
\]

\[3.15\]

\[
E_2 = \frac{E_f E_m}{(E_f V_m + E_m V_f)}
\]

\[3.16\]

\[
\nu_{21} = \frac{\nu_{12} E_2}{E_1}
\]

\[3.17\]

It has to be noted that more sophisticated approaches could have been used to estimate transverse properties \((E_2\) and \(\nu_2\), e.g. Hashin (1979). However, in fibre-reinforced CMCs the differences between such models and the simple expressions adopted here are small.

By substituting \([3.11]\) and \([3.12]\) in \([3.13]\) and \([3.14]\) respectively and solving first for \(\sigma_{1TS}\) and then for \(\sigma_{2TS}\) we can obtain the thermal shock-induced stresses along the principal axes of the material as:
\[ \sigma_1^{TS} = AO_1 \Delta T \]  \hspace{1cm} [3.18] \\
\[ \sigma_2^{TS} = AO_2 \Delta T \]  \hspace{1cm} [3.19]

where:

\[ Q_1 = \frac{(E_1 \alpha_1 + \nu_{21} E_2 \alpha_2)}{(1-\nu_{21})} \]  \hspace{1cm} [3.20] \\
\[ Q_2 = \frac{\nu_{12} E_2 \alpha_1 + E_2 \alpha_2}{(1-\nu_{12})} \]  \hspace{1cm} [3.21]

The stress reduction factor, \( A \), has also been included in [3.18] and [3.19]. The thermal shock-induced stress in the matrix can be found by employing the iso-strain condition in the axial (1-) direction as:

\[ \varepsilon_1 = \varepsilon_1^m = \varepsilon_1^f = \frac{\sigma_1^{TS}}{E_1} = \frac{\sigma_{1,M}^{TS}}{E_m} = \frac{\sigma_{1,f}^{TS}}{E_f} \]  \hspace{1cm} [3.22]

where \( \sigma_{1,f}^{TS} \) is the thermal shock-induced stress in the fibres. Equation [3.22] yields through [3.18]:

\[ \sigma_{1,M}^{TS} = \left( \frac{E_m}{E_1} \right) \sigma_1^{TS} = \frac{AE_m Q_1 \Delta T}{E_1} \]  \hspace{1cm} [3.23]

At the onset of cracking \( \Delta T = \Delta T_c = T_{\text{max}} - T_0 \), where \( T_{\text{max}} \) is the temperature from which the material should be quenched in a medium of temperature \( T_0 \) for cracking to initiate.

### 3.2.1.2.2. The Residual Stress Field

As stated above, due to differences in CTE between the matrix and the reinforcing fibres, residual stresses are established in CMCs when they are cooled down from their high processing temperatures (e.g. \( >1200^\circ\text{C} \) for most glass ceramic-matrix composites). There have been a
number of attempts to quantify the magnitude of these stresses and model the effect they have on mechanical properties. Usually, co-axial cylinder models subject to thermo-mechanical loading are utilised (e.g. Kuntz et al. 1993) and include micro-mechanical analyses of stress transfer between fibre and matrix (e.g. Powell et al. 1993).

In this case, we use the model of Budiansky et al. (1986) that states that residual stresses are governed by the misfit strain, $\Omega$, between the fibre and the matrix, which if the misfit arises solely from thermal expansion differences is given by:

$$\Omega = (\alpha_m - \alpha_f) \Delta T_F$$  \[3.24\]

The parameter $\Delta T_F$ in [3.24] is the temperature difference between processing temperature ($T_p$)\(^*\) and the temperature of operation. For example, room temperature operation is at 20-22°C, whereas in the case of thermal (cold) shock it is at the temperature to which the material has been heated prior to quenching (i.e. $\Delta T_F = T_p - T_1$). The axial residual stress in the matrix, $\sigma_{1,M}^{RES}$, is then given by:

$$\sigma_{1,M}^{RES} = \frac{(1 + E_1/E_f)E_mE_fV_f\Omega}{2\left[1 - \frac{(1-2\nu_{12})}{2(1-\nu_{12})}\left(1 - \frac{E_1}{E_f}\right)\right]E_1(1-\nu_{12})}$$

$$= \frac{(1 + E_1/E_f)E_mE_fV_f(\alpha_m - \alpha_f)}{2\left[1 - \frac{(1-2\nu_{12})}{2(1-\nu_{12})}\left(1 - \frac{E_1}{E_f}\right)\right]E_1(1-\nu_{12})} \Delta T_F$$  \[3.25\]

This can be written more simply as:

$$\sigma_{1,M}^{RES} = \Theta_1 \Delta T_F$$  \[3.26\]

* In the case of a glass- or glass dominated- matrix CMC, $T_p$ is equal to the glass transition temperature of the glass.
At the onset of thermal shock cracking $\Delta T_F = T_p - T_{\text{max}}$. The model predictions were found to be in close agreement with the values obtained by the more complex, analytical model of Powell et al. (1993), which is based on the analysis of co-axial isotropic cylinders.

3.2.1.2.3. The Matrix Cracking Stress

The strength of the matrix in the direction parallel to the aligned fibres in UD CMCs has been the subject of numerous investigations (Parthasarathy et al. 2003). Blissett et al. (1997) used the classic model of Aveston et al. (ACK) (1971) which has been shown to be valid for the assumption of ‘long’ initial flaws and, thus, provides a lower bound estimate of the matrix strength (Marshall et al. 1986). By contrast, Boccaccini (1998) chose the model of Pagano and Kim (1994) which is valid for initial damage in the form of localised cracks that do not interact and arrest when they encounter the nearest fibre. The major difference between the two models has to do with the interfacial shear stress, $\tau$, included in the ACK model but absent from that of Pagano and Kim. Micrographs, such as that of Fig. 2.6, suggest that the ‘long’ crack assumption can be considered valid for the initial thermal shock damage observed on UD CMCs. In addition, the interfacial condition has been shown to be the single most important factor that determines the properties of fibre-reinforced CMCs under various conditions. Thus, the ACK model was determined to be more appropriate to describe the onset of thermal shock damage.

This gives matrix failure strain as:

$$
\varepsilon_{\text{mu}} = \left( \frac{12 \gamma_m E_m E_f V_f^2}{E_1 E_m^2 r V_m} \right)^{\frac{1}{3}}
$$  \hspace{1cm} [3.27]

where $\gamma_m$ is the fracture surface energy, and $r$ is the fibre radius. The stress in the matrix to initiate cracking is then given by:
\[ \sigma_{m\mu} = E_m \varepsilon_{m\mu} = \left( \frac{6\tau \Gamma_m E_m E_f V_f^2}{E_1 r V_m} \right)^{\frac{1}{3}} \]  

[3.28]

where \( \Gamma_m (= 2\gamma_m) \) is the matrix fracture energy.

3.2.1.3. Application of the Critical Condition For Fracture due to Thermal Shock

As all the parameters included in [3.3] have been determined, we can now proceed with the application of the critical condition in order to determine the critical quenching temperature difference, \( \Delta T_c \). Substituting [3.23], [3.26] and [3.28] in [3.3] we find:

\[
\frac{A E_m E_1 \Delta T_c}{E_1} + \Theta_1 \Delta T_F = \left( \frac{6\tau \Gamma_m E_m E_f V_f^2}{E_1 r V_m} \right)^{\frac{1}{3}}
\]  

[3.29]

By solving [3.29] for \( \Delta T_c \), we get the critical quenching temperature difference for the onset of matrix cracking as a function of \( \Delta T_F \) as:

\[
\Delta T_c = \frac{E_1}{AE_m E_1} \left[ \left( \frac{6\tau \Gamma_m E_m E_f V_f^2}{E_1 r V_m} \right)^{\frac{1}{3}} - (\Theta_1 \Delta T_F) \right]
\]  

[3.30]

In [3.30], \( \Delta T_c = T_{\text{max}} - T_0 \) and \( \Delta T_F = T_F - T_{\text{max}} \). As \( T_{\text{max}} \) features on both sides of the equation we can proceed by substituting \( \Delta T_c \) and \( \Delta T_F \) in [3.30] and, subsequently, solving for \( T_{\text{max}} \). In this way, we can find the temperature from which the material should be quenched into a medium of temperature \( T_0 \) to initiate multiple matrix cracking due to thermal shock as:
Finally, the critical temperature difference for the onset of multiple matrix cracking is given by:

\[
\Delta T_c = \left[ \left( \frac{6T_m E_m E_f V_j^2}{E_t r V_n} \right)^{\frac{1}{3}} - \Theta_1 T_p + \frac{AE_m Q_1 T_o}{E_1} \right] - T_o \quad [3.32]
\]

It can be noted that [3.32] provides \( \Delta T_c \) as a function of \( T_p, T_o \) and a number of material properties. Room-temperature values of these properties are usually employed as information of their change with increasing temperature is scarce. In the following paragraph, the predictions of [3.32] are compared with experimental data for a range of UD CMCs.

3.2.1.4. Comparison with Experimental Results


It should be noted that, in contrast to Blissett et al. (1997) (\( A = 0.5 \)) and Boccaccini (1998) (\( A = 0.6 \)), the stress reduction factor, \( A \), is not assigned a single pre-determined value but is
allowed to vary between 0 - 0.66. This is mainly because \( h \), on which the value of \( A \) depends, cannot be readily determined during a water quench test, and its value has been reported to vary between very low values and a maximum of 60 kW m\(^{-2}\) K\(^{-1}\) depending on temperature and material surface finish. In addition, \( A \) is a function of the size of the component under investigation. However, it has been shown that the maximum value \( A \) can attain in such tests is 0.66 (Wang and Singh 1994). The calculated and experimentally-determined values of \( \Delta T_c \) can be seen in Table 3.2.

### Table 3.1. Material properties used in the calculation of \( \Delta T_c \)

<table>
<thead>
<tr>
<th>Material</th>
<th>( E ) (GPa)</th>
<th>( \alpha ) ( \times 10^{-9} \text{K}^{-1} )</th>
<th>( v )</th>
<th>( \Gamma ) (J m(^{-2}))</th>
<th>( r ) (( \mu \text{m} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nicalon</td>
<td>190</td>
<td>3.3</td>
<td>0.2</td>
<td>-</td>
<td>8</td>
</tr>
<tr>
<td>CAS</td>
<td>90</td>
<td>4.6</td>
<td>0.25</td>
<td>25</td>
<td>-</td>
</tr>
<tr>
<td>Duran</td>
<td>63</td>
<td>3.3</td>
<td>0.2</td>
<td>7.5</td>
<td>-</td>
</tr>
<tr>
<td>Pyrex</td>
<td>63</td>
<td>3.3</td>
<td>0.2</td>
<td>7.5</td>
<td>-</td>
</tr>
<tr>
<td>LAS</td>
<td>83</td>
<td>0.9</td>
<td>0.3</td>
<td>30</td>
<td>-</td>
</tr>
</tbody>
</table>

### Table 3.2. The properties of the four CMCs under consideration used in the model and the experimentally-determined and predicted (through (3.32)) values of \( \Delta T_c \). The values of \( \tau \) used were experimentally-determined room-temperature ones obtained from the literature.

<table>
<thead>
<tr>
<th>Material</th>
<th>( V_f )</th>
<th>( \Delta T_F ) (°C)</th>
<th>( \tau ) (MPa)</th>
<th>( \Delta T_c ) (°C)</th>
<th>( \Delta T_c ) (°C) Predicted(^1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nicalon/CAS</td>
<td>0.34</td>
<td>1200</td>
<td>15</td>
<td>400</td>
<td>&gt;485</td>
</tr>
<tr>
<td>Nicalon/Duran</td>
<td>0.4</td>
<td>1000</td>
<td>14</td>
<td>~585</td>
<td>&gt;840</td>
</tr>
<tr>
<td>Nicalon/Pyrex</td>
<td>0.5</td>
<td>1000</td>
<td>~10(^2)</td>
<td>&gt;600</td>
<td>&gt;896</td>
</tr>
<tr>
<td>Nicalon/LAS</td>
<td>0.4</td>
<td>1350(^2)</td>
<td>2-3</td>
<td>800</td>
<td>&gt;900</td>
</tr>
</tbody>
</table>

\(^1\) Minimum predicted values of \( \Delta T_c \) corresponding to \( A=0.66 \)

\(^2\) Average value of \( \tau \), as reported values range from 2-20 MPa (Bleay and Scott 1991, Lewis 2000)

\[^3\] [3.26], [3.37] are not valid for UD Nicalon/LAS as the misfit stress does not arise solely from \( \Delta \alpha \) (Cao et al. 1990). Reported room-temperature residual stress values are \( \sigma_{RES}=50 \) MPa and \( \sigma_{RES}=20 \) MPa (Beyerle et al. 1992, Cao et al. 1990). Thus, \( \Theta_1 \) and \( \Theta_2 \) are back-calculated by applying [3.26] and [3.37] for \( \Delta T_F=1350^\circ \text{C}. \)
3.2.1.5. Discussion

Before discussing the results from the application of [3.32], it is interesting to compare the present approach with the models of Blissett et al. (1997) and Boccaccini (1998) (Equations [2.34]-[2.35]). To facilitate such a comparison, [3.30] can be written, after substitution of \( Q_1 \) from [3.20], as:

\[
\Delta T_c = \frac{(1-v_{12}v_{21})}{A(E_m/E_1)(E_1\alpha_1 + v_{21}E_2\alpha_2)}(\sigma_{mu} - \sigma_{1M}^{RES}) \quad [3.33]
\]

Although there are differences in \( \sigma_{mu} \) and \( \sigma_{1M}^{RES} \) depending on the models chosen for their description, the main difference is centred in the parameter \((1-v_{12}v_{21}/E_1\alpha_1 + v_{21}E_2\alpha_2)\) present in [3.33], which shows that, contrary to the other models for \( \Delta T_c \), the anisotropic properties of the material have been taken into consideration in the present approach. In addition, the appearance of the parameter \((E_m/E_1)\) shows that the critical condition for the onset of thermal shock fracture (equation [3.3]) has been applied in terms of matrix stresses. Blissett et al. (1997) chose to characterise matrix stresses by applying [2.10] using matrix properties, while Boccaccini (1998) seems to have applied [2.10] with volume-averaged properties in order to characterise the composite stresses required to cause matrix fracture.

Concentrating now on the application of [3.32], the results presented in Table 3.2 show that it significantly overestimates the \( \Delta T_c \) of all CMCs for all possible values of the stress reduction factor, \( A \). While it can be argued that the values of material properties included in [3.32] may change as a result of the short-term high-temperature exposure to which test specimens are subjected during a quench test, these changes are expected to be small and not enough to account for the discrepancies observed between experimental and predicted values of \( \Delta T_c \). However, further consideration of the analysis presented in section 3.2.1 reveals that, because of the bi-axial nature of the thermal shock-induced stresses, in addition to an axial thermal shock-induced
stress, $\sigma_1^{TS}$, there is a transverse thermal shock-induced stress, $\sigma_2^{TS}$, given by [3.19], that can be thought of as acting perpendicular to the fibre/matrix interface during the shock. Hence, $\sigma_2^{TS}$ may reduce the value of the radial clamping stress at the interface on which the value of the interfacial shear stress, $\tau$, is assumed to depend. This means that during the shock the value of $\tau$ is reduced from its room-temperature value to a significantly lower one, which will depend on the magnitude of the applied stress $\sigma_2^{TS}$. As can be seen from [3.32], a reduction in $\tau$ would cause a reduction in $\sigma_{min}$, which would in turn lead to lower predicted values of $\Delta T_c$. In other words, by taking into account the bi-axiality of the applied thermal stress field, it can be argued that the discrepancy between experimental and predicted values of $\Delta T_c$ is caused by the use in [3.32] of values of $\tau$ measured at room temperature.

It has to be noted that a different value of $\tau$ should be applicable during the shock even without taking into account the effect of $\sigma_2^{TS}$. This is because the clamping stress at the interface depends on the residual radial stress caused by differences in CTE between matrix and fibres as the CMC is cooled from its processing temperature. Since this stress is proportional to $\Delta T_F$ and the $\Delta T_F$ of heated material ($= T_p - T_{\text{max}}$) is lower than that of material at room temperature ($= T_p - T_0$), the value of the clamping stress, and consequently of $\tau$, should be changed at the onset of fracture.

As it has been qualitatively argued that the applied thermal stresses can be thought of as affecting the radial clamping stress at the fibre/matrix interface, an attempt is presented in the following section to model changes in $\tau$ at various $\Delta T$s using a modified Coulomb-type relationship usually employed to characterise the results of micro-indentation tests.
3.2.2. Modelling Changes in Interfacial Shear Stress due to Thermal Shock

3.2.2.1. Introduction

The enhanced toughness of fibre-reinforced CMCs with non-oxide matrices compared with monolithic ceramics is mainly achieved through properly engineered fibre-matrix interfaces that allow fibres to debond ahead of advancing cracks and slide past the matrix (Parthasarathy et al. 2003). One school of thought in the experimental analysis and description of interfacial behaviour in CMCs views the sliding stress at the fibre/matrix interface as a Coulomb law of friction (Kerans and Parthasarathy 1991, Jero et al. 1991, Mackin et al. 1992, Parthasarathy et al. 1997, Drissi-Habti and Nakano 1997). This means that the frictional stress is assumed to be directly proportional to the radial clamping stress, which includes contributions mainly from radial residual thermal stresses and fibre roughness-induced compressive stresses (Lewis 2000).

In the case of thermal shock, the interface can be considered to be under the influence of a tensile thermal shock-induced stress, given by [3.19], that reduces the magnitude of the radial clamping stress. The extent of this reduction at each $\Delta T$ would depend on the respective value of the thermal shock-induced stress. In the following paragraphs, a Coulomb-type expression is first derived for an interface under thermal shock and is then applied to the cases of the thermally shocked CMCs mentioned in paragraph 3.2.1.6.

3.2.2.2. Model Derivation

Following the review of the relevant theory presented by Drissi-Habti and Nakano (1997) and ignoring Poisson effects, we postulate that the value of interfacial shear stress, $\tau$, when the surface considered in Fig. 3.1 is subjected to a thermal shock $\Delta T$, is described by a Coulomb-type relationship of the form:

$$\tau = -\mu \sigma_2$$  \[3.34\]
In [3.34], \( \mu \) is the coefficient of friction between fibre and matrix and \( \sigma_2 \) is the clamping stress at the fibre/matrix interface. The value of \( \sigma_2 \) in the case of thermal shock treatment is given by:

\[
\sigma_2 = \sigma_2^{RS} + \sigma_2^{RA} + \sigma_2^{TS}
\]  

[3.35]

Equation [3.37] states that the value of the clamping stress, \( \sigma_2 \), depends on the difference between (a) the sum of the radial residual thermal stress, \( \sigma_2^{RES} \), and the fibre roughness-induced stress, \( \sigma_2^{RA} \), and (b) the thermal shock-induced radial stress, \( \sigma_2^{TS} \). So, [3.34] becomes through [3.35]:

\[
\tau = -\mu (\sigma_2^{RES} + \sigma_2^{RA} + \sigma_2^{TS})
\]  

[3.36]

We continue to employ the model of Budiansky et al. (1986) for the calculation of \( \sigma_2^{RES} \), in contrast to Drissi-Habti and Nakano (1997) and other workers. The model gives \( \sigma_2^{RES} \) as:

\[
\sigma_2^{RES} = \Theta_2 \Delta T_f
\]  

[3.37]

where:

\[
\Theta_2 = \frac{E_m(1-V_f)(\alpha_m - \alpha_f)}{2 \left[ 1 - \frac{1-2V_{12}}{2(1-V_{12})} \left( 1 - \frac{E_1}{E_f} \right) (1-V_{12}) \right]}
\]  

[3.38]

The model produces similar results to those of Powell et al. (1993) and agreement with the model employed in the analysis of micro-indentation tests is fair.

The fibre roughness-induced compressive stress, \( \sigma_2^{RA} \), is characterised by the following expression as (Drissi-Habti and Nakano 1997):
\[ \sigma_2^{\text{Red}} = C \left( -\frac{A_r}{r} \right) \]  \hspace{1cm} [3.39] 

where:

\[ C = \frac{E_m E_f}{E_f (1 + \nu_m) + E_m (1 - \nu_f)} \]  \hspace{1cm} [3.40] 

In [3.40], \( r \) is the fibre radius and \( A_r \) is the roughness amplitude of the fibre surface. Finally, [3.36] becomes through [3.19], [3.37] and [3.39]:

\[ \tau = -\mu \left[ (\Theta_2 \Delta T_f) + \left( -\frac{C A_r}{r} \right) + (\Delta Q_2 \Delta T) \right] \]  \hspace{1cm} [3.41] 

3.2.2.3. Application of the modified Coulomb-type Model

Equation [3.41] is now applied for the UD CMCs mentioned in paragraph 3.2.1.6, using the data presented in Tables 3.1 and 3.2, at increasing values of \( \Delta T \). The stress reduction factor, \( A \), is allowed to vary between 0 - 0.66, while the roughness amplitude of the Nicalon fibre has been determined to be \( \sim 30 \) nm with the use of atomic force microscopy (AFM) (Chawla et al. 1995, Vanswijgenhoven et al. 1998). In addition, a number of values have been reported for \( \mu \), mostly in the range 0 - 0.2, depending on whether the fibres have been coated before composite manufacture or a thin interfacial layer has been formed during processing due to fibre/matrix reaction (Lewis 2000). In this study, \( \mu \) values are obtained by applying [3.41] at room temperature (where \( \sigma_2^{\text{TS}} = 0 \)) using experimentally-determined \( \tau \) values reported in the literature (Table 3.2). The values obtained, which are presented in Table 3.3, are in good agreement with published results from micro-indentation studies (e.g. Lara-Curzio and Ferber 1994) determined that \( \mu = 0.05 - 0.06 \) for Nicalon/CAS).
Table 3.3. Calculated values of friction coefficient, $\mu$

<table>
<thead>
<tr>
<th>Material</th>
<th>Nicalon/CAS</th>
<th>Nicalon/Duran</th>
<th>Nicalon/Pyrex</th>
<th>Nicalon/LAS</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\mu$</td>
<td>0.0544</td>
<td>0.062</td>
<td>0.087</td>
<td>0.0144</td>
</tr>
</tbody>
</table>

The results of the application of [3.41] are presented in Fig. 3.2(a)-(d).
Fig. 3.2. Interfacial shear stress as a function of $\Delta T$ given by [3.41] for UD (a) Nicalon/CAS, (b) Nicalon/Duran™, (c) Nicalon/Pyrex™, and (d) Nicalon/LAS. The ranges observed at each $\Delta T$ correspond to $\Lambda = 0 - 0.66$. 
3.2.2.4. Discussion

In section 3.2.1 of this chapter it was argued qualitatively that the interfacial shear stress, $\tau$, which governs the mechanical behaviour of CMCs, may be reduced from its room-temperature to a lower value during thermal shock. Fig. 3.2(a)-(d) show that such reductions are possible if the thermal shock stress component $\sigma_{2}^{TS}$ is taken into consideration and modelled as acting perpendicular to the fibre/matrix interface.

The extent of such reductions depends on the relative magnitudes of $\sigma_{2}^{TS}$ and the residual clamping stress, $\sigma_{2}^{RES}$, since the roughness-induced clamping stress, $\sigma_{2}^{RA}$, is independent of temperature or temperature differential. In the case of Nicalon/CAS (Fig. 3.2(a)), $\sigma_{2}^{TS}$ and $\sigma_{2}^{RES}$ act in a synergistic way since the value of $\sigma_{2}^{TS}$ is increasing while $\sigma_{2}^{RES}$ is decreasing for increasing $\Delta T$. This causes large reductions in $\tau$ even at low values of $\Delta T$, which partly explains why this material has the lowest $\Delta T_c$ (the other reason being that the matrix is under residual tension in the fibre direction). The Pyrex™- and Duran™-matrix composites also exhibit large reductions in $\tau$ with increasing $\Delta T$, albeit of a smaller rate than the CAS-matrix system. This is because no thermal residual stresses are present in these materials, which is evident in Fig. 3.2(b) and (c) as $\tau$ becomes equal to its room-temperature value for $A = 0$ irrespective of $\Delta T$. In the LAS-matrix system, changes in $\tau$ for increasing $\Delta T$ are small as the effect of $\sigma_{2}^{TS}$ on $\tau$ opposes that of $\sigma_{2}^{RES}$. This can be observed in Fig. 3.2(d) as for $A = 0$ $\tau$ actually increases due to residual stress relaxation with increasing $\Delta T$.

It is also of interest to investigate whether the reductions in $\tau$ predicted by [3.41] correspond to the values of $\tau$ required for thermal shock cracking to initiate at the experimentally-observed values of $\Delta T_c$. The values $\tau$ should acquire so that [3.32] gives correct predictions can be
obtained by solving [3.32] for \( t \) and applying the resulting equation at the experimentally-determined values of \( \Delta T_c \) (Table 3.2). Equation [3.32] becomes:

\[
\tau = \left( \frac{E_r r V_m}{6 F_m E_m E_f V_f} \right) \left( \frac{E_m}{E_f} \right) \Delta Q_i \Delta T_c + \Theta_i \Delta T_f \]  

[3.42]

Similarly, [3.41] is applied for the known values of \( \Delta T_c \). In both equations, \( A \) is again varied from 0 - 0.66. The results are presented in Table 3.4.

<table>
<thead>
<tr>
<th>( \tau ) (MPa)</th>
<th>Nicalon/CAS ( (\Delta T_c=400^\circ C) )</th>
<th>Nicalon/Duran ( (\Delta T_c=585^\circ C) )</th>
<th>Nicalon/Pyrex ( (\Delta T_c&gt;600^\circ C) )</th>
<th>Nicalon/LAS ( (\Delta T_c=800^\circ C) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>from [3.42]</td>
<td>0.4-11.3</td>
<td>0-5.2</td>
<td>0-3.1</td>
<td>0-1.4</td>
</tr>
<tr>
<td>from [3.41]</td>
<td>5.6-13.5</td>
<td>1.44-14</td>
<td>1.64-10</td>
<td>0.5-2.4</td>
</tr>
</tbody>
</table>

As it can be seen, the values of \( \tau \) predicted by [3.41] and [3.42] for the same \( \Delta T_c \) overlap for all CMCs under consideration. This is better visualised if the values predicted by [3.42] for increasing \( \Delta T \) are superimposed on the graphs of Figures 3.2(a)-(d). An example (for Nicalon/CAS) is shown in Fig. 3.3. Similar graphs can be obtained for the other three CMCs.

**Fig. 3.3. Change in \( \tau \) for increasing \( \Delta T \) from [3.41]-dotted line and from [3.42]-continuous line.**
Table 3.4 and Fig. 3.3 reveal that the proposed modified Coulomb-type model for the description of possible changes in $\tau$ during thermal shock not only predicts correctly the trends of the changes of $\tau$ with $\Delta T$ but can also provide quantitative estimates for the values of $\tau$ as a function of $\Delta T$ that correlate well with experimental data.

It has to be noted that more sophisticated approaches for modelling $\tau$ are available in the literature, e.g. Kuntz et al. (1993). Use of such models will share similar features with the present one (e.g. inclusion of $\sigma_2^{TS}$) and may describe the underlying phenomena more accurately, especially if phenomena such as the Poisson effect are shown to be significant during thermal shock.

### 3.2.3. Determination of the stress reduction factor ‘$A$’

The previous analysis was performed by assuming that the stress reduction factor, $A$, varied from 0-0.66 for reasons mentioned in paragraph 3.2.1.6. As we have now established that the two equations describing change in $\tau$ with $\Delta T$ (i.e. [3.41] and [3.42]) produce overlapping results, estimates of $A$ can be obtained. This is achieved by plotting $\tau$ (obtained from both equations) against $A$ (=0-0.66) for all $\Delta T$s. The point where the two branches corresponding to the experimentally-determined $\Delta T_c$ meet denotes the value of $A$ for the particular material and quenching medium. The resulting graphs for the composites under consideration are presented in Fig. 3.4(a)-(d). The values of $A$ derived for each composite are presented in Table 3.5.

Although these values can only be considered simple estimates as they are subject to inaccuracies regarding material properties and limitations of the incorporated models, it is
interesting to note that they all fall within the range $A=0.5-0.6$. The same limits to the value of $A$ can be set by combining the work of Blissett et al. (1997) and Boccaccini (1998).
Fig. 3.4. Determination of the stress reduction factor, A, for water-quenching of UD (a) Nicalon/CAS, (b) Nicalon/Duran™, (c) Nicalon/Pyrex™, and (d) Nicalon/LAS.
Table 3.5. Values of the stress reduction factor, $A$, for four different UD CMCs determined by the method outlined in section 3.2.3.

<table>
<thead>
<tr>
<th>Material</th>
<th>Nicalon/CAS ($\Delta T_c$=400°C)</th>
<th>Nicalon/Duran ($\Delta T_c$=585°C)</th>
<th>Nicalon/Pyrex ($\Delta T_c$&gt;600°C)</th>
<th>Nicalon/LAS ($\Delta T_c$=800°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A$</td>
<td>0.53</td>
<td>0.57</td>
<td>&lt;0.6</td>
<td>0.54</td>
</tr>
</tbody>
</table>

3.2.4. The present approach as a predictive model for $\Delta T_c$

The above analysis can also be used to obtain approximate estimates for the $\Delta T_c$ of UD CMCs. More analytically, we can substitute the value of $\tau$ from [3.41] in [3.32], then solve for $T_{\text{max}}$ and subsequently determine $\Delta T_c (= T_{\text{max}}-T_o)$. Two estimates can then be obtained; the first assuming an average value of the stress reduction factor $\overline{A} = 0.55$, which seems to be a valid approximation at least for this class of CMCs, and the second assuming the maximum possible value of $A$, i.e. $A = 0.66$. Both estimates can also be determined graphically using the plots of Fig. 3.4(a)-(d). The values of $\Delta T_c$ obtained are presented in Table 3.6.

Table 3.6. Estimates of $\Delta T_c$ for four different UD CMCs determined by the methods outlined in section 3.2.4.

<table>
<thead>
<tr>
<th>$\Delta T_c$ ($^\circ$C)</th>
<th>Nicalon/CAS ($\Delta T_c$=400°C)</th>
<th>Nicalon/Duran ($\Delta T_c$=585°C)</th>
<th>Nicalon/Pyrex ($\Delta T_c$&gt;600°C)</th>
<th>Nicalon/LAS ($\Delta T_c$=800°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\overline{A} = 0.55$</td>
<td>370</td>
<td>607</td>
<td>671</td>
<td>780</td>
</tr>
<tr>
<td>$A = 0.66$</td>
<td>&gt;300</td>
<td>&gt;500</td>
<td>&gt;550</td>
<td>&gt;650</td>
</tr>
</tbody>
</table>

The estimates for $\overline{A} = 0.55$ give the best agreement with experimental data as the error in the calculated values of $\Delta T_c$ ranges from 2.5-12%. However, the approximation of $\overline{A} = 0.55$ cannot be guaranteed to be reasonable in the case of other CMCs with appreciably different thermal and mechanical properties. For $A = 0.66$, only fair agreement with experimental data is achieved as the error ranges between 8-25%. However, the value of $\Delta T_c$ obtained is the most conservative
possible and can be utilised as a guideline indicating the worst case scenario, i.e. the resistance of
the material to thermal shock under the most severe conditions possible.

### 3.2.5. Discussion

A theoretical investigation of the conditions for the onset of multiple matrix cracking due to
thermal (cold) shock in UD CMCs was conducted in this study. A model was developed to
predict the critical quenching temperature differential, $\Delta T_c$, that required as inputs the processing
temperature of the composite, the temperature of the quenching medium, and material properties
measured at room-temperature. The approach considers the anisotropic stress field generated
during the shock, which is an improvement compared with available models. Numerical
predictions for water-quenched CAS-, Pyrex™-, Duran™-, and LAS-matrix composites
reinforced with Nicalon fibres suggested that the value of the effective interfacial shear stress
may be significantly reduced during the shock compared with the standard (room temperature)
value due to the presence of a thermal shock-induced stress component that acts perpendicular to
the fibre/matrix interface. The phenomenon was modelled successfully using a modified
Coulomb-type friction law and satisfactory correlation with experimental data was achieved.
Subsequently, the combined models were used to provide predictions for the stress reduction
factor that characterises the heat transfer condition during the shock. An average value of $A =
0.55$ was determined to be a satisfactory approximation for this type of CMCs that have similar
mechanical and thermal properties. A methodology based on the assumed average and the
maximum value of the stress reduction factor provided satisfactory and conservative estimates of
$\Delta T_c$ respectively.
3.3. FRACTURE ON THE END FACES OF UD NICALON/CAS UNDER CONDITIONS OF THERMAL SHOCK

3.3.1. Introduction

After analysing multiple matrix cracking at right angles to the fibre direction on faces containing longitudinal fibres of a UD CMC, work now proceeds to analyse the damage reported on the end faces of this material. Such cracks originate at the centre of the face and run parallel to the long top and bottom edges (Fig. 2.7).

This part of the chapter is divided into two distinct sections. The first section presents methods to predict the onset of fracture due to thermal shock. Both strength- and fracture mechanics-based approaches are presented. In the second section, the morphology of the cracks is explained.

3.3.2. The Onset of Cracking

3.3.2.1. Strength-based Approach

3.3.2.1.1. The Critical Condition for Cracking due to Thermal Shock

The first way we can approach this problem is by considering the stress field developed on the end (or transverse) face of a UD CMC under conditions of thermal shock. In such a case, fracture due to thermal shock should occur when the thermal shock-induced stress becomes equal to the mechanical strength of the transverse face, $S_{TR}$, i.e. when:

$$\sigma_3^{TS} = S_{TR}$$  \[3.43\]
The stress induced by thermal shock needs to be defined before application of [3.43] can proceed.

3.3.2.1.2. The Thermal Shock-Induced Stress Field

This face can be considered to be macroscopically isotropic. If, as in the previous section, only maximum values of stress at the surface are taken into account, then the thermal shock-induced stress, following the nomenclature of Fig. 3.5, is given by:

\[
\sigma_{3}^{TS} = AQ_3 \Delta T \tag{3.44}
\]

Since the UD material is transversely isotropic,:

\[
Q_3 = \frac{E_3 \alpha_3}{1 - \nu_{23}} = \frac{E_2 \alpha_2}{1 - \nu_{23}} \tag{3.45}
\]

Fig. 3.5. Schematic of the transverse face of a UD CMC under thermal shock. Thermal shock-induced stresses are also indicated.
3.3.2.1.3. Application of the Critical Condition for Cracking due to Thermal Shock

By applying [3.43] using [3.44], we have at the onset of fracture:

\[ S_{TR} = \Delta Q_3 \Delta T_c \]  

[3.46]

which leads through [3.45] to:

\[ \Delta T_c = \frac{S_{TR} (1 - \nu_{23})}{AE_{2} \alpha_2} \]  

[3.47]

In [3.47], \( E_2 \) and \( \alpha_2 \) are given by [3.16] and [3.7] respectively, \( \nu_{23} \) can be assumed to be equal to \( \nu_{21} \) (given by [3.17]), and \( \Lambda = \bar{\Lambda} = 0.55 \).

3.3.2.1.4. Discussion

Such an approach is difficult to implement as there is no model that can provide a valid value for \( S_{TR} \). Studies of cross-ply CMCs (e.g. Pryce 1991) have shown that the strength of the transverse ply depends on its thickness and the problem is better approached using fracture mechanics methods. This is better shown if we calculate the required transverse strength of a UD Nicalon/CAS so that [3.47] predicts the correct, experimentally-determined, \( \Delta T_c \). By solving [3.47] for \( S_{TR} \) and putting \( \Delta T_c = 400^\circ \text{C} \) (Blissett et al. 1997) we get \( S_{TR} \approx 130 \text{ MPa} \). This is a very high value since most studies have shown that the strength of much thinner transverse plies in cross-ply Nicalon/CAS is between 25-40 MPa (Pryce and Smith 1992, Beyerle et al. 1992) depending on the laminate configuration. Even if residual stresses at the ply level are taken into account (~25 MPa tensile for the transverse plies according to Beyerle et al.), the strength of plies much thinner than the transverse face of a UD Nicalon/CAS is more than 50% less than the required one. For the transverse face of UD Nicalon/CAS to fracture under such a large thermal shock-induced stress, the flaw length that comes under this stress must be very small. This
implies that maybe not the whole area of the face suffers the maximum applied thermal stress, an issue which will be addressed later in this chapter. In the following section, fracture mechanics methods are applied in an attempt to investigate the above effects further.

3.3.2.2. Fracture Mechanics-based Approach

3.3.2.2.1. The Critical Condition For Cracking due to Thermal Shock

Fracture initiates when the applied thermal shock stress intensity factor, $K_{fTS}$, becomes equal to the fracture toughness, $K_{IC}$, of the material, i.e. when:

$$K_{fTS} = K_{IC}$$  \[3.48\]

Before we apply this condition, we need to calculate the applied stress intensity factor as a result of thermal shock loading and define the fracture toughness of the material.

3.3.2.2.2. The Thermal Shock-Induced Stress Intensity Factor

According to Blissett et al. (1997), fracture in Nicalon/CAS originates at the centre of the end face. Thus, if we assume the existence of a pre-existing surface crack of half-length $c$, the thermal shock-induced stress intensity factor at the tip of this central crack, $K_{fTS}$, will be given by:

$$K_{fTS} = \sigma_{3TS}^2 \sqrt{\pi c}$$  \[3.49\]

Equation [3.49] becomes through [3.44] and [3.45]:

$$K_{fTS} = \sigma_{3TS}^2 \sqrt{\pi c}$$  \[3.49\]
In order to apply [3.50] we need an estimate of the original crack half-length, \( c \). However, this can not be determined with any certainty and only estimates can be made.

Alternatively, the results of different approaches can be considered. Such analyses consider the variation of the full thermo-elastic stress field across the through-thickness direction and utilise fracture mechanics by assuming a pre-existing flaw in the depth direction. An example is given by the analysis of Zhao et al. (2000).

![Diagram of a plate of thickness 2H subjected to thermal shock](image)

**Fig. 3.6.** Schematic of a plate of thickness 2H whose top and bottom faces are subjected to thermal shock (after Zhao et al. 2000)

In Fig. 3.6, a component with thickness 2H is subjected to thermal shock at the top and bottom faces. The authors assume that there is a pre-existing crack of length \( H \) and, based on this, derive expressions for the applied stress intensity factor due to thermal shock taking into account the varying nature of the applied stress across the thickness. According to thermal shock theory (see Chapter 2), the half-thickness \( H \) should be equal to or greater than a critical dimension, \( t_c \), such that the stress at the top and bottom faces can reach its maximum value, i.e. \( H \geq t_c \). If \( H \) is made...
equal to the critical dimension, \( t_c \), the equation derived by Zhao et al. for crack channelling (i.e. growth in the x-direction) has the form:

\[
K_{i}^{TS} = A'Q_3\Delta T\sqrt{\pi t_c}
\]  

[3.51]

where:

\[
A' = \frac{A''}{\sqrt{\pi}}
\]  

[3.52]

The authors postulated further that a crack of depth \( H \) in a plate under thermal shock can propagate either through the thickness (plane strain cracking), i.e. in the z-direction of Fig. 3.6, or through the thickness and then laterally, which could lead to spalling. The relevant mode here, however, is crack channelling since surface propagation is considered. For crack channelling, the authors derived:

\[
A'' = \left(2.54 + \frac{5.4}{\beta}\right)^{-1}
\]  

[3.53]

Thus, through [3.52],:

\[
A' = \left(4.5 + \frac{9.57}{\beta}\right)^{-1}
\]  

[3.54]

In [3.54], the Biot modulus, \( \beta \), is given by:

\[
\beta = t_c \frac{h}{k}
\]  

[3.55]

The formula derived for \( K_{i}^{TS} \) using the analytical result of Zhao et al. (2000) is equivalent to the one defined previously for a pre-existing surface crack. Thus, we can write that:
In order to apply [3.56], estimates of the critical dimension, \( t_c \), and the parameter \( A' \) are needed. It has already been stated in Chapter 2 that:

\[
K_{TS} = AQ_3 \Delta T \sqrt{\pi \varepsilon} = A'Q_3 \Delta T \sqrt{\pi \varepsilon} \tag{3.56}
\]

In [3.57], \( a' \) and \( b' \) are coefficients of the equation that describes the stress reduction factor, \( A \), i.e.:

\[
t_c = \frac{b' k}{a' h} \tag{3.57}
\]

In [3.57], \( a' \) and \( b' \) are coefficients of the equation that describes the stress reduction factor, \( A \), i.e.:

\[
A = \left( a + \frac{b}{\beta} - ce^{\frac{d}{\beta}} \right)^{-1} \tag{3.58}
\]

For an infinite plate \( a = 1.5, b = 3.25, c = 0.5 \) and \( d = -16 \) whereas for an infinite rod \( a = 1.5, b = 4.67, c = 0.5, d = -51 \) (Manson 1966). Since the specimens in question cannot be approximated by either infinite plates or infinite rods, we write down the expression of the stress reduction factor for a sample with finite dimensions using as coefficients the averages of the values given above. Thus,

\[
A = \left[ 1.5 + \frac{3.96}{\beta} - 0.5e^{\frac{-33.5}{\beta}} \right]^{-1} \tag{3.59}
\]

A similar approach has been adopted by Maensiri and Roberts (2002), who used \( b = 4 \) and \( d = -30 \) for rectangular specimens of alumina nanocomposites. The thermal conductivity, \( k \), included in [3.57] is that in the through-thickness direction, given by:
if we are dealing with the transverse face of a UD CMC. For the other faces of the UD material:

\[ k = k_2 = \frac{k_f k_m}{(k_f V_m + k_m V_f)} \]  

[3.61]

Equation [3.57] can now be applied, using [3.60] and the coefficients of [3.59], to provide the critical dimension. However, as \( h \) varies widely (0 - 60 kW m\(^{-2}\) K\(^{-1}\)) there is large scatter in the values this formula provides. Instead, an alternative approach is adopted in this study. According to this, [3.59] can be written through [3.55] as:

\[ A = \left[ 1.5 + \frac{3.96k}{t_c h} - 0.5e^{-\frac{(-33.5)k}{t_c h}} \right]^{-1} \]  

[3.62]

It can be shown that the maximum value the stress reduction factor can take is \( A_{\text{max}} = 0.66 \) (see paragraph 2.2). At the same time, the maximum value reported for the coefficient of heat transfer during thermal shock (water quenching) is \( h_{\text{max}} = 60 \) kW m\(^{-2}\) K\(^{-1}\). Substituting these values in [3.62] we get:

\[ A_{\text{max}} = \left[ 1.5 + \frac{3.96k}{t_c h_{\text{max}}} - 0.5e^{-\frac{(-33.5)k}{t_c h_{\text{max}}}} \right]^{-1} \]  

[3.63]

The method utilised to obtain the critical dimension in this investigation is to ask the question ‘what value should \( t_c \) take for \( A \) to become equal to 0.66 at \( h = 60 \) kW m\(^{-2}\) K\(^{-1}\)?’, and perform a trial and error procedure using a spreadsheet.

It must be noted that this is only an approximate
procedure that depends on the input values. It would be more reassuring if the result is compared with an experimentally-determined value for the critical dimension.

If the value of \( t_c \) is known with confidence, [3.62] can then be used with to extract information about \( A' \). More specifically, using known values of the stress reduction factor, \( A \), and the critical dimension, \( t_c \), estimates for \( h \) can be obtained from [3.62]. Then, these estimates can be introduced in [3.54] to produce the respective values of \( A' \).

3.3.2.2.3. The Fracture Toughness of the Transverse Face of a UD CMC

Two approaches can be proposed for the determination of the fracture toughness of interest, \( K_{IC} \). First, it can be noted that the process of opening of a crack on the transverse face by a thermal shock-induced stress field that is maximum at the surface and reduces rapidly in the through-thickness direction bears resemblance to the variety of tests performed to determine \( R \)-curve behaviour in ceramic materials, e.g. the double cantilever beam test. When resistance to crack propagation of particulate-reinforced ceramics is determined using such methods, it is usually found that the initial value of fracture toughness is equal to that of the matrix (e.g. Trusty 1994). Thus, a first estimate for the initial resistance of the transverse face of the UD material to crack propagation through its thickness can be:

\[
K_{IC}^A = K_m
\]

[3.64]

where \( K_m \) is the fracture toughness of the matrix. Kahraman et al. (1997) who performed double torsion (DT) tests to determine transverse fracture toughness in UD Nicalon/CAS II CMCs determined values of the critical strain energy release rate, \( G_{IC} \), which were almost equal to the value for the matrix.
A second estimate for $K_{IC}$ can be postulated if the propagation of a crack along the surface of the transverse face is considered. From photomicrographs such as that of Fig. 2.7, it can be seen that an advancing surface crack is deflected continuously at successive fibre matrix interfaces. At $\Delta T_c$, these cracks are shallow, being limited to the surface of the material. Thus, a good estimate of the fracture toughness should be provided by the formula:

$$K_{IC}^h = K_m \sec^2 \left( \frac{\theta}{2} \right)$$

where $\theta$ is the angle through which the path of the advancing crack is deflected when it encounters the fibre-matrix interface. This formula was derived by Evans and Faber (1983) to describe the effect of the inclusion of spherical particulate reinforcements on the fracture toughness of a ceramic material.

In [3.64] and [3.65], the fracture toughness of the matrix, $K_m$, is related to the fracture energy, $G_m$, of the matrix through:

$$K_m = \sqrt{\frac{G_m E_m}{1 - \nu_m^2}}$$

3.3.2.2.4. Application of the Critical Condition for Cracking due to Thermal Shock

At $\Delta T = \Delta T_c$, i.e. at the onset of fracture, [3.56] becomes:

$$K_{IC}^{TS} = K_{IC} = A O_3 \Delta T_c \sqrt{\pi c} = A' O_3 \Delta T_c \sqrt{\pi c}$$
The critical temperature differential is then given by:

$$\Delta T_c = \frac{K_{IC}}{A'Q_3\sqrt{\pi \epsilon}}$$  \[3.68\]

3.3.2.2.5. Correlation with Experimental Results

Equation \[3.68\] is applied in this paragraph to the case of fracture of the end face of UD Nicalon/CAS, reported by Blissett \textit{et al.} (1997). For this material, apart from the properties included in Tables 3.1 and 3.2, we have: $k_m = 1.8$ W m$^{-1}$ K$^{-1}$ (Blissett 1995), $k_f = 2.97$ W m$^{-1}$ K$^{-1}$ (www.coiceramics.com) and $\nu_{23} \approx \nu_{21}$. The angle of deflection of an advancing crack can be assumed to be equal to an average value of $\theta = 45^\circ$.

The procedure outlined in paragraph 3.3.2.2.2 gives $t_c = 0.95$ mm. This compares very well with $t_c \approx 1.1$ mm that Becher and Warwick (1993) found experimentally for a glass ceramic material (Pyroceram 9606, Corning Glass Works, Corning, NY) with a slightly higher thermal conductivity. Thus, since it has already been determined that $\bar{A} = 0.55$ for this class of materials or $A = 0.53$ for UD Nicalon/CAS specifically, computation of $A'$ can proceed. The values obtained are $A' = 0.19$ and $A' = 0.18$.

Predictions for $\Delta T_c$ can now be made using \[3.68\] for the two estimates of $K_{IC}$ defined in paragraph 3.3.2.2.3. The results are presented in Table 3.7, where the formula is applied to monolithic CAS as well, also investigated by Blissett \textit{et al.} (1997). For this material the value of critical dimension obtained is $t_c = 0.78$ mm and, by assuming $A = 0.55$, it is found that $A' = 0.18$. 
Table 3.7. The results of the application of [3.68] to the end face of a UD Nicalon/CAS and monolithic CAS.

<table>
<thead>
<tr>
<th>ΔTc (°C)</th>
<th>A' = 0.18</th>
<th>A' = 0.19</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K_AKc</td>
<td>K_BKc</td>
</tr>
<tr>
<td>400°C</td>
<td>292</td>
<td>343</td>
</tr>
<tr>
<td>360°C</td>
<td>276</td>
<td>325</td>
</tr>
</tbody>
</table>

The results of Table 3.7 show that for monolithic CAS the discrepancy with the experimentally-determined value is 4%. By contrast, the error in the calculated values for the transverse face of the UD Nicalon/CAS is much greater: 14-19% for $K_{KC} = K_B^{B}$ (given by [3.58]), and 27-31% for $K_{KC} = K_A^{A}$ (given by [3.57]).

3.3.2.2.6. Discussion

The values obtained for the ΔTc of the end face of a UD Nicalon/CAS and for monolithic CAS show that the fracture mechanics approach describes in a satisfactory way the underlying phenomena. In addition, the accuracy of the prediction for CAS shows that accurate knowledge of the material properties included in [3.68] is a pre-requisite for successful application of this equation.

In the case of the end face of UD Nicalon/CAS doubts can be cast in the values entered for two crucial parameters: the fracture toughness, $K_{KC}$, and the transverse modulus, $E_2$. The doubts regarding the fracture toughness were partly overcome by utilising two different reasonable estimates. In the case of the transverse modulus, the value computed from [3.16] (110 GPa) has been shown to be an upper bound for the modulus of transverse plies in this material. More specifically, Pryce and Smith (1992) used their experimental findings and laminated plate theory.
to show that $E_2 = 85-110$ GPa for the transverse plies of a range of cross-ply Nicalon/CAS. The lowest value was found for the composite with the thickest, central transverse ply ($(0_2^\circ/90_4^\circ)_s$). Since this thick transverse ply can be considered to be as close as possible to the end face of the UD material, the lower bound value for transverse modulus value can be adopted in this investigation. Application of [3.61] for $E_2 = 85$ GPa gives $\Delta T_c = 360-378^\circ$C for $K_{jc} = K_{jc}^A$, and $\Delta T_c = 420-443^\circ$C for $K_{jc} = K_{jc}^B$, i.e. the discrepancy with the experimental value is significantly reduced to 5-10%.

There are other factors that may affect the prediction of $\Delta T_c$, e.g. the simplifying assumptions made in the calculation of $t_c$ and $A'$, errors in the various parameters obtained from the literature, the approximate nature of $\Delta T_c$ determination in water-quench tests etc. However, a main source for the observed discrepancy is perhaps likely to be that the formula of Zhao et al. (2000) (equation [3.53]) was derived for an infinite plate.

Equation [3.56] can also be used to give an indication of values of the length $2c$ of a surface crack that would have the same effect on the thermal shock resistance of the end face. More specifically:

$$c = t_c \left( \frac{A'}{A} \right)^2$$  \[3.69\]

The value obtained for UD Nicalon/CAS is of the order of equal to the half-thickness of each ply. In other words, the initial flaw is assumed to be of the order of the ply thickness, i.e. $2c_c \approx t_p$ (where $t_p$ is the thickness of each ply). Since ply thickness is equal to the total thickness of the sample ($t$) divided by the number of plies ($N$) from which it is comprised, the initial crack half-length is approximated by:
which is equivalent to \(3.68\). The result implies that only part of the surface is subjected to the maximum applied thermal shock stress, i.e. there is a variation in stress across the surface dimensions. The fact that the crack appears along the centreline of the face and runs parallel to the large top and bottom faces shows that such a variation possibly exists across the specimen thickness.

Although a satisfactory approach was demonstrated for the onset of thermal shock cracking, the morphology of thermal shock cracks on the end faces of UD CMCs cannot be explained by considering the applied thermal loading. A possible way to explain the observed phenomena is explored in the next section.

\[
c = \frac{t}{2N} \quad [3.70]
\]

and now \([3.50]\) gives:

\[
\Delta T_c = \frac{K_{IC}}{A_Q} \sqrt{\frac{2N}{\pi t}} \quad [3.71]
\]

which is equivalent to \([3.68]\). The result implies that only part of the surface is subjected to the maximum applied thermal shock stress, i.e. there is a variation in stress across the surface dimensions. The fact that the crack appears along the centreline of the face and runs parallel to the large top and bottom faces shows that such a variation possibly exists across the specimen thickness.

Although a satisfactory approach was demonstrated for the onset of thermal shock cracking, the morphology of thermal shock cracks on the end faces of UD CMCs cannot be explained by considering the applied thermal loading. A possible way to explain the observed phenomena is explored in the next section.
3.3.3. The Morphology of Cracks on End Faces of UD CMCs

3.3.3.1. Analysis

The longitudinal and transverse faces of the UD CMC of Fig. 3.5, held initially at high temperature, are assumed to come into sudden contact with a quenching medium of much lower temperature. The quench is such that thermal shock is inflicted concurrently on all material surfaces. For the thermal shock-induced stress to reach its maximum value at each material surface, the thickness just below each surface (i.e. in the depth dimension) must be equal or higher than twice the value of a critical dimension, $t_c$. This is illustrated in Fig. 3.7(a) and (b).

![Fig. 3.7. Schematics of a plate (thickness=2H) whose top and bottom faces are subjected to thermal shock for which (a) $H=t_c$, (b) $H>t_c$, and (c) $H<t_c$.](image)

It can be seen that in the case of Fig. 3.7(a) and (b) the critical dimensions of the top and bottom faces do not overlap, i.e. $H < t_c$. This allows the temperature gradient between the surface and its interior due to the shock to be established unhindered, as the gradient develops at the instance of the shock between the surface and a depth in the component equal to $t_c$ (irrespective of total thickness). This in turn means that the stress reduction factor, $A$, can reach its maximum value for the particular conditions and material, (since the value of $A$ depends on the temperature...
gradient) and, thus, the thermal shock-induced stress at each surface can reach its maximum value.

By contrast, in the case where $H < t_c$, i.e. in Fig. 3.7(c), it can be seen that the two critical dimensions overlap. This implies that the two separate temperature gradients interact and neither is fully established. This results in a reduction in $A$ and, consequently, the shock-induced stress at the surface will be only a fraction of its possible maximum value. This is the reason behind the well-established fact that components thinner than a critical thickness exhibit higher thermal shock resistance than their thicker counterparts (Wang and Singh 1994).

Now, returning to the UD CMC of Fig. 3.5, it can be stated that the critical dimension of the transverse face ($t_{c3}$) will be different from that of the other, longitudinal faces ($t_{ci}$). By taking into account [3.68] and the method outlined for the calculation of the critical dimension, the difference arises because the thermal conductivity, $k$, for the transverse face, given by [3.61], differs from $k$ for the longitudinal faces, given by [3.62]. If, for illustrative purposes, the width of the specimen is made much bigger than its thickness and the critical dimensions of each face are superimposed, we get the schematic of Fig. 3.8.

Again, it is assumed that all surfaces are subjected to thermal shock at the same time. The critical dimension of the transverse face ($t_{c3}$) is along the 1-direction, those of the side longitudinal faces ($t_{ci}$) along the 3-direction, and those of the top and bottom longitudinal faces ($t_{c1}$) along the 2-direction.
Close observation reveals that, apart from the volume at the centre that is drawn in red, the respective critical dimensions of the adjoining faces overlap in the rest of the sample up to a length equal to $t_{c3}$ in the 1-direction. The situation corresponds to the condition of Fig. 3.7(c) but with critical dimensions of different length. So, in these areas the respective temperature gradients interact, which means that A at the corresponding surfaces, and thus the shock-induced stress, do not reach their respective maximum values.

By contrast, in the volume bound by the red lines no interaction of gradients takes place; the sole temperature gradient established is the one corresponding to the transverse face. Thus, the narrow area on the transverse face will experience the maximum possible severity of the applied thermal shock.
3.3.3.2. Discussion

The above analysis provides the background for a satisfactory explanation of the cracking phenomena reported on the transverse face of UD Nicalon/CAS samples of thickness 2.2 mm and width 10 mm (Blissett et al. 1997). As reported in paragraph 3.3.2.2.4 \( t_{c3} = 0.95 \) mm, while for the longitudinal faces \( t_{c1} = 1.05 \) mm. If the values for \( t_{c1} \) are superimposed across the thickness of an image of the transverse face, it becomes obvious why the crack appears at that location. The image can be seen in Fig. 3.9.

The central area bounded by the two dotted lines corresponds to the area where the stress reduction factor reaches its maximum value. This area will always experience the highest stresses irrespective of applied temperature differential. This means that although the areas adjacent to the bound one will start to crack as \( \Delta T \) increases, the extent of damage in the central area will always be larger. This is evident in the image of Fig. 3.9 taken from a sample quenched at \( \Delta T = 800^\circ C \): although cracks are evident at various places on the surface, the major, deep crack still runs in the highlighted central area.

The above concept can also explain the direction of crack propagation. More specifically, although surface flaws are expected to be uniformly distributed on the material surface, the crack originates at one inside the bound area and simply follows the path perpendicular to which the maximum stress is applied. In addition, comparison of the thickness of the highlighted area (= 0.1 mm) with the flaw length required for crack initiation (equal to the individual ply thickness or 0.183 mm) shows that no cracking can commence at right angles to the horizontal.
Fig. 3.9. The critical dimensions of the top and bottom faces are seen here superimposed on a photo-micrograph of an end face of UD CMC. The area bounded by the dotted lines is where maximum shock-induced stress occurs.
3.4. CONCLUDING REMARKS

The cracking phenomena due to thermal shock on the surface of a UD CMC were explained and analysed in this chapter.

Models that predict the critical quenching temperature differential for cracking onset with satisfactory accuracy were developed. In addition, a theoretical framework that explains the morphology of the cracking phenomena was established.

It is of great interest to extend and apply the theories and models developed in this chapter to more complex CMC laminates. Although some data on the thermal shock behaviour of 2-D CMCs already exists (see Chapter 2), detailed experimental observations need to be made on a wide range of CMC laminates. Such an investigation was performed as part of the research reported in this thesis. The results are presented in the following two chapters.
Chapter 4:

Materials and Experimental Techniques
4.1. MATERIALS

The materials used in this study were manufactured by Corning Inc. and supplied courtesy of Rolls Royce plc. They comprised a calcium aluminosilicate (CAS) glass-ceramic matrix reinforced with silicon carbide (Nicalon*) fibres arranged in three different ply architectures: $(0^\circ/90^\circ)_s$, $(0^\circ/90^\circ)_{3s}$, and plain-weave (PW) woven. The first and the second materials were manufactured by hot pressing 4 and 12 plies, respectively, of UD Nicalon fibres impregnated with the matrix material to give CMCs of thickness ~0.7 mm and ~2.2 mm, respectively, and fibre volume fraction of 0.34. The PW woven material was manufactured by hot pressing 12 plies of woven Nicalon-fibre cloth impregnated with the matrix material to give a plate with a thickness of 2.2 mm and low porosity. The fibre volume fraction of this material has been determined by Ironside (1996) to be 0.35.

Electron probe microanalysis of similar unidirectional material coming from the same source (Blissett 1995) showed that the matrix material was a glass ceramic from the ternary CaO-Al$_2$O$_3$-SiO$_2$ system, which also included small particles that were either aluminium- or zirconium-rich. The fibre and the matrix material had elastic moduli of 190 and 90 GPa respectively, while the respective values for thermal expansion coefficient were $\alpha_f = 3.3 \times 10^{-6}$ °C$^{-1}$ and $\alpha_m = 4.6 \times 10^{-6}$ °C$^{-1}$.

* Nicalon is a trademark of Nippon Carbon Co.

** In more detail, the material was identified as stoichiometric CaO-Al$_2$O$_3$-2SiO$_2$, the anorthite phase of the system.
4.2. EXPERIMENTAL PROCEDURE

4.2.1. Introduction

The experimental procedure comprised a series of thermal shock tests on polished material samples, which were subsequently used either for damage characterisation or for mechanical testing. In the following paragraphs, details are provided of the sample preparation routine, the thermal shock and mechanical testing procedures, as well as of the means and method used for damage characterisation.

4.2.2. Sample Preparation

Test samples for damage observation and mechanical testing were cut to size from the supplied material using a high-speed Diamant Boart, sliding-bed cutting machine fitted with a water-cooled cutting wheel coated with 200 μm diamond particles. The dimensions of the specimens used for damage observation were 6 mm x 6 mm x 0.7 mm for the (0°/90°)ₕ laminate, and 6 mm x 6 mm x 2.2 mm for the (0°/90°)₃ₜ laminate. For the PW woven laminate, samples with two different sets of dimensions were prepared: 12 mm x 6 mm x 2.2 mm, and 8 mm x 4 mm x 2.2 mm. The PW woven Nicalon/CAS specimens used for mechanical testing were 50 mm x 5 mm x 2.2 mm.

Parallel longitudinal faces of samples of all CMCs were prepared for damage observation. These faces were 6 mm x 0.7 mm for the (0°/90°)ₕ laminate, 6 mm x 2.2 mm for the (0°/90°)₃ₜ, and 12 mm x 2.2 mm, 6 mm x 2.2 mm, 8 mm x 2.2 mm, and 4 mm x 2.2 mm for the PW woven material. By preparing adjacent longitudinal faces in the cross-ply CMCs, damage due to thermal shock could also be observed for the (90°/0°)ₕ and (90°/0°)₃ₜ configurations.
Preparation of the above surfaces was performed using a Struers Planopol-2 machine fitted with a Pedemax-2 rotating head. Suitable specimen holders were designed and were manufactured by the School of Engineering Workshop. Three of them were used each time to ensure the balance of the rotating head. Each specimen holder could accommodate up to three specimens depending on the surface that had to be polished. In this case, one specimen was clamped in each holder for each polishing cycle.

The preparation routine comprised eight stages – six grinding and two polishing. Silicon carbide paper with grain size 320–4000 grit was used as the grinding media. Each stage lasted 15-60 seconds with ultrasonic cleaning of the specimens performed in between. The polishing stages were carried out using 3 µm and 1 µm diamond wheels and spray, and resulted in a 1µm finish. The full preparation procedure is summarised in Tables 4.1. (a)-(b).

The same procedure was employed to prepare parallel longitudinal faces (50 mm x 2.2 mm) on the test samples of PW woven Nicalon/CAS used for mechanical testing.

Table 4.1: The (a) grinding, and (b) polishing procedure followed for surface preparation.

<table>
<thead>
<tr>
<th>Grind</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Media</td>
<td>SiC</td>
<td>SiC</td>
<td>SiC</td>
<td>SiC</td>
<td>SiC</td>
<td>SiC</td>
</tr>
<tr>
<td>Grit</td>
<td>320</td>
<td>500</td>
<td>800</td>
<td>1200</td>
<td>2400</td>
<td>4000</td>
</tr>
<tr>
<td>Lubricant</td>
<td>Water</td>
<td>Water</td>
<td>Water</td>
<td>Water</td>
<td>Water</td>
<td>Water</td>
</tr>
<tr>
<td>Speed (rpm)</td>
<td>300</td>
<td>300</td>
<td>300</td>
<td>300</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>Pressure (N)</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Time (seconds)</td>
<td>Till 15</td>
<td>15</td>
<td>30</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
</tbody>
</table>
4.2.3. Thermal Shock Simulation Procedure

The quench test, as described in paragraph 2.3.2, was used to produce the thermal shock condition. The quenching medium was a large quantity (>10 litres) of room-temperature water (20-22°C); the temperature of the water was measured before and after each thermal shock test using a thermocouple probe. All thermal treatments of the specimens were carried out using two Elite Thermal Systems Limited electric muffle furnaces (Models BAF 7/15 and BSF 12/22) fitted with Eurotherm Model 2416 electronic temperature controllers.

For each thermal shock test, the test specimen was placed on an alumina crucible and was inserted into the furnace, which was already set at a pre-determined temperature. The crucible was placed at the centre of the furnace. Each test specimen was kept in the furnace for 15-20 minutes to ensure uniform temperature distribution. Specimens tested at the highest temperature differentials investigated ($\Delta T=700-800{^\circ}C$) were heated for shorter periods of time (7-10 mins), as it was found that the longer soak resulted in the formation of a thin glassy layer over the material surfaces, probably a by-product of oxidation processes, which obscured crack observation. The furnace was subsequently opened, the crucible was removed, and the test specimen was rapidly dropped into the water bath. The time taken for the specimen to reach the
water bath after its removal from the centre of the furnace was about 1-2 seconds. It is believed that this did not affect the specimen temperature considerably, so the specimen could be considered to have its inside-the-furnace temperature when it impacted the water bath. The water bath temperature remained constant after quenching. The test specimen was subsequently removed from the water bath and was allowed to dry before microscopic examination or mechanical testing could proceed. It should also be noted that as throughout this study the quenching medium temperature was maintained constant at 20-22°C, the target furnace temperature was in all cases 20-22°C higher than the values given for temperature differentials. Temperature differentials investigated ranged between \( \Delta T=100-800°C \), while 2-4 specimens were tested at each \( \Delta T \).

4.2.4. Mechanical Testing

4.2.4.1. Tensile Testing

Aluminium end tabs were bonded to the ends of test coupons prior to testing using a Permabond F246 rubber toughened acrylic adhesive, leaving a final gauge length of 30 mm. The applied strain was measured using a Vishay Micro-Measurements CEA-06-250UN-350 precision strain gauge, which was attached to the centre of one of the large faces (50 mm x 5 mm) of the test coupon using a cyano-acrylate adhesive (Fig. 4.1). The output of the strain gauge was recorded using a Solartron SI 3535D data logger.
Tensile tests were carried out using an Instron model 1195 tensile testing machine with a 100 kN load cell at a cross-head speed of 0.5 mm/min. All samples were loaded continuously to failure. The load and strain outputs were stored as data files in a PC running Windows™ software, which was connected to the Instron machine and the digital strain indicator.

Processing of the recorded data was performed using Microsoft Excel™ software. Mechanical properties were determined from the generated graphs following ASTM C1275-95 and BS EN 658-1:1998 where appropriate.

4.2.4.2. Flexure Testing

Flexure testing was performed using a three-point bend test rig that had been designed and built in-house. The support span was 32 mm. The upper platen, that contained the two supports on which the test specimen was placed, was firmly pinned to an Instron 1175 Testing Machine. The lower platen, which had the central loading point fixed on it, was attached to the cross-head beam of the testing machine (Fig. 4.2). A 10 kN load-cell was employed. The output of the load cell and the cross-head displacement were monitored in real-time and recorded by a PC running
Windows™ software. The flexural testing procedure and the analysis of the results were performed following the ASTM C 1341-97 Standard Test Method exactly.

Fig. 4.2. Photograph of the bend test rig.

4.2.5. Damage Observation

Microscopic examination of the thermally-shocked specimens was carried out using a reflected light microscope (Zeiss Axiophot). The microscope was connected to a PC with suitable software so that the images could be seen on the computer screen in real time and could also be processed and subsequently stored as computer files. Every thermally-shocked face was examined using the microscope and was photographed section by section. Then, the stored images were assembled using the Panavue Image Assembler™ software. The resulting image
showed the whole area of the surface on which the cracking pattern was subsequently imposed manually after careful observation of the real surface under the microscope. More detailed observation of cracking patterns was performed using a scanning electron microscope (Hitachi environmental S-3200N), which was operated at high potentials (>15 kV) to obtain sufficient resolution.
Chapter 5:

Thermal Shock Damage on Cross-Ply CMCs
5.1. INTRODUCTION

The effect of a thermal shock on the microstructure of several cross-ply Nicalon/CAS laminates of various configurations is presented in this chapter. The configurations examined include simple \((0^\circ/90^\circ)_s\) and \((90^\circ/0^\circ)_s\) laminates, as well as multi-layer \((0^\circ/90^\circ)_3s\) and \((90^\circ/0^\circ)_3s\) laminates.

The results for each laminate are presented as follows: first, the critical quenching temperature difference for the onset of damage due to thermal shock is given. Identification and description of the main damage modes, as well as their location on the polished surface of the material, follows. Then, the way in which the various damage modes accumulate on the material surface when successively higher temperature differentials are applied is described in detail. This is accompanied by quantification of the extent of damage at each temperature differential investigated.

After the above procedure has been completed for all laminates, qualitative and quantitative comparisons are made between the observed thermal shock characteristics of the various laminates. This also facilitates the identification of general trends in the way thermal shock loading affects this class of CMCs.

It has to be noted that some aspects of the thermal shock resistance of the \((0^\circ/90^\circ)_3s\) (as well as some of those of a \((0^\circ_2/90_4^\circ)_3\)) laminate have already been assessed in the work of Blissett (1995) and have been presented in Blissett et al. (1998) (see Chapter 2). The current investigation aims to verify, reinforce and extend significantly the work of Blissett (1995) on the \((0^\circ/90^\circ)_3s\) laminate as well as to complement it with detailed data on configurations not covered in the abovementioned investigation.
5.2. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED SIMPLE CROSS-PLY NICALON/CAS LAMINATES

5.2.1. The (0°/90°), Laminate

5.2.1.1. Critical Quenching Temperature Difference ($\Delta T_c$)

No damage was observed on the surfaces of material samples after quenching through temperature differentials lower than 450°C, i.e. for $\Delta T < 450^\circ$C. Some of the samples quenched through $\Delta T=450^\circ$C, the majority of the samples quenched through $\Delta T=480^\circ$C and almost all of the samples tested through $\Delta T=500^\circ$C showed evidence of matrix damage in the form of shallow, hair-like cracks. Thus, it was decided that the critical quenching temperature differential for this laminate lies in the range 450-500°C, i.e. $\Delta T_c=450-500^\circ$C. The actual value of $\Delta T_c$ seems to vary depending on experimental details, such as the angle of impact with the quenching medium, and the extent of pre-existing damage on the surfaces of the material. Generally, surfaces that exhibit at least some open porosity crack at 450°C or at temperature differentials close to this value.

5.2.1.2. Damage Modes due to Thermal Shock

5.2.1.2.1. Nomenclature

The description of thermal shock damage on this laminate is given with reference to the nomenclature of Fig. 5.1. As it can be seen, the central, thick transverse (90°) ply is designated as T1 (Transverse 1) while the adjacent longitudinal (0°) plies are designated as L1 (Longitudinal 1).
5.2.1.2.2. General

One fracture mode due to thermal shock was identified on the surfaces of material samples shocked through $\Delta T \geq 450^\circ C$, as 'matrix cracking'. If the direction of matrix cracks is taken into account, matrix cracks can be further divided into those that run parallel and those that run perpendicular to the x-axis, i.e. to the horizontal. Hence, two types of cracking phenomena are described in detail in the following paragraphs:

a. Horizontal Matrix Cracks (HMCs)

b. Perpendicular Matrix Cracks (PMCs)

It should be noted that no fibre breaks/failures could be observed even at the highest temperature differentials investigated ($\Delta T = 700-800^\circ C$).
5.2.1.2.3. **Horizontal Matrix Cracks**

HMCs were the first form of damage seen after quenching through $\Delta T=450-500^\circ C$ (Fig. 5.2). They were located exclusively in the thick, central 90° ply (T1) and propagated approximately parallel to the horizontal (x-axis). Each one was deflected at the successive fibre-matrix interfaces it encountered on its path. For this reason Graham et al. (2003), who observed similar crack patterns on the transverse faces of UD Nicalon/LAS II after thermal shock, referred to them as ‘thermal debond’ cracks. HMCs also seemed to appear randomly on the ply surface, although most of them could be seen towards the centreline (C-C’) of the ply.

![Fig. 5.2. Photomicrograph of shallow, hair-like HMC on T1 at $\Delta T=450^\circ C$.](image)

5.2.1.2.4. **Perpendicular Matrix Cracks**

PMCs were detected on the surfaces of thermally-shocked specimens of this laminate after quenching through $\Delta T=500^\circ C$, exclusively in the two 0° plies (L1) adjacent to the thick, central 90° ply. These cracks ran across the ply thickness, leaving the fibres on their path unaffected (Fig. 5.3).
5.2.1.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT)

5.2.1.3.1. General

The effect of increasing applied quenching temperature difference on samples of this laminate can be seen in the sequence of Fig. 5.4(a)-(e), where the crack patterns observed under the microscope have been superimposed onto RLM images of the respective material surfaces. In the following paragraphs, both qualitative and quantitative descriptions of the effect of increasing ΔT on each damage mode referred to previously is given.

5.2.1.3.2. Description of Thermal Shock Damage Development

At ΔT=450-500°C, only a small number of HMCs were observed in T1. They did not penetrate deep in the matrix and were short in length. The small number (1-2) of PMCs that were seen in L1 at ΔT=500°C exhibited similar characteristics. In addition, they did not span the entire 0° ply thickness but arrested at fibre-matrix interfaces inside the ply (Fig. 5.3).
At \( \Delta T=600^\circ C \) a number of short, random HMCs were again observed in T1, while some PMCs in L1 could be seen to extend and bridge the whole 0° ply thickness. Some HMCs seemed to connect and form 1-2 longer cracks in T1 at \( \Delta T=700^\circ C \) (Fig. 5.5). At the same temperature differential, some PMCs not only bridged the 0° ply thickness but also extended into the adjacent 90° ply (Fig. 5.6).
Almost all PMCs, which had increased in number significantly, could be seen traversing the thickness of L1 at ΔT=800°C, while 1-2 longer HMCs ran along the T1 length.

The application of higher temperature differentials did not result in significant morphological changes in either HMCs or PMCs. Both damage mechanisms remained surface features of small depth. It must also be noted that at all temperature differentials PMCs were evenly distributed between the two 0° plies termed L1.

5.2.1.3.3. Quantification of Thermal Shock Damage Development

In Figs. 5.7 and 5.8 the increase in crack density for both PMCs and HMCs with increasing applied temperature differential is shown.
Fig. 5.7. Crack density as a function of $\Delta T$ for PMCs for $(0^\circ/90^\circ)_s$ Nicalon/CAS laminate.

Fig. 5.8. Crack density as a function of $\Delta T$ for HMCs for $(0^\circ/90^\circ)_s$ Nicalon/CAS laminate.
Crack densities for HMCs are given in crack length per unit area (mm/mm$^2$ or mm$^{-1}$) while PMC crack densities are given as cracks per millimetre (cracks/mm), which is the normal method for describing the density of this fracture mode in UD and cross-ply composites.

The failure of small, individual HMCs to connect at higher temperature differentials and form much longer cracks is evident in the graph of Fig. 5.8. As can be seen, there is only a moderate increase in crack density with increasing severity of the applied shock. By contrast, the density of PMCs increases at a higher rate. This is more evident in the graph of Figure 5.9, where both types of cracking are compared in terms of crack length/unit area. Although PMCs appear at higher $\Delta T$, they constitute the larger percentage of the total damage accumulated at the higher temperature differential investigated.

It must be noted that although an attempt was made to deduce different trends in crack density increase between successive quenching temperature differentials, the observed scatter in the measured crack density values did not allow for firm conclusions to be reached.

![Graph showing crack density comparison](image)

**Fig. 5.9.** Comparison between crack densities of PMCs, HMCs and their total at each $\Delta T$. Relevant trends for each damage mode are also shown.
5.2.2. The (90°/0°), Laminate

5.2.2.1. Critical Quenching Temperature Difference ($\Delta T_c$)

Damage due to thermal shock was observed under the optical microscope after quenching through temperature differentials higher than 500°C. Thus, the critical quenching temperature differential for this laminate was determined to be $\Delta T_c=500^\circ$C.

5.2.2.2. Damage Modes due to Thermal Shock

5.2.2.2.1. Nomenclature

The description of thermal shock damage on this laminate is given with reference to the nomenclature of Fig. 5.10. The central, thick 0° ply is designated as L1 while the adjacent 90° plies are designated as T1.

![Fig. 5.10. Schematic diagram defining the nomenclature used to describe damage due to thermal shock on a (90°/0°) laminate and (b) micrograph of the surface of a (90°/0°) laminate (its thickness is about 0.7 mm).](image-url)
5.2.2.2. General

The main mode of damage due to thermal shock on this laminate was matrix cracking. Horizontal matrix cracks (HMCs) developed parallel to the x-axis while perpendicular matrix cracks (PMCs) could be seen running at right angles to the x-axis. No damage to the fibres could be detected even at the highest temperature differentials investigated.

5.2.2.2.3. Perpendicular Matrix Cracks

PMCs were the first type of damage due to thermal shock detected on the surfaces of this laminate. They appeared exclusively in the central, thick L1 ply at $\Delta T=500^\circ C$. They could be seen leaving the longitudinal fibres on their path unaffected, being arrested at fibre-matrix interfaces inside the 0° ply or at the interface between 0° and 90° plies. Such cracks can be seen in Fig. 5.11.

5.2.2.2.4. Horizontal Matrix Cracks

HMCs or ‘thermal debond’ cracks appeared in the T1 plies of this laminate after quenching through $\Delta T=550^\circ C$. Only a few of these cracks could be observed and they were deflected at successive fibre-matrix interfaces. A major, long HMC could not be identified. The morphology of such cracks is evident in the photomicrograph of Fig. 5.12.

![Fig. 5.11. PMC in L1 at $\Delta T=500^\circ C$.](image1)

![Fig. 5.12. HMC in T1 at $\Delta T=550^\circ C$.](image2)
5.2.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential (ΔT)

5.2.2.3.1. General

Changes to the number and the morphology of both types of matrix cracking for this laminate under increasing applied ΔT are shown in the sequence of Fig. 5.13(a)-(e). They are described in the following two paragraphs.

![Photomicrographs of quenched surfaces of (90/0) Nicalon/CAS with superimposed crack pattern observed at each ΔT. The faces shown are 6 mm x 0.7 mm.](image)
5.2.2.3.2. Description of Thermal Shock Damage Development

PMCs originating at $\Delta T=500^\circ C$ were few in number and did not penetrate deep inside the matrix material. In addition, they did not span the full thickness of the L1 plies. Most of the PMCs could be seen traversing the thickness of the central L1 ply at $\Delta T=550^\circ C$, while all of them bridged it at $\Delta T=600^\circ C$ before being arrested at the interface between 0° and 90° plies (Fig. 5.14). At the highest temperature differentials ($\Delta T=700-800^\circ C$), some PMCs could be seen propagating a short distance inside the adjacent 90° plies.

![Fig. 5.14. PMC bridging L1 thickness (~0.35 mm) at $\Delta T=600^\circ C$.](image)

A small number of short HMCs were almost evenly distributed between the T1 plies at all temperature differentials investigated. The increase in $\Delta T$ was accompanied in most cases with a moderate increase in their length.

The depth and opening of PMCs and HMCs was not altered by the application of higher temperature differentials. They both remained surface features throughout the temperature range investigated.
5.2.2.3.3. Quantification of Thermal Shock Damage Development

The change in crack density of both PMCs and HMCs with increasing shock severity is shown in Fig. 5.15 and 5.16 in terms of cracks per unit length and crack length per unit area, respectively. The number of PMCs increases significantly for higher ΔTs while the crack density of HMCs shows only a moderate increase. The large difference in the rate of increase between the two types of matrix cracking is evident in the graph of Fig. 5.17. It can be seen that at all ΔTs, about 2/3 of the total thermal shock damage is due to the formation and extension of PMCs.

![Graph showing crack density as a function of ΔT for PMCs for (90°/0°) Nicalon/CAS laminate.](image)

Fig. 5.15. Crack density as a function of ΔT for PMCs for (90°/0°), Nicalon/CAS laminate.
Fig. 5.16. Crack density as a function of ΔT for HMCs for (90°/0°), Nicalon/CAS laminate.

Fig. 5.17. Comparison between crack densities of PMCs, HMCs and their total at each ΔT. Relevant trends for each damage mode are also shown.
5.2.3. Summary

Damage due to thermal shock in simple cross-ply laminates was described and quantified in detail in this section. The main damage mechanism was found to be matrix cracking. Matrix cracks advanced parallel to the horizontal in transverse plies and at right angles to the horizontal in longitudinal plies. They were deflected at fibre-matrix interfaces at every quenching temperature investigated, so no fibre failures were observed.

The $(90^\circ/0^\circ)_s$ laminate exhibited better resistance to thermal ‘cold’ shock than the $(0^\circ/90^\circ)_s$ laminate. However, damage in both laminates originated in the thick, central ply and then, at higher temperature differentials, extended to adjacent plies.

Matrix cracks in both laminates were found to remain shallow, surface features irrespective of the severity of thermal shock loading. However, damage in the form of PMCs was more extensive than damage in the form of HMCs in both laminates, especially at higher quenching temperature differences.
5.3. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED
MULTI-LAYER CROSS-PLY NICALON/CAS LAMINATES

5.3.1. The (0°/90°)₃₉ Laminate

5.3.1.1. Critical Quenching Temperature Difference (ΔTₖ)

Polished surfaces of this system did not exhibit any damage after quenching through temperature
differentials lower than 350°C, i.e. for ΔT<350°C. After quenching through ΔT≥350°C, surface
cracking could be seen under the optical microscope. Thus, the ‘critical quenching temperature
difference’ for this Nicalon/CAS laminate is ΔTₖ=350°C.

5.3.1.2. Damage Modes due to Thermal Shock

5.3.1.2.1. Nomenclature

The description of thermal shock damage on this laminate is given with reference to the
nomenclature of Fig. 5.18.

5.3.1.2.2. General

The form of thermal shock damage observed on this laminate was matrix cracking. This can be
further divided into PMCs and HMCs depending on the orientation of matrix cracks relative to
the horizontal x-axis. Longitudinal fibres remained unaffected even at high shocks.

5.3.1.2.3. Horizontal Matrix Cracks

HMCs were the first form of damage observed after quenching through ΔTₖ=350°C. They were
located exclusively in 90° plies. Depending on the specimen under observation, these cracks
emanated either from flaws, such as pores, and were contained inside the ply or originated from
the edges of the ply and ran towards its centre (Fig. 5.19(a)-(b)). They were continuously deflected at successive fibre-matrix interfaces.

Fig. 5.18. (a) Schematic diagram defining the nomenclature used to describe damage due to thermal shock on a (0°/90°)₃s laminate and (b) micrograph of the surface of a (0°/90°)₃s laminate (its thickness is 2.2 mm).

5.3.1.2.4. Perpendicular Matrix Cracks

PMCs were detected on the surfaces of thermally-shocked specimens after quenching through ΔT=400°C, exclusively in 0° plies. These cracks ran perpendicular to the horizontal (i.e. to the longitudinal fibres of the 0° plies), leaving the fibres on their path unaffected, and arrested either at a fibre-matrix interface inside the ply or at the interfaces between 0° and 90° plies.
5.3.1.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential ($\Delta T$)

5.3.1.3.1. General

The evolution of cracking at temperature differentials between $\Delta T=350-800^\circ C$ is shown in the sequence of RLM images of Fig. 5.20 (a)-(f).
(a) $\Delta T = 350^\circ C$

(b) $\Delta T = 400^\circ C$

(c) $\Delta T = 500^\circ C$
Fig. 5.20(a)-(f) Photomicrographs of quenched surfaces of $(0^\circ/90^\circ)_3$ Nicalon/CAS with superimposed crack pattern observed at each $\Delta T$. The faces shown are 6 mm x 2.2 mm.
5.3.1.3.2. Description of Thermal Shock Damage Development

At $\Delta T = 350-400^\circ C$ only random HMCs could be generally seen in the thick, central transverse ply (T1). However, a much longer crack was also evident on some specimens quenched at this temperature differential. These cracks were limited to the surface of the material.

At $\Delta T = 400^\circ C$ PMCs appeared at the 0° plies (L1) adjacent to T1 while longer, random HMCs could again be seen in T1. Damage, in the form of PMCs and HMCs, appeared in the 0° plies designated as L2 and the 90° plies designated as T2, respectively, at $\Delta T = 450^\circ C$. At this temperature differential, a long HMC propagated along the central T1 ply (Fig. 5.21) while in the T2 plies only individual HMCs appeared.

![Fig. 5.21. Long HMC in T1 at $\Delta T = 450^\circ C$.](image)

Similar patterns were observed at $\Delta T = 500^\circ C$. However, almost all PMCs now spanned the thickness of the L1, L2 plies while some started to extend into the adjacent 90° plies. In addition, some HMCs in the T2 plies connect to form longer cracks.

At $\Delta T = 600^\circ C$ all plies of this system sustained some form of thermal shock damage; T1 contained a long, deep HMC, T2 exhibited shorter and shallower HMCs, while individual,
random HMCs could be seen in T3. In addition, all longitudinal plies (L1, L2, L3) contained PMCs, which seemed to decrease in number on going from the centreline (C-C’) towards the top or bottom edges of the surface.

The application of even higher ΔTs (=700-800°C) lead to an increase in the number of PMCs in the longitudinal plies, although it again looked as if the closer the ply was located to the centreline, the higher the crack density. In addition, some PMCs (especially in L1) could be seen to extend into the adjacent transverse plies (T1 and T2). HMCs followed a more random pattern. There was always a long, deep crack that travelled almost the full length of the ply in either T1 or T2. The rest of these plies contained shorter and shallower cracks while the cracks located in T3, although continuously increasing in number and length, failed to connect into longer HMCs even at the highest ΔT.

In general, the application of higher ΔTs did not affect the morphology of PMCs. By contrast, HMCs located in transverse plies at or close to the centreline of the face became deeper and their opening, as well as their length, increased significantly at the highest temperature differentials investigated (Fig. 5.22).

It has to be noted that PMCs were evenly distributed between the longitudinal plies of the same designation (i.e. L1, L2 or L3). This was not exactly the case for HMCs as these were distributed in a more random fashion, especially at the higher temperature differentials, between the pairs of transverse plies (i.e. T1, T2 or T3) depending on the specimen under investigation.
5.3.1.3.3. Quantification of Thermal Shock Damage Development

The increase in PMC density with increasing shock severity for each set of longitudinal plies (L1, L2, L3) is shown in the graph of Fig. 5.23. It is evident that crack density (CD) is always higher for the plies located towards the centre of the sample surface, i.e. \( CD_{L1} > CD_{L2} > CD_{L3} \) at each \( \Delta T \) investigated. The rates of increase of cracking in each set of plies are comparable.

Fig. 5.24 shows the change in HMC density with increasing temperature differential. A significant increase in cracking can be observed, especially at the higher thermal shocks. In addition, the scatter in experimental data is larger at the higher temperature differentials, which reflects the randomness in the appearance and point of origin of long cracks in T1 and/or T2.

Comparison between PMCs and HMCs in Fig. 5.25 reveals that the rate of increase in density of PMCs is much higher than that of HMCs and, at high temperature differentials, the PMCs are the
major part of the total crack density. However, this graph fails to capture the big morphological differences between the two types of matrix cracking at $\Delta T \geq 600^\circ C$.

Fig. 5.23. Crack density as a function of $\Delta T$ for PMCs for each set of longitudinal plies of $(0^\circ/90^\circ)_3s$ Nicalon/CAS laminate. Note that $CD_{L1} > CD_{L2} > CD_{L3}$ at all $\Delta Ts$.

Fig. 5.24. Crack density as a function of $\Delta T$ for HMCs on $(0^\circ/90^\circ)_3s$ Nicalon/CAS laminate.
Fig. 5.25. Comparison between crack densities of PMCs, HMCs and their total at each $\Delta T$. Relevant trends for each damage mode are also shown.
5.3.2. The (90°/0°)₃s Laminate

5.3.2.1. Critical Quenching Temperature Difference (\(\Delta T_c\))

Initial damage due to thermal shock was detected on the surfaces of this laminate after quenching through temperature differentials higher than 400°C. Thus, \(\Delta T_c = 400°C\) for this laminate.

5.3.2.2. Damage Modes due to Thermal Shock

5.3.2.2.1. Nomenclature

The description of thermal shock damage on this laminate is given with reference to the nomenclature of Fig. 5.26.

---

**Fig. 5.26.** (a) Schematic diagram defining the nomenclature used to describe damage due to thermal shock on a (90°/0°)₃s laminate and (b) micrograph of the surface of a (90°/0°)₃s laminate (its thickness is 2.2 mm).
5.3.2.2.2. General

Thermal shock damage on this laminate for the range of temperature differentials investigated ($\Delta T=0$-$800^\circ$C) was in the form of perpendicular and horizontal matrix cracks (PMCs and HMCs).

5.3.2.2.3. Horizontal Matrix Cracks

HMCs as a result of thermal shock were first visible on this laminate after quenching through $\Delta T_c=400^\circ$C. They could be seen to propagate along the surface of the 90° plies. Although they generally ran horizontally, successive fibre-matrix interfaces deflected them continuously.

5.3.2.2.4. Perpendicular Matrix Cracks

PMCs were also detected after quenching through the critical temperature differential, i.e. at $\Delta T_c=400^\circ$C. They appeared only in 0° plies. Their advance was at right angles to the longitudinal fibres, which remained unaffected since PMCs were deflected at fibre-matrix interfaces.

5.3.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential ($\Delta T$)

5.3.2.3.1. General

The evolution of cracking at temperature differentials between $\Delta T=400$-$800^\circ$C is shown in the sequence of RLM images of Fig. 5.27 (a)-(e).
5.3.2.3.2. Description of Thermal Shock Damage Development

At the onset of fracture ($\Delta T_c=400^\circ C$), damage was visible only in the central plies of this laminate. PMCs in L1 did not bridge the ply thickness while HMCs in T1 were associated mainly with open pores.

The application of temperature differentials up to $\Delta T=500^\circ C$ did not change the morphology of both types of matrix cracks: they remained shallow surface features. However, PMCs were also
visible in L2 and L3 and the presence of HMCs extended to T2. At ΔT=500°C, HMCs of significant length could be seen originating either inside T1 and T2 or from the ply edges running towards the centre of the specimen.

The length and depth of HMCs in T1 became much larger at ΔT=600°C (Fig. 5.28). At this temperature differential, damage was detected in every ply of the laminate. PMCs could be seen bridging the thicknesses of their respective plies and some extended into adjacent transverse plies. The population of these cracks seemed higher the closer the longitudinal ply was located to the centreline (C-C') of the polished surface. The same was true for the traverse plies.

Application of even higher temperature differentials resulted in long, deep HMCs in T1 and T2 (with depth always being higher in T1), longer HMCs in T3, and multiplication of PMCs in all longitudinal plies. However, the depth of PMCs, even in L1, did not increase (Fig. 5.29). In addition, only short HMCs were visible in the outer T3 plies. PMCs were distributed uniformly between longitudinal plies with the same designation. By contrast, HMCs accumulated in the pairs of transverse plies in a more irregular fashion.
The differences in depth between HMCs in T1, T2, and T3 can be clearly seen at the SEM images of Fig. 5.30, which were taken after quenching through $\Delta T=800^\circ C$. 

Fig. 5.29. PMCs and HMC at high temperature differentials.
5.3.2.3.3. Quantification of Thermal Shock Damage Development

The accumulation of damage in the $0^\circ$ plies of this laminate at increasing thermal shocks can be seen in the graph of Fig. 5.31.

Significant increases in crack density are evident and at each temperature differential $CD_{L1} > CD_{L2} > CD_{L3}$. The rate of increase in cracking for L1 looks to be higher than those of L2 and L3. However, the scatter of the experimental results does not allow firm conclusions to be reached.

The accumulation of damage in the transverse plies of this laminate is shown in Fig. 5.32. The density of HMCs increases continuously at a high rate with the application of higher temperature differentials. It has to be noted that only the total crack density is plotted at each $\Delta T$. Generally, it can be assumed that $CD_{T1} > CD_{T2} > CD_{T3}$ at each temperature differential. However, whether
CD_{T1} or CD_{T2} was higher was mostly a random result that depended on the point of origin of the respective HMCs. In general, HMCs emanating from ply edges ran longer lengths.

**Fig. 5.31.** Crack density as a function of ΔT for PMCs for each set of longitudinal plies of (90°/0°)_{3s} Nicalon/CAS laminate. Note that CD_{L1}>CD_{L2}>CD_{L3} at all ΔTs.

**Fig. 5.32.** Crack density as a function of ΔT for HMCs on (90°/0°)_{3s} Nicalon/CAS laminate.
A comparison between the crack densities of each type of damage at each ΔT can be seen in Fig. 5.33. PMCs accumulate at a much higher rate than HMCs.

![Graph showing crack densities](image)

**Fig. 5.33.** Comparison between crack densities of PMCs, HMCs and their total at each ΔT. Relevant trends for each damage mode are also shown.

### 5.3.3. Summary

Damage modes due to thermal shock and their accumulation with increasing shock severity in specimens of two laminate configurations of multi-layer cross-ply CMCs were described in detail in this section.

Matrix cracks of various orientations were identified as the main mode of damage. In longitudinal plies these cracks propagated at right angles to the surface fibre length whereas in transverse plies they ran along the length of the ply. As these matrix cracks were deflected upon
encountering fibre-matrix interfaces, no fibre breaks could be detected in any ply, even at the highest thermal shocks.

The critical quenching temperature difference was found to be higher for the $(90^\circ/0^\circ)_3s$ laminate. Damage in both laminates originated in the central, thick plies and, in the case of the $(90^\circ/0^\circ)_3s$ system, in those plies adjacent to them. The application of higher differentials saw damage being extended to the outer plies until, at intermediate shocks ($\Delta T=600^\circ C$), the surfaces of all the plies were fractured. At even higher quenching temperature differences, damage became more extensive, especially in terms of PMCs in longitudinal plies. However, HMCs in transverse plies were observed, in addition to increasing in length, to penetrate deeper and deeper into the matrix.

The extent of thermal shock damage exhibited a gradient across the material surface: higher crack densities and deeper HMCs were located at or close to the centreline. On moving towards the outer plies the extent of the damage was reduced significantly.

In terms of number of cracks and their measured length as a function of the surface area, damage in the form of PMCs was found to be much more extensive compared with that in the form of HMCs, especially at severe thermal shocks. However, whereas PMCs propagated only at the surface of the laminate, HMCs could be seen to extend deeply into the matrix for $\Delta T\geq 600^\circ C$, as was evident from their increased opening. Unfortunately, the depth they penetrated could not be determined with any accuracy experimentally. However, judging from the crack openings at the surface, the extent of their propagation on the surface (from edge to edge for some specimens at severe shocks), and the fact that in these configurations they cannot meet any ply interface in the depth direction, it can be concluded that their advance must be significant and possibly compromises the structural integrity of the laminate.
A number of general observations can be made regarding the thermal shock behaviour of the laminates analysed in the previous sections.

The main mode of damage after thermal shock treatment in all laminates, irrespective of configuration, is the formation of cracks in the matrix. These cracks were deflected at fibre-matrix interfaces and, thus, did not cause any damage to the fibres, as is to be expected for optimally-designed fibre-reinforced CMCs. This is in accordance with reports of thermal shock damage in similar materials published in the literature (e.g. Kagawa et al. 1993, Blissett et al. 1997 and 1998, Boccaccini et al. 1997 and 1998). However, matrix cracks in different plies advanced in different directions: at right angles to the exposed fibre length in longitudinal plies and along the length of the ply surface in transverse plies. This seems to be a general feature of this class of CMCs. More specifically, Kagawa et al. (1993), Blissett et al. (1997), and Boccaccini et al. (1997 and 1998) reported PMCs on the longitudinal faces of UD Nicalon/Pyrex™, Nicalon/CAS, and Nicalon/DURAN™ respectively. In addition, Blissett et al. (1998) presented evidence of PMCs in the longitudinal plies of (0°/90°)3s and (0°/90°)3s laminates. By contrast, cracks similar to the HMCs described in this chapter have been reported for the end faces of UD Nicalon/CAS (Blissett et al. 1997) and Nicalon/LAS II (Graham et al. 2003), as well as for the transverse plies of (0°/90°)3s and (0°/90°)3s laminates (Blissett et al. 1998). This provides evidence for the biaxiality of the surface stresses developed as a result of thermal shock, as under uniaxial tensile testing only PMCs (as defined here) can be seen on the surfaces of UD or cross-ply CMCs (e.g. Pryce and Smith 1992 and 1994, Beyerle et al. 1992).

Fibre failures, such as those reported by Blissett et al. (1997), were not detected in this investigation. As these authors associated the occurrence of such damage patterns with material...
degradation after high-temperature exposure, it can be concluded that their absence in this study resulted from the short times that the material samples were held at high temperature, which did not allow the formation of any oxidation products on the material surfaces.

The results included in this chapter further validate the approach taken to explain and model cracks similar to HMCs on the end faces of UD Nicalon/CAS in the second part of Chapter 3. It was found that cracking always originated at or near the centreline of the face and crack densities in the central plies were higher at all ATs compared with those of plies located towards the top and bottom edges. This shows that, due to the interaction of temperature gradients and the resulting increase of the stress reduction factor on moving from the top/bottom edges towards the centreline, the highest stress at each AT occurs always at the centreline and gradually reduces as the top and bottom edges are approached. This was true especially for PMCs in longitudinal plies. HMCs followed a more random pattern but plies towards the centreline always had longer and deeper cracks.

Sometimes HMCs could also be seen originating from the side edges, something not predicted by the approach presented in Chapter 3. However, that approach made the assumption that thermal shock was applied at each specimen face at the same time instance. Clearly, this is not true in water quench tests as the angle of impact with the water plays an important role. However, it can still be seen that interaction of temperature gradients was always present at the top and lower part of the specimen face under investigation (no cracks emanated in any specimen from either the top or bottom edges). This possibly shows that, because of the larger area of the top and bottom specimen faces (6 mm x 6 mm here), the temperature gradients corresponding to them are the dominant feature, irrespective of experimental details (i.e. angle of impact etc.). The
gradients emanating from the edges of the side faces, which are much smaller (6 mm x 2.1 mm) or (6 mm x 0.65 mm here) seem to be more sensitive to these details.

The proposed effect of temperature gradient interaction is also evident in the thermal shock resistance of the simple cross-ply laminates. These laminates exhibit critical temperature differentials ~100°C higher than those of their multi-layer counterparts. It must be noted that in the quenched samples the dimensions (half-thicknesses) in the depth and length directions of the surfaces in question were higher than the critical ones. However, the half-thickness of the top and bottom faces was significantly lower than the critical one. This resulted in the interaction of temperature gradients on the whole area of the surfaces under investigation. Thus, the stress reduction factor on these surfaces never achieved its maximum value (even at the centreline) and, thus, these systems experienced lower stresses and showed higher resistance to thermal shock.

The lower thickness of the simple cross-ply laminates, and the subsequent interaction of temperature gradient during thermal shock, seems also to affect the energy available for crack propagation. All matrix cracks in these systems, irrespective of them being PMCs or HMCs, are confined to the surfaces of these materials even when subjected to the highest temperature differential. By contrast, HMCs in the thicker multi-layer laminates became much deeper as the applied shock increased in severity.

The difference with which each type of ply accommodates the energy available for crack propagation must be highlighted. The application of more severe shocks in longitudinal (0°) plies results in the rapid multiplication of surface cracks, which also seem to appear at regular intervals along the ply length. This is similar to the situation under tensile testing (e.g. Pryce and
Smith 1992), and implies that stress transfer may take place during thermal shock between fibre/matrix and between different plies. This stress transfer mechanism results in the energy available for cracking in 0° plies to be consumed mainly in multiplying the number of cracks; by contrast, in 90° plies, as no transfer takes place, the energy available results in the extension (in length and depth) of a small number of cracks that appear at preferential sites. These cracks do not increase in number but become deeper and deeper at higher temperature differentials, something that would affect the integrity of the material. From this aspect, transverse plies behave in a way similar to monolithic or particulate-reinforced ceramic materials under thermal shock loading. By contrast, longitudinal plies show true 'composite', and thus superior, behaviour under thermal shock conditions.
5.5. CONCLUDING REMARKS

Damage due to thermal shock on a range of cross-ply Nicalon/CAS CMCs was characterised and quantified in detail in this chapter.

The damage modes were found to be essentially the same as those reported in the literature and modelled analytically in the previous chapter for UD CMCs of the same composition. In addition, their appearance and evolution for higher shocks seemed to be consistent with the theoretical concepts developed in the previous chapter to explain the location and extension of cracks on end faces of UD CMCs.

The critical temperature differentials for cross-ply CMCs were found to be similar with those of their UD counterparts, although small variations were observed due to different laminate configurations. The effect on $\Delta T_c$ of quenching a sample that has at least one dimension smaller than the critical one was also highlighted.

Before proceeding into analytical predictions of the onset of thermal shock damage in these materials using the tools described in the previous chapter, experimental work was extended to CMC laminates with woven (plain weave) reinforcement architecture. The findings of this investigation are presented in the following chapter.
Chapter 6:

Thermal Shock Behaviour of Woven CMCs
6.1. INTRODUCTION

The behaviour of PW woven Nicalon/CAS CMCs under conditions of thermal shock is examined in detail in this chapter. First, the thermal shock resistance of this material is determined and the various damage modes due to thermal shock are clearly identified. The way damage accumulates on the surfaces of material samples for shocks of increasing severity is described and quantified. The overall response of this laminate is subsequently compared and contrasted with those of UD and cross-ply laminates of the same constitution (i.e. Nicalon fibres, CAS matrix).

In the second part of this chapter, the mechanical behaviour of the PW woven Nicalon/CAS laminate after thermal shock treatment is assessed. Stress-strain curves from both tensile and flexural (3-point bending) tests are presented and are compared with those of the untreated material. Through the analysis of these curves, the effect of thermal shock on the mechanical properties of the material at various quenching temperature differences is quantified. The changes in properties identified are then correlated with the damage modes described in the first part of this chapter. Finally, possible implications for the behaviour of Nicalon/CAS laminates of other configurations under thermal shock are discussed.
6.2. DAMAGE CHARACTERISATION OF THERMALLY-SHOCKED PW WOVEN NICALON/CAS

6.2.1 Critical Quenching Temperature Difference (ΔTc)

Damage due to thermal shock was detected using optical microscopy of samples of this material after quenching through temperature differentials higher than 400°C, i.e. ΔTc=400°C.

6.2.2. Damage Modes due to Thermal Shock

6.2.2.1. Nomenclature

As can be seen from Fig. 6.1, the polished surface a PW woven Nicalon/CAS consists of three different areas: areas that contain longitudinal fibres, areas that contain transverse fibres, and zones with only matrix material present.

![Fig. 6.1. Reflected light micrograph of the surface of a PW woven Nicalon/CAS CMC.](image)

If the undulating nature of all of the above areas is neglected, the woven material can be described as a laminate that contains three different, alternating types of 'ply': 0° plies (i.e. plies with longitudinal fibres), 90° plies (i.e. plies with transverse fibres), and pure matrix plies. This nomenclature will be utilised for the description of the various damage modes and their accumulation in this material after thermal shock loading.
6.2.2.2. General

Two modes of damage were identified on the surfaces of specimens thermally shocked through \( \Delta T \geq 400^\circ C \), namely ‘matrix cracks’ and ‘fibre failures’. Matrix cracks can be further divided into horizontal matrix cracks (HMCs) and perpendicular matrix cracks (PMCs) if the orientation of the cracks relative to the horizontal direction (x-axis) is taken into account.

6.2.2.3. Perpendicular Matrix Cracks

Perpendicular matrix cracks were the first form of thermal shock damage easily identifiable after quenching through \( \Delta T = 400^\circ C \). They ran mainly through pure matrix areas (plies) at right angles to the horizontal, and arrested when they encountered fibres in adjacent plies. These adjacent plies could be either two areas with longitudinal fibres (0° plies), or two areas with transverse fibres (90° plies) or one 0° ply and one 90° ply (Figures 6.2 (a)-(c)).

The same form of damage could also be observed in individual 0° plies. These cracks were short in length, and spanned the matrix between adjacent fibres (Figure 6.3).
Fig. 6.2: Perpendicular matrix cracks on woven Nicalon/CAS running between (a) two 0° plies (b) two 90° plies (c) one 0° ply and one 90° ply. Fibre diameter is 15 µm.

Fig. 6.3: Perpendicular matrix crack inside a 0° ply.

Consequently, it can be concluded that the critical temperature difference for the onset of damage in the 0° plies of a woven material is also ΔT=400°C. However, observation and quantification of perpendicular cracks in the 0° plies was unfortunately very difficult due to the apparent small width of the plies and the significant amount of unavoidable damage incurred by them during the specimen preparation stages.
6.2.2.4. Horizontal Matrix Cracks

HMCs were uniquely observed in the 90° plies. Although they seemed to appear randomly on the specimen surface, closer inspection revealed that most of them occurred in 90° plies towards the centreline of the face. They were bounded by either two 0° plies or by one 0° ply and one pure matrix ply (Figures 6.4 (a)-(b)). In addition, they were contained within the 90° plies, with a small number arresting when they encountered adjacent plies.

![Image](image_url)

Fig. 6.4. Horizontal matrix crack inside a 90° ply bounded by either (a) two 0° plies, or (b) by one 0° ply and one pure matrix ply.

The critical quenching temperature difference for the onset of this damage mode could not be readily determined. HMCs first appeared on some specimens at \( \Delta T = 400^\circ C \). They were very short in length and they originated from either the edges of the specimen or pre-existing damage (e.g. voids) in the 90° plies. Such cracks were visible on all specimens only after quenching through \( \Delta T = 450^\circ C \). Consequently, the critical quenching temperature differential for the onset of horizontal matrix cracking in the woven Nicalon/CAS is taken to be in the range of \( \Delta T = 400-450^\circ C \).
6.2.2.5. Fibre Failures

A fibre failure occurred when a PMC was not deflected or arrested upon encountering a fibre-matrix interface but instead ran through it and continued across the length of the fibre (Figure 6.5(a)-(b)). This phenomenon took place at high temperature differentials ($\Delta T = 700-800^\circ C$) and only in samples that had been kept in the furnace for longer time intervals (15-20 mins). Close inspection of the material surface after testing revealed that the appearance of these cracks could be associated with oxidation processes that altered the microstructure after high temperature exposure.

![Image](image_url)

Fig. 6.5: Fibre fracture due to the propagation of a perpendicular matrix crack at (a) $\Delta T = 700^\circ C$ and (b) $\Delta T = 800^\circ C$ on the surface of samples soaked for longer times. Oxidation of the fibre-matrix interface is evident.

6.2.3. Evolution of Thermal Shock Damage with Increasing Applied Quenching Temperature Differential ($\Delta T$)

6.2.3.1. General

The accumulation of damage with increasing applied $\Delta T$ on samples of this material can be seen in the sequence of Fig. 6.6(a)-(e), where the crack patterns observed under the microscope have been added manually to RLM images of the respective material surfaces.
6.2.3.2. Description of Thermal Shock Damage Development

Perpendicular cracks appeared at $\Delta T_c=400^\circ C$ in matrix-rich areas as described above. At this temperature differential the cracks were short and spanned pure matrix plies of small thickness. They were located in plies towards the centreline of the polished surface.

With increasing $\Delta T$, matrix cracks could be seen running through pure matrix plies of larger thickness. At $\Delta T=600^\circ C$, some PMCs that originated in pure matrix plies ran through 0° plies and continued into the adjacent pure matrix plies before arresting at the next fibre-containing ply (Fig. 6.7).
Some penetration into 90° plies was also observable at $\Delta T=600°C$, but was found to be very small in extent, i.e. the cracks stopped propagating after encountering two or three transverse fibres (Fig. 6.8).

At $\Delta T \geq 600°C$, PMCs could be seen to be evenly spread throughout the surface of the laminate. The depth these cracks penetrated into the matrix and their opening showed only a very small increase with increasing severity of the thermal shock treatment. Thus, in general they remained shallow and narrow surface features at all $\Delta T$s. Some deeper cracks observed even at the lower temperature differentials were found to be pre-existing cracks that had grown due to the applied thermal shocks.

Fully formed horizontal matrix cracks appeared in the 90° plies at $\Delta T \geq 450°C$. These cracks had a number of characteristics when they occurred at temperature differentials $\Delta T \leq 500°C$. First, they rarely ran the full length of the 90° ply. Secondly, they never crossed into adjacent plies, i.e. they always arrested inside the 90° ply or when they encountered adjacent plies. In addition, their depth remained small.
At $\Delta T \geq 600^\circ C$, horizontal cracks became deeper and more widespread (Fig. 6.9(a)-(c)), but they were still contained inside 90° plies.

HMCs could be seen crossing into other plies at the higher differentials investigated ($\Delta T = 700-800^\circ C$) to the extent that in some specimens quenched at $\Delta T = 800^\circ C$ individual HMCs along the centre of the surface connected to form a major, deep crack that traversed the face from edge to edge (Fig. 6.10). This effect was more pronounced in specimens that had been kept for longer times in the furnace.
It must be noted that most HMCs appeared towards the centre of the face at all temperature differentials. In addition, those located at or very close to the centre exhibited greater depths and were longer. Some could be seen originating from the side edges of the surface.

6.2.3.3. Quantification of Thermal Shock Damage Development

The accumulation of damage on woven Nicalon/CAS specimens with increasing severity of thermal shock treatment is shown in Fig. 6.11.

Both PMC and HMC densities increase continuously up to the highest temperature differential. The density of PMCs is always higher than that of HMCs, with the difference between the two getting larger with increasing ΔT. However, the graph of Fig. 6.12 gives a different picture as far as PMCs are concerned.

Here, the numbers of cracks measured on surfaces of the same dimensions at different temperature differentials are shown. These surfaces had been subjected to longer soaking times, which caused microstructural changes (oxidation). It can be seen that the rate of increase of
PMCs slows down for $\Delta T=600-700^\circ C$ and actually their number reduces in the interval $\Delta T=700-800^\circ C$.

**Fig. 6.11.** Crack density as a function of $\Delta T$ for PMCs and HMCs on PW woven Nicalon/CAS.

**Fig. 6.12.** Comparison of number of cracks as a function of $\Delta T$ on quenched surfaces of equal size for samples soaked for longer times.
6.2.4. Discussion

The main damage mode due to thermal shock in this material was the same as that for the range of cross-ply systems described in the previous chapter, i.e. matrix cracking. Similarly, PMCs were observed in areas of longitudinal plies and HMCs in areas with transverse fibres. The so-called pure matrix plies seem to comprise an independent constituent of this laminate as they developed damage in the form of PMCs irrespective of the type of the plies adjacent to them. The way different types of ‘ply’ accommodate the energy available for cracking can be explained by employing the stress transfer mechanism described in section 5.4.

The appearance of fibre damage at high temperature differentials on the surfaces of specimens soaked for longer times in the furnace is consistent with the observations of Blissett et al. (1997) for UD Nicalon/CAS. In both cases, it can be associated with degradation of the carbonaceous fibre-matrix interface due to oxidation processes that allows penetration by an advancing crack and the subsequent fracture of the fibre. In addition, the apparent reduction in PMC density at the highest temperature differential observed for these samples is also consistent with that reported by the same authors for oxidised UD Nicalon/CAS CMCs.

The PW woven Nicalon/CAS laminate exhibited thermal shock resistance similar to the multi-layer cross-ply laminates. This was expected since it had comparable total thickness and ply constitution. The small differences observed in the value of the critical temperature differential should be assigned to small microstructural variations due to the reduced individual ply thickness and the existence of matrix-rich areas.

The modelling approach used to explain cracking morphologies on the end faces of UD and on a number of cross-ply Nicalon/CAS laminates seems to hold for this laminate as well. Damage
originates at or close to the centreline of the surface under observation and extends, at higher quenching temperature differences, towards the top and bottom edges. However, matrix cracks in this laminate, especially PMCs, appeared to be more evenly distributed on the surface even at or close to the $\Delta T_c$. This may be the result of the undulating nature of the various ‘plies’ that compose this system, which probably distribute the applied thermal shock-induced stresses over a larger surface area. The initiation of some HMCs from the side edges of the surface under investigation can be explained along the same lines used to justify the appearance of similar cracks in cross-ply laminates in the previous chapter.

Although the morphology of PMCs and HMCs exhibited similar trends with those observed for their multi-layered cross-ply counterparts (i.e. PMCs increased significantly in number but not in depth, HMCs penetrated increasingly into the matrix) with the application of more severe shocks, there are reasons to believe that the woven configuration shows better behaviour under conditions of thermal shock. First, comparisons of PMC and HMC densities of each simple and multi-layer cross-ply laminate and the woven material at each $\Delta T$ are presented in Fig. 6.13 and Fig. 6.14, respectively.

Careful observation of the above graphs reveals that, at least as far as crack length on the quenched surface is concerned, the woven material exhibits much lower crack densities for both PMCs and HMCs than its multi-layered cross-ply counterparts. Actually, the densities are comparable with those of the simple cross-ply laminates. This, apart from the different ply configuration of the woven CMC, can be assigned to the undulating nature of the different areas within it. PMCs are distributed over a larger area of the surface (as mentioned previously) while HMCs were observed to arrest when they encountered areas of different architecture. Since the length of the transverse plies in woven Nicalon/CAS is nominally much smaller than that in the
simple and multi-layer cross-ply materials, the average length to which HMCs can propagate is restricted. Only at the highest temperature differential ($\Delta T=700-800^\circ C$), and in a small number of samples, were HMCs seen to extend into other plies.

The presence of other areas on the path of HMCs that can act as 'crack-stoppers' leads to the postulate that a similar effect may be taking place in the depth direction as well. Thus, the extent HMCs propagate through the matrix of the woven material should be reduced compared with the multi-layer cross-ply laminates.

Figs. 6.13. Comparison of PMC density at various $\Delta T$s for a range of Nicalon/CAS laminates.
6.14. Comparison of HMC density at various ΔTs for a range of Nicalon/CAS laminates.

It is of great interest to explore what effect damage due to thermal shock has on the structural integrity of the PW woven Nicalon/CAS. This is the theme of the section that follows.
6.3. MECHANICAL TESTING OF THERMALLY-SHOCKED

PW WOVEN NICALON/CAS

6.3.1. Introduction

An assessment of the mechanical properties of samples of PW woven Nicalon/CAS after they
had been subjected to thermal shock treatment is presented in this section. Both tensile and
flexural tests were conducted, the experimental details of which were provided in Chapter 4. The
effect of thermal shock is documented for Young’s modulus and proportional limit stress (PLS)
in the case of tensile tests, and for Young’s modulus, PLS and flexural strength in the case of
bending tests.

6.3.2. Tensile Testing

The stress-strain curves of thermally-shocked specimens obtained from tensile tests are presented
and compared with that of the untreated material in the graph of Fig. 6.15. The curve of the
unshocked sample was found to be similar to that obtained by Ironside (1996), who conducted
tensile tests on the same material but utilised much longer specimen dimensions.

For $\Delta T \leq 500^\circ C$, the resulting stress-strain curves were identical with that of the unshocked
material. They exhibited an initial linear region, which was followed by non-linear behaviour
and a final quasi-linear part of reduced gradient up to material failure.

After increasing the severity of the applied shock to temperature differentials higher than
$\Delta T = 500^\circ C$, the stress-strain curves maintained similar characteristics, i.e. the linear/non-
linear/linear pattern. However, a significant drop in the properties of the thermally-treated material can be observed.

Fig. 6.15. Tensile stress-strain curves for unquenched and quenched PW woven Nicalon/CAS. (RT: room-temperature treatment, i.e. unquenched).

This degradation effect is more evident in the graphs of Figs. 6.16 and 6.17, where changes in the values of Young’ modulus (E) and PLS with increasing applied ΔT are presented.
Fig. 6.16. Effect of increasing severity of applied thermal shock on the tensile Young's modulus of PW Nicalon/CAS.

Fig. 6.17. Effect of increasing severity of applied thermal shock on the tensile proportional limit stress of PW Nicalon/CAS.
Although the small number of specimens used to obtain these properties means that the above results should be approached very carefully, it is evident that both E and PLS exhibit a gradual reduction for $\Delta T \geq 600^\circ C$, the rate of which seems to decrease for the range $\Delta T = 700-800^\circ C$.

The fracture surfaces of specimens quenched through $\Delta T = 600$, 700, and 800$^\circ C$ are compared with those of an untreated sample in Fig. 6.18(a)-(d). No change in failure mode can be seen; in all cases material failure coincided with the fracture of fibre bundles under tension, which was accompanied by extensive pull-out of the reinforcing fibres from the matrix.
Fig. 6.18(a)-(d) The fracture surfaces of unquenched and quenched PW Nicalon/CAS after tensile failure. Test samples had a thickness of 2.2 mm.
6.3.3. Flexural Testing

The stress-strain curves of thermally-shocked samples obtained from flexure tests are plotted in
the graph of Fig. 6.19. In the same graph, the respective curve of the untreated material is also
shown to facilitate comparison of behaviour under bending before and after thermal treatment.

![Fig. 6.19. Flexural stress-strain curves for unquenched and quenched PW woven Nicalon/CAS.]

All curves presented in the above graph exhibit similar characteristics: an initial linear part of
large gradient followed by non-linearity before and after reaching the maximum stress point.
However, for samples shocked through temperature differentials higher than 600°C, a gradual
reduction in properties is evident. This is better visualised in the graphs of Figs. 6.20, 6.21, and
6.22, where the change in Young’s modulus, PLS, and flexural strength with increasing
temperature differential can be seen, respectively.
Fig. 6.20. Effect of increasing severity of applied thermal shock on the flexural Young's modulus of PW Nicalon/CAS.

Fig. 6.21. Effect of increasing severity of applied thermal shock on the flexural proportional limit stress of PW Nicalon/CAS.
Fig. 6.22. Effect of increasing severity of applied thermal shock on the flexural strength of PW Nicalon/CAS

A gradual reduction in all properties for increasing quenching temperature difference is evident. The onset of this reduction is mainly at ΔT=600°C.

The fracture surfaces of specimens quenched through ΔT=600, 700, and 800°C are shown in the sequence Fig. 6.23(b)-(d), together with the image of the fracture surface of an untreated sample (Fig. 6.23(a)). No change in fracture mode can be detected; failure occurs in all samples by fracture across the fibre planes and extensive fibre pull-out.
Fig. 6.23(a)-(d) The fracture surfaces of unquenched and quenched PW Nicalon/CAS after flexural failure. Test samples had a thickness of 2.2 mm.
The mechanical behaviour under tension and flexure of samples of PW woven Nicalon/CAS subjected to thermal shock was assessed in this section. Two important points should be made.

First, the trends obtained in the change of mechanical properties with increasing $\Delta T$ from tensile and flexural tests were identical. In both cases, the material seemed not to be affected by the application of shocks up to $\Delta T=500^\circ C$. Application of higher temperature differentials resulted in a gradual reduction of all properties monitored.

Second, the material exhibited true 'composite' behaviour irrespective of testing method utilised and severity of thermal treatment. This is evident in the non-linearity of the stress-strain curves obtained, which are a characteristic of this class of materials, and the extensive pull-out of the fibres from the matrix observed on the fracture surfaces of failed samples. In addition, the failure modes of thermally-treated and untreated samples were identical.

This shows that damage due to thermal shock alone does not alter the general behaviour of this material; it only affects its mechanical properties. By contrast, several authors have reported changes in failure mode under flexure after thermal shock treatment (e.g. Wang et al. 1996). This discrepancy may be attributed to a number of reasons. First, there may be differences in interlaminar shear strength between the materials that renders some more susceptible to thermal shock damage and results in a change of failure mode. Second, extra care was taken in this investigation to separate thermal shock effects from oxidation-related material degradation. As a result, the samples tested did not exhibit any visible chemical degradation even after high-temperature exposure. Degradation of the fibre-matrix interface in this class of materials is usually correlated with changes in failure mode as the material becomes more brittle. Finally, the
observed discrepancy may be evidence of the superior behaviour of the material with a PW woven architecture compared with UD, cross-ply, and woven materials of other types of weave. It was seen in the previous section that the PW woven CMCs accumulate damage at a lower rate and, as they employ more crack-stopping mechanisms, limit its extent.

However, it is interesting to correlate the reduction in properties observed in the PW woven material for increasing temperature differential with the damage modes due to thermal shock that were described in the previous section. This is the theme of the following section.
6.4. CORRELATION OF CHANGES IN MECHANICAL PROPERTIES DUE TO THERMAL SHOCK WITH OBSERVED THERMAL SHOCK DAMAGE

In the first section of this chapter it was shown that thermal shock damage on this material consisted of matrix cracks, characterised as PMCs and HMCs with respect to their direction of advance, and fibre failures, which were associated with degradation of the fibre-matrix interface due to the reactivity of the material with the environment. As all samples used in mechanical tests were held at high temperature for short periods of time, no degradation was evident on their surfaces. Thus, none of the changes in mechanical properties reported previously can be assigned to damage sustained by the fibres during thermal shock treatment.

This leaves two crack morphologies that may affect mechanical properties: PMCs in pure matrix and longitudinal ‘plies’ and HMCs in transverse plies. The first observation that can be made is that the critical temperature differential for the onset of either type of matrix cracking does not coincide with the respective temperature differential for which property degradation begins. More specifically, damage appears for $\Delta T=400$-450°C while property degradation commences at $\Delta T=600°C$. This is a well-documented feature of this class of materials as it has also been reported by a number of other authors (Wang et al. 1996, Boccaccini et al. 1997 and 1998). It can be put into context in two different ways: either it reinforces the evidence of superior performance of fibre-reinforced CMCs under conditions of thermal shock compared with monolithic and particulate-reinforced ceramics, since not only do these materials exhibit no abrupt decrease in properties but also they can sustain some damage without any effect on their properties; or it reveals the limitations of mechanical testing as a technique that can detect damage due to thermal shock. The latter has been highlighted in the work of the abovementioned...
authors who used a mechanical resonance technique that was able to detect property degradation of thermally-shocked specimens at the temperature differential at which matrix damage first appeared. It seems logical that since some damage has been incurred it would have an effect on properties. However on the other hand, damage detected at low temperature differentials, whether PMCs or HMCs, was in the form of very shallow, hair-like cracks barely visible under the optical microscope that should not affect the integrity of the material more than any form of surface flaw induced in the specimen preparation stages.

This idea is reinforced by the observation that the onset of material degradation begins at the same temperature differential that HMCs in transverse plies start to penetrate deeply into the matrix. By contrast, PMCs remain surface features throughout the range of quenching temperature differentials employed in this study. However, at the same time as HMCs deepened, PMCs started to extend into adjacent plies. In order to be able to tell what effect each damage mode had on the material properties, additional thermal shock experiments were devised and conducted.

More specifically, polished samples of PW woven Nicalon/CAS suitable for flexural testing were quenched through $\Delta T=700^\circ$C. After the samples had been dried and before testing, they were polished lightly using diamond paste until all the PMCs that had appeared in the pure matrix and longitudinal plies could not be detected using optical microscopy. HMCs were not affected by this procedure and were still visible inside the transverse plies. Subsequently, the specimens were tested in flexure. The stress-strain curve obtained is presented in Fig. 6.24, where it is plotted together with that obtained for specimens quenched through the same temperature differential but containing both HMCs and PMCs.
The curves of Fig. 6.24 reveal that specimens without PMCs exhibit the same Young's modulus and a slightly higher PLS. This leads to the conclusion that HMCs that penetrate deeply into the matrix are responsible for the reduction in the stiffness of the material. By contrast, PMCs that increase in length seem to be mainly responsible for the documented reduction in PLS. However, as the onset of reduction in PLS coincides with the increase in depth of HMCs, it cannot be ruled out that HMCs may also play some part in the observed reduction.

From the results presented in this section it is not clear why a reduction is observed in the strength of this material at high temperature differentials. Strength degradation is usually associated with damage to the reinforcing fibres. However, no fibre failure could be detected after thermal shock treatment in this study. Three reasons can be put forward in trying to explain
this phenomenon. First, the inherent strength of the fibres may be affected by high-temperature exposure. Second, the large densities of cracking phenomena sustained by the samples at high differentials may affect the general integrity of the material and this is evidenced as a reduction in strength. However, a more convincing explanation may be found in the differences the values of strength showed even for specimens quenched through the same temperature differential. As only a very small number of specimens was available for mechanical testing, the perceived reduction in strength may simply be a result of experimental scatter in the measured strength values, which is quite normal for ceramic materials.

6.5. CONCLUDING REMARKS

The thermal shock behaviour of PW Nicalon/CAS was extensively studied in this chapter. The main damage modes were found to be similar to those observed in cross-ply CMCs with the same constituents. However, the extent of damage on the woven material was lower at the same temperature differentials.

Mechanical testing of thermally-shocked specimens revealed a gradual reduction in properties. However, the onset of this reduction was found to be at a higher $\Delta T$ than the onset of damage on material surfaces. It was shown that it coincided with significant changes in the morphology of one mode of damage, namely matrix cracking in the transverse plies.

As information about thermal shock damage has now been collected on a wide range of CMC laminates, an attempt should follow to describe the onset of this damage analytically. This can be performed by extending the theoretical concepts developed for the UD material in Chapter 3 to the case of 2-D CMCs. This is the theme of the following chapter.
Chapter 7:

The Onset of Thermal Shock Fracture in 2-D CMCs
7.1. INTRODUCTION

The analytical expressions developed in Chapter 3 to predict the onset of thermal shock damage on surfaces of UD CMCs are applied in this chapter to predict the appearance of the cracking phenomena on the surface of CMCs reinforced with 2-D fibre architecture.

Although the basic model principles are the same, the analysis has to account for the variation in composition and dimensions among the different CMC laminates. The critical conditions are modified and the thermally-induced stresses are re-defined.

It must be noted that the type of critical condition that applies to each CMC to describe the onset of thermal shock fracture depends on the arrangement of its plies. By taking into account the experimental findings of the previous two chapters, it can be said that the criterion for perpendicular matrix cracking is applicable to the case of CMCs whose central plies are longitudinal ones. By contrast, the fracture mechanics-based criterion for fracture of the end face of a UD CMC is better suited for the CMC laminates with transverse central plies.
7.2. THE CRITICAL CONDITIONS FOR CRACKING DUE TO THERMAL SHOCK

7.2.1. Longitudinal (0°) Plies

Cracking due to thermal shock in the longitudinal plies of 2-D CMCs commences when the applied thermal stresses become equal to the matrix strength. This can be written analytically as:

\[ \sigma_{x,M}^{TS} + \sigma_{x,M}^{RES} + \sigma_{x,M}^{RS} = \sigma_{mu} \]  

[7.1]

In [7.1], \( \sigma_{x,M}^{TS} \) is the axial applied thermal shock stress, \( \sigma_{x,M}^{RES} \) describes the axial residual thermal stress at the ply level due to differences in CTE between fibre and matrix, while \( \sigma_{x,M}^{RS} \) is the axial residual thermal stress at the laminate level due to CTE differences between the different plies that constitute the composite. Finally, \( \sigma_{mu} \) is the matrix fracture stress.

Equation [7.1] describes the onset of fracture in 2-D laminates whose central ply is longitudinal. This covers (90°/0°)_s and (90°/0°)_3s cross-ply CMCs, as well as the longitudinal plies of woven laminates located towards the centreline of the quenched surface.

7.2.2. Transverse (90°) Plies

As it was shown in Chapter 3, a fracture mechanics-based criterion is applicable to the onset of thermal shock damage in composite surfaces containing transverse plies. Thus, damage appears when the applied thermal shock-induced stress intensity factor becomes equal to the relevant fracture toughness, i.e. when:

\[ K_{I}^{TS} = K_{IC} \]  

[7.2]
This criterion can be used to predict the onset of thermal shock damage in \((0°/90°)_s\) and \((0°/90°)_{35}\) cross-ply CMCs, as well as in the transverse 'plies' of woven laminates located towards the centreline of the surface under investigation.

7.3. THE APPLIED STRESS FIELD

7.3.1. The Thermal Shock-Induced Stress

7.3.1.1. Cross-ply CMCs

Analysis of the stresses due to thermal shock in a cross-ply CMC is performed, as in Chapter 3, using maximum values of stress that occur at the surface. We consider the surface of a rectangular plate of cross-ply CMC initially at temperature \(T_1\) (Fig. 7.1). The composite has thickness \(t\), and consists of longitudinal plies of total thickness \(t_0\) and transverse plies of total thickness \(t_{90}\). Each ply comprises a matrix of volume fraction \(V_m\) with properties \(E_m, \alpha_m, \nu_m\), which contains fibres of volume fraction \(V_f\) with properties \(E_f, \alpha_f, \nu_f\).

![Fig. 7.1. A cross-ply composite subjected to thermal shock. Thermal shock-induced stresses are also shown.](image)
If the material is rapidly cooled from $T_1$ to $T_o$ and perfect heat transfer between the plate and the cooling medium is assumed, the surface immediate adopts the temperature $T_o$ while the other parts of the plate remain at $T_1$. This case corresponds to having a plate that can freely expand in the z-direction (i.e. perpendicular to the plane of Fig. 7.1), with suppressed expansion in the x- and y-directions.

In the absence of displacement restrictions, the longitudinal and the transverse plies would expand along the x-direction and the y-direction by thermal strains of:

$$
\varepsilon_{x,0}^{th} = \alpha_L (T_o - T_1) \quad [7.3]
$$

$$
\varepsilon_{x,90}^{th} = \alpha_T (T_o - T_1) \quad [7.4]
$$

$$
\varepsilon_{y}^{th} = \left( \frac{t_0}{t} \right) \alpha_T (T_o - T_1) + \left( \frac{t_{20}}{t} \right) \alpha_T (T_o - T_1) \quad [7.5]
$$

The subscript ‘0’ denotes longitudinal ply whereas the subscript ‘90’ denotes transverse ply. The CTEs in each ply along and at right angles to the fibres, $\alpha_L$ and $\alpha_T$, are given by [3.6] and [3.7] respectively. Since thermal expansion in both directions is completely suppressed, elastic strains are created that compensate the thermal strains, i.e.

$$
\varepsilon_{x,0}^{el} + \varepsilon_{x,0}^{th} = 0 \quad [7.6]
$$

$$
\varepsilon_{x,90}^{el} + \varepsilon_{x,90}^{th} = 0 \quad [7.7]
$$

$$
\varepsilon_{y}^{el} + \varepsilon_{y}^{th} = 0 \quad [7.8]
$$
From equations [7.3]-[7.8] we have:

$$\varepsilon_{x,0}^{el} = -\varepsilon_{x,0}^{th} = -\alpha_L(T_o - T_i) = \alpha_L(T_i - T_o) = \alpha_L \Delta T \tag{7.9}$$

$$\varepsilon_{x,90}^{el} = -\varepsilon_{x,90}^{th} = -\alpha_T(T_o - T_i) = \alpha_T(T_i - T_o) = \alpha_T \Delta T \tag{7.10}$$

$$\varepsilon_{y}^{el} = -\varepsilon_{y}^{th} = -\left(\frac{t_0}{t}\right)\alpha_T(T_o - T_i) - \left(\frac{t_{90}}{t}\right)\alpha_T(T_o - T_i) = \left(\frac{t_0}{t}\right)\alpha_T \Delta T + \left(\frac{t_{90}}{t}\right)\alpha_T \Delta T \tag{7.11}$$

The elastic strains cause ‘thermal stresses’ along the principal axes of the material and can be written as:

$$\varepsilon_{x,0}^{el} = \frac{\sigma_{x,0}^{TS}}{E_L} - \frac{\nu_{LT}\sigma_y^{TS}}{E_T} \tag{7.12}$$

$$\varepsilon_{x,90}^{el} = \frac{\sigma_{x,90}^{TS}}{E_T} - \frac{\nu_{TL}\sigma_y^{TS}}{E_L} \tag{7.13}$$

$$\varepsilon_{y}^{el} = \left(\frac{\sigma_y^{TS}}{E_T} - \frac{\nu_{LT}\sigma_{x,0}^{TS}}{E_L}\right)\left(\frac{t_0}{t}\right) + \left(\frac{\sigma_y^{TS}}{E_T} - \frac{\nu_{TT}\sigma_{x,90}^{TS}}{E_L}\right)\left(\frac{t_{90}}{t}\right) \tag{7.14}$$

where $\nu_{LT}$, $\nu_{TL}$ and $\nu_{TT}$ are the relevant Poisson’s ratios. It can be assumed that $\nu_{LT} = \nu_{12}$, given by [3.8], and $\nu_{TT} = \nu_m$. In addition, $\nu_{TL}$ can be approximated by [3.17] using simple rule of mixture estimates of the axial and transverse moduli of the laminate.

By substituting [7.12], [7.13] and [7.14] in [7.9], [7.10] and [7.11] and solving for the induced thermal shock stresses we get:

$$\sigma_{x,0}^{TS} = A Q_{x,0} \Delta T \tag{7.15}$$

$$\sigma_{x,90}^{TS} = A Q_{x,90} \Delta T \tag{7.16}$$

$$\sigma_y^{TS} = A Q_y \Delta T \tag{7.17}$$
where:

\[ Q_{x,0} = E_t \alpha_L + \nu_{LT} E_L \left[ t_0 (\alpha_L + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) \right] \frac{1}{t_0 (1 - \nu_{LT} \nu_{LT}) + t_{90} (1 - \nu_{TT}^2)} \]  

[7.18]

\[ Q_{x,90} = E_T \alpha_T + \nu_{LT} E_L \left[ t_0 (\alpha_L + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) \right] \frac{1}{t_0 (1 - \nu_{LT} \nu_{LT}) + t_{90} (1 - \nu_{TT}^2)} \]  

[7.19]

\[ Q_y = \frac{E_T \left[ t_0 (\alpha_T + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) \right]}{t_0 (1 - \nu_{LT} \nu_{LT}) + t_{90} (1 - \nu_{TT}^2)} \]  

[7.20]

The stress reduction factor, \( A \), has also been included in [7.15], [7.16] and [7.17].

7.3.1.2. Woven CMCs

The woven CMC is modelled as a composite of thickness \( t \) that consists of longitudinal ‘plies’ of total thickness \( t_0 \), transverse ‘plies’ of total thickness \( t_{90} \), and matrix ‘plies’ of total thickness \( t_m \) (Fig. 7.2). The subscript ‘m’ denotes matrix ply.

Assuming the same initial conditions and thermal treatment as in the case of cross-ply CMCs we have that:

**Fig. 7.2.** A woven CMC subjected to thermal shock. Thermal shock-induced stresses are also shown.
\[ \varepsilon_{x,0} = \varepsilon_{x,90} = \varepsilon_{x,m} = 0 \]  \hspace{1cm} \textbf{[7.21]}

\[ \varepsilon_y = 0 \]  \hspace{1cm} \textbf{[7.22]}

\[ \sigma_{y,0}^{TS} = \sigma_{y,90}^{TS} = \sigma_{y,m}^{TS} = \sigma_y^{TS} \]  \hspace{1cm} \textbf{[7.23]}

\[ \sigma_z = 0 \]  \hspace{1cm} \textbf{[7.24]}

The resulting equations have the following form:

\[ \varepsilon_{x,0}^{el} = \frac{\sigma_{x,0}^{TS}}{E_L} - v_{TL} \frac{\sigma_y^{TS}}{E_T} - \alpha_L \Delta T = 0 \]  \hspace{1cm} \textbf{[7.25]}

\[ \varepsilon_{x,90}^{el} = \frac{\sigma_{x,90}^{TS}}{E_T} - v_{TT} \frac{\sigma_y^{TS}}{E_T} - \alpha_T \Delta T = 0 \]  \hspace{1cm} \textbf{[7.26]}

\[ \varepsilon_{x,m}^{el} = \frac{\sigma_{x,m}^{TS}}{E_m} - v_m \frac{\sigma_y^{TS}}{E_m} - \alpha_m \Delta T = 0 \]  \hspace{1cm} \textbf{[7.27]}

\[ \varepsilon_y^{el} = \left( \frac{\sigma_y^{TS}}{E_T} - v_{LT} \frac{\sigma_{x,0}^{TS}}{E_L} - \alpha_L \Delta T \right) \frac{t_0}{t} + \left( \frac{\sigma_y^{TS}}{E_T} - v_{TT} \frac{\sigma_{x,90}^{TS}}{E_T} - \alpha_T \Delta T \right) \frac{t_{90}}{t} + \]

\[ + \left( \frac{\sigma_y^{TS}}{E_m} - v_m \frac{\sigma_{x,m}^{TS}}{E_m} - \alpha_m \Delta T \right) \frac{t_m}{t} = 0 \]  \hspace{1cm} \textbf{[7.28]}

Solution of this system of equations provides the thermal shock-induced stresses in each ply, after the inclusion of the stress reduction factor, as:

\[ \sigma_{x,0}^{TS} = A\eta_{x,0} \Delta T \]  \hspace{1cm} \textbf{[7.29]}

\[ \sigma_{x,90}^{TS} = A\eta_{x,90} \Delta T \]  \hspace{1cm} \textbf{[7.30]}

\[ \sigma_{x,m}^{TS} = A\eta_{x,m} \Delta T \]  \hspace{1cm} \textbf{[7.31]}

\[ \sigma_y^{TS} = A\eta_y \Delta T \]  \hspace{1cm} \textbf{[7.32]}
where:

\[
Q_{x,0} = E_L \alpha_L + \frac{\nu_{TL} E_m E_T \left[ t_0 (\alpha_T + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) + t_m \alpha_m (1 + \nu_m) \right]}{t_m E_T (1 - \nu_m^2) + t_{90} E_m (1 - \nu_{TT}^2) + t_0 E_m (1 - \nu_{LT} \nu_{TL})} \tag{7.33}
\]

\[
Q_{x,90} = E_T \alpha_T + \frac{\nu_{TT} E_m E_T \left[ t_0 (\alpha_T + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) + t_m \alpha_m (1 + \nu_m) \right]}{t_m E_T (1 - \nu_m^2) + t_{90} E_m (1 - \nu_{TT}^2) + t_0 E_m (1 - \nu_{LT} \nu_{TL})} \tag{7.34}
\]

\[
Q_{x,m} = E_m \alpha_m + \frac{\nu_m E_m E_T \left[ t_0 (\alpha_T + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) + t_m \alpha_m (1 + \nu_m) \right]}{t_m E_T (1 - \nu_m^2) + t_{90} E_m (1 - \nu_{TT}^2) + t_0 E_m (1 - \nu_{LT} \nu_{TL})} \tag{7.35}
\]

\[
Q_y = \frac{E_m E_T \left[ t_0 (\alpha_T + \nu_{LT} \alpha_L) + t_{90} \alpha_T (1 + \nu_{TT}) + t_m \alpha_m (1 + \nu_m) \right]}{t_m E_T (1 - \nu_m^2) + t_{90} E_m (1 - \nu_{TT}^2) + t_0 E_m (1 - \nu_{LT} \nu_{TL})} \tag{7.36}
\]

It can be noted that when \( t_m = 0 \), [7.33]-[7.36] reduce to [7.18]-[7.20].

### 7.3.2. The Residual Stress Field

#### 7.3.2.1. Cross-Ply CMCs

In order to calculate residual stresses in each ply due to CTE differences between different plies we assume that after the material has been cooled from its processing temperature the thermal strains generated remain compatible, i.e. that:

\[
E_{x,0}^{RS} = E_{x,90}^{RS} = E_{x,m}^{RS} \tag{7.37}
\]

In addition, any residual stresses in the y-direction are neglected, i.e. \( \sigma_y^{RS} = 0 \). The strains in the longitudinal and transverse plies along the x-direction can be written as:
\[ \varepsilon = \frac{\sigma_{x,0}^{RS}}{E_L} - \alpha_x \Delta T_F \]  
\[ 7.38 \]

\[ \varepsilon = \frac{\sigma_{x,90}^{RS}}{E_T} - \alpha_T \Delta T_F \]  
\[ 7.39 \]

where again \( \Delta T_F = T_p - T_{\text{max}} \). In addition:

\[ \sigma_{x,0}^{RS} + \sigma_{x,90}^{RS} = 0 \]  
\[ 7.40 \]

Solution of these equations gives the residual stresses at the laminate level as:

\[ \sigma_{x,0}^{RS} = \Theta_{x,0} \Delta T_F \]  
\[ 7.41 \]

\[ \sigma_{x,90}^{RS} = \Theta_{x,90} \Delta T_F \]  
\[ 7.42 \]

where:

\[ \Theta_{x,0} = \frac{t_0 E_T E_L (\alpha_T - \alpha_L)}{t_90 E_T + t_0 E_L} \]  
\[ 7.43 \]

\[ \Theta_{x,90} = \frac{t_0 E_T E_L (\alpha_T - \alpha_L)}{t_90 E_T + t_0 E_L} \]  
\[ 7.44 \]

### 7.3.2.2. Woven CMCs

Following the same procedure for the three-part model of a woven CMC we get:

\[ \sigma_{x,0}^{RS} = \Theta_{x,0} \Delta T_F \]  
\[ 7.45 \]
\[
\sigma_{x,90}^{RS} = \Theta_{x,90} \Delta T_F \tag{7.46}
\]
\[
\sigma_{x,m}^{RS} = \Theta_{x,m} \Delta T_F \tag{7.47}
\]
where:
\[
\Theta_{x,0} = \frac{t_{90} E_L E_T \left( \alpha_m - \alpha_T \right) + t_0 E_L^2 \left( \alpha_m - \alpha_T \right)}{t_m E_m + t_{90} E_T + t_0 E_L} - E_L \left( \alpha_m - \alpha_T \right) \tag{7.48}
\]
\[
\Theta_{x,90} = \frac{t_{90} E_T \left( \alpha_m - \alpha_T \right) + t_0 E_L E_T \left( \alpha_m - \alpha_T \right)}{t_m E_m + t_{90} E_T + t_0 E_L} - E_T \left( \alpha_m - \alpha_T \right) \tag{7.49}
\]
\[
\Theta_{x,m} = \frac{t_{90} E_T E_m \left( \alpha_m - \alpha_T \right) + t_0 E_L E_m \left( \alpha_m - \alpha_T \right)}{t_m E_m + t_{90} E_T + t_0 E_L} \tag{7.50}
\]

It must be noted that the thermal residual stresses inside each longitudinal ply due to CTE difference between fibre and matrix are still assumed to be given by \([3.26]\) and \([3.37]\).

### 7.4. APPLICATION OF THE CRITICAL CONDITIONS

#### 7.4.1. Longitudinal Plies

The critical condition \([7.1]\) for the longitudinal plies of a 2-D laminate becomes through \([7.15]\) or \([7.29]\) (depending on whether the laminate is cross-ply or woven), \([7.41]\) or \([7.45]\), \([3.26]\) (for \(\Theta_i = \Theta_L\)), and \([3.28]\):

\[
\frac{AE_m Q_x \Delta T_c}{E_L} + \Theta_L \Delta T_F + \frac{E_m \Theta_{x,0} \Delta T_F}{E_L} = \left( \frac{6d E_m E_f V_f^2}{E_L r V_m} \right)^{\frac{1}{3}} \tag{7.51}
\]
The parameter \((E_m/E_L)\) has been inserted following [3.22], as we are interested in matrix stresses. The approach taken to obtain \(\Delta T_c\) is a simplified graphical one since it has already been established in Chapter 3 that \(\bar{A} = 0.55\). Thus, the change in interfacial shear stress, \(\tau\), as a function of \(\Delta T\) is plotted for this value of \(A\) using [7.51] and a Coulomb-type formula, similar to [3.41], that can be written in this case, through [3.37], as:

\[
\tau = -\mu \left[ (\Theta_T \Delta T_R) + \left( -\frac{CA_T}{r} \right) + (AQ_\alpha \Delta T) \right] \tag{7.52}
\]

where \(\Theta_1 = \Theta_2\). The value of \(\mu\) can be found with the same method employed in Chapter 3, i.e. by applying [7.52] at room-temperature and using experimentally-determined values of \(\tau\).

Solution of [7.51] for \(\tau\) gives:

\[
\tau = \left( \frac{E_L r V_m}{6 \Gamma_m E_m E_f V_f^2} \right) \left( \frac{E_m}{E_L} \right) A Q_{\alpha,0} \Delta T + \left( \frac{\Theta_L + \frac{E_m \Theta_{\alpha,0}}{E_L}}{\Delta T_R} \right) \tag{7.53}
\]

The onset of thermal shock fracture occurs at the value of \(\Delta T\) where the two equations describing \(\tau\) meet.

### 7.4.2. Transverse Plies

The required fracture toughness in the case of the transverse plies of 2-D CMCs is again given by either [3.64] or [3.65]. In addition, the shock-induced stress intensity factor can be written following the approach outlined in paragraph 3.3.2.2.2, through [7.17] or [7.32], as:
Thus, the critical condition [7.2] for the onset of thermal shock fracture is:

\[ K_{\text{c}} = \frac{\Delta Q}{A} \sqrt{m} = \frac{\Delta' Q}{A'} \sqrt{m_e} \]  

[7.55]

and the critical quenching temperature difference:

\[ \Delta T_c = \frac{K_{\text{c}}}{\Delta' Q} \sqrt{m_e} \]  

[7.56]

In [7.56], \( t_c \) and \( A' \) are provided by the method outlined in paragraph 3.3.2.2.2, where the thermal conductivity of the composite given by a simple rule of mixtures formula involving the conductivities of the longitudinal ([3.60]) and the transverse ([3.61]) plies.

7.5. CORRELATION WITH EXPERIMENTAL RESULTS

7.5.1. Longitudinal plies

The method outlined in section 7.4.1 is applied here for the prediction of the onset of thermal shock fracture in the longitudinal plies of cross-ply and woven Nicalon/CAS CMCs investigated in the previous two chapters. Two points have to be made before proceeding.

First, it was shown that the \( (90^\circ/0^\circ)_{s} \) laminate exhibited higher \( \Delta T_c \), since one of its dimensions was less than the critical one and, thus, interaction of gradients occurred. This effect can be incorporated in the present approach by initially obtaining a value for the HTC, \( h \), through [3.62]
for \( t \) equal to the critical dimension and \( A = 0.53 \) or 0.55. Subsequently, [3.62] is re-applied at this \( h \) by putting \( t \) equal to half the dimension of the material that is less than the critical one. This procedure gives for the \((90^\circ/0^\circ)_s\) laminate a value of \( A \approx 0.41 \) or 0.39.

The second issue that has to be highlighted involves the volume fractions of the constituents of the longitudinal (and transverse) plies in the woven Nicalon/CAS. Clearly, in this case \( V_f \) is not equal to 0.35 (i.e. the composite fibre volume fraction), as the model presented in paragraph 7.3.1.2 incorporates the observation that a significant proportion of the composite thickness corresponds to pure matrix plies. Close observation of photomicrographs of woven Nicalon/CAS surfaces, such as that of Fig. 6.1, reveals that approximately we have: \( (t_{90}/t) = 0.333 \), \( (t_{90}/t) = 0.375 \), and \( (t_\perp/t) = 0.292 \). Thus, the total fraction of longitudinal and transverse plies in the composite is about 0.71 (or 71%). The fibre volume fraction in these plies for a composite fibre volume fraction of 0.35 is 0.35/0.71, i.e. \( V_{f,\text{ply}} = 0.49 \) and \( V_{m,\text{ply}} = 0.51 \). The approach is similar to the one followed by Aubard (1995), who modelled a 2-D SiC /SiC CMC as an assembly of four unidirectional plies, and an area (‘ply’) that contained matrix and pores. A simple rule of mixtures calculation using the above ply ratios and the values obtained for \( V_{f,\text{ply}} \) and \( V_{m,\text{ply}} \) further validates this approach since it leads to a calculated value of composite modulus as 118 GPa. Ironside (1996) has reported for the same material an experimental value of 120.6 GPa, i.e. the error is only 2%.

Plots of [7.52] and [7.53] for the \((90^\circ/0^\circ)_3\), the \((90^\circ/0^\circ)_s\), and the woven Nicalon/CAS laminates can be seen in Figs. 7.3(a)-(c) respectively. Only the resulting graphs for \( A = 0.55 \) (for the first and third) and \( A = 0.41 \) (for the second) are shown.
(a)

(b)
Fig. 7.3. Prediction of the onset of thermal shock damage in the central longitudinal plies of (a) (90°/0°)\textsubscript{3s}, (b) (90°/0°)\textsubscript{s}, and (c) woven Nicalon/CAS laminates. The line showing a reduction of $r$ as a function of $\Delta T$ (blue line) corresponds to [7.52], while the black line corresponds to [7.53]. The corresponding $\Delta T$ at the point where they meet is the $\Delta T_c$. Predictions are shown for $A=0.55$.

The resulting $\Delta T_c$ for each CMC from the above plots and from their equivalent ones for $A=0.53$ (for the (90°/0°)\textsubscript{3s} and the woven laminates) and $A=0.39$ (for the (90°/0°)\textsubscript{3s} laminate) are presented in Table 7.1.

<table>
<thead>
<tr>
<th>Nicalon/CAS</th>
<th>(90°/0°)\textsubscript{3s} \textsubscript{AT}=400°C</th>
<th>(90°/0°)\textsubscript{s} \textsubscript{AT}=500°C</th>
<th>Woven \textsubscript{AT}=400°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Delta T_c$ (°C)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$A=0.55$</td>
<td>386</td>
<td>-</td>
<td>374</td>
</tr>
<tr>
<td>$A=0.53$</td>
<td>402</td>
<td>-</td>
<td>390</td>
</tr>
<tr>
<td>$A=0.41$</td>
<td>-</td>
<td>533</td>
<td>-</td>
</tr>
<tr>
<td>$A=0.39$</td>
<td>-</td>
<td>547</td>
<td>-</td>
</tr>
</tbody>
</table>
From the results of Table 7.1, it can be seen that the error in the prediction for the \((90°/0°)_3s\) laminate is 0.5-3.5\%, for the \((90°/0°)_s\) is 6.5-9.5\%, while for the longitudinal plies of the woven laminate it is 2.5-4\%.

7.5.2. Transverse Plies

Equation [7.56] is applied in this paragraph for the prediction of the onset of thermal shock damage in the thick, central transverse plies of \((0°/90°)_3s\) and \((0°/90°)_s\), as well as for central 90° plies of woven Nicalon/CAS CMCs. The prediction for the \(\Delta T_c\) of the \((0°/90°)_s\) laminate reported by Blissett et al. (1998) is also included. It must be noted that the two points highlighted in the previous paragraph regarding the stress reduction factor, \(A\), and consequently the parameter \(A'\), of the \((90°/0°)_s\) laminate as well as the fibre volume fraction of longitudinal and transverse plies in the woven material are still applicable here. The results are presented in Table 7.2.

**Table 7.2.** The results of the application of [7.55] to a range Nicalon/CAS laminates. The values of \(A'\) were calculated with the method described in paragraph 3.3.2.2.2. In addition, \(K_{IC}^A\) and \(K_{IC}^B\) are given by [3.64] and [3.65] respectively.

<table>
<thead>
<tr>
<th>Nicalon/CAS</th>
<th>((0°/90°)_3s) ((\Delta T_c=350°C))</th>
<th>((0°/90°)_s) ((\Delta T_c=450°C))</th>
<th>((0°/90°)_s) ((\Delta T_c=400°C))</th>
<th>Woven ((\Delta T_c=400°C))</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\Delta T_c)(^{\circ}C)</td>
<td>(A') = 0.18</td>
<td>(A') = 0.19</td>
<td>(A') = 0.13</td>
<td>(A') = 0.14</td>
</tr>
<tr>
<td></td>
<td>(K_{IC}^A)</td>
<td>(K_{IC}^B)</td>
<td>(K_{IC}^A)</td>
<td>(K_{IC}^B)</td>
</tr>
<tr>
<td></td>
<td>272</td>
<td>320</td>
<td>-</td>
<td>270</td>
</tr>
</tbody>
</table>

It can be seen that the discrepancy between predicted and experimentally-determined values is 8-26\% for the \((0°/90°)_3s\), 0.2-21\% for the \((0°/90°)_s\), 15-32\% for the woven, and 20-36\% for the \((0°/90°)_s\) laminate. The better results are produced for \(K_{IC} = K_{IC}^B\).
7.6. DISCUSSION

The resulting predictions for those laminates where thermal shock damage initiates at central, longitudinal plies are close to the experimentally-determined values of $\Delta T_c$. An increased discrepancy is only evident in the prediction for the $(0^\circ/90^\circ)_s$ laminate. This error may be associated with the simple method employed to incorporate the effect of reduced thickness on the value of the stress reduction factor. The assumption that the same HTC, $h$, is applicable for different temperature differentials is not strictly valid (see experimental data in Wang and Singh 1994). Nevertheless, it can be considered a useful working approximation since the observed trends are described accurately and the predictions made are satisfactory.

The predictions for the central, transverse plies of $(0^\circ/90^\circ)_3s$ and $(0^\circ/90^\circ)_s$ laminates are reasonably good. Less satisfactory is the result for the $(0_2^\circ/90_4^\circ)_s$ CMC, where the error can reach 36% of the experimental value. However, a possible explanation can be provided if the Young’s moduli of the above cross-ply Nicalon/CAS CMCs are considered carefully. A simple rule of mixtures estimate for the $(0^\circ/90^\circ)_3s$ and $(0^\circ/90^\circ)_s$ laminates gives their moduli as 117 GPa. This is within the experimental range of 110-120 GPa reported by Pryce and Smith (1992) for these materials. The modulus for the $(0_2^\circ/90_4^\circ)_s$ laminate is calculated to be 115 GPa from the rule of mixtures whereas the previous authors reported an experimental average value of 101 GPa. A usual way to accommodate for this discrepancy is to adjust the modulus of the transverse plies from 110 GPa (computed using [3.16] for $V_f=0.34$) to a lower value such that the experimental value is obtained. Using this adjusted value (90 GPa), the prediction for the onset of thermal shock damage in the $(0_2^\circ/90_4^\circ)_s$ CMC becomes $\Delta T_c=369-389^\circ\text{C}$, i.e. the error is reduced significantly to 3-8%.
The discrepancy in the computed value for the $\Delta T_c$ of the woven Nicalon/CAS can be considered acceptable bearing in mind the simplifications involved in modelling the woven material as an assembly of plies of three different types. For example, the undulation of the plies is completely ignored and the large variation in ply thickness is not accounted for. In addition, it is not guaranteed that the central transverse plies in this material are always included in the area of maximum thermal shock-induced stress.

As stated in the previous chapter, the appearance of PMCs in the matrix plies of the PW woven Nicalon/CAS constitutes a damage mechanism unique for this material. The way these cracks are distributed denotes a form of ‘composite behaviour’ since arrays of these shallow cracks can be observed that become almost equidistant at high $\Delta T$s. However, no model exists that can describe the onset of fracture in these plies when embedded in a woven material. A simple estimate can be provided by following a fracture mechanics approach. More specifically, assuming the existence of a surface crack $c$, the shock-induced stress intensity factor can be written as:

$$K_f^{TS} = \left( \sigma_{x,m}^{TS} + \sigma_{x,m}^{NS} \right) \frac{1}{\pi c}$$  \hspace{1cm} [7.57]

Equation [7.57] becomes through [7.2], [7.31] and [7.47] at the onset of fracture due to thermal shock:

$$K_{IC} = \left( A_Q s^c, \Delta T_c + \Theta_{s,m} \Delta T_c \right) \frac{1}{\pi c}$$  \hspace{1cm} [7.58]

Since $\Delta T_c = T_{max} - T_o$ and $\Delta T_c = T_p - T_{max}$ we have:
\[ \Delta T_c = \frac{\left( \frac{K_c}{\sqrt{\pi c}} + \frac{A Q_{x,m} T_o - \Theta_{x,m} T_p}{(A Q_{x,m} - \Theta_{x,m})} \right)}{T_o} - T_o \]  

[7.59]

The value of \( c \) is assumed to be equal to the half-thickness of the average matrix ply. The average matrix ply thickness is obtained if the total matrix ply thickness is divided by the number of matrix plies observed across any composite thickness (~7). Thus, \( c = 0.292/(7 \times 2) \).

Application of [7.59] gives \( \Delta T_c = 315-330^\circ C \), i.e. with an error of 17-20%. If instead of using an average value the central matrix ply with the largest thickness is identified and used for the definition of \( a \), then the prediction is much closer to the experimental value.

7.7. CONCLUDING REMARKS

The theoretical models developed in Chapter 3 for a UD CMC were modified and extended here to the case of 2-D ceramic composite laminates.

It was found that they were able to describe the underlying trends observed in the cracking phenomena and provide satisfactory predictions for the onset of damage under conditions of thermal shock, especially in the case where damage first appears in longitudinal plies. Inclusion of experimental observations regarding material microstructure and properties in the predictive models greatly enhances the accuracy of predictions for the transverse central and matrix plies of the relevant 2-D CMCs.
Chapter 8:

Concluding Remarks
8.1. INTRODUCTION

The behaviour of fibre-reinforced CMCs under conditions of thermal shock was investigated in this study.

Experimental work concentrated on a range of 2-D Nicalon/CAS composites, including cross-ply \((0°/90°)_s\), \((90°/0°)_s\), \((0°/90°)_{3s}\), \((90°/0°)_{3s}\), and PW woven laminates. By employing the water quench test as well as optical and electron microscopy, damage due to thermal shock on these materials was characterised comprehensively. In addition, mechanical testing of thermally-treated samples allowed the effect of thermal shock on the mechanical properties of PW woven Nicalon/CAS to be quantified.

Closed-formed analytical modelling based on strength and/or fracture mechanics concepts was employed to predict the onset of thermal shock fracture, i.e. the critical quenching temperature differential, in both UD and 2-D CMCs.

The conclusions drawn from this study are presented in section 8.2. Subsequently, proposals are made regarding ways in which the present work could be extended and complemented.
8.2. CONCLUSIONS

1. The thermal shock resistance of a range of 2-D Nicalon/CAS CMCs was found to be comparable with that of UD Nicalon/CAS material of similar thickness. In other words, ply configuration was shown to have only a minimal effect. Significant increases in $\Delta T_c$ were observed only after material thickness was reduced beyond a critical value.

2. Damage due to thermal shock always originated at the central plies of each 2-D CMC. Faces of cross-ply CMCs with $0^\circ$ (longitudinal) central plies (i.e. $(90^\circ/0^\circ)_3$, $(90^\circ/0^\circ)_n$) were found to have slightly higher thermal shock resistance than surfaces with $90^\circ$ (transverse) central plies (i.e. $(0^\circ/90^\circ)_3$, $(0^\circ/90^\circ)_n$). In addition, damage due to thermal shock always exhibited a gradient across the material thickness, being higher towards the central region, irrespective of shock severity. Both phenomena were explained by considering the interaction of temperature gradients of adjacent material surfaces.

3. Damage due to thermal shock in 2-D CMCs was in the form of matrix cracking that left the fibres unaffected. Microstructural changes due to high temperature exposure resulted in the occurrence of fibre failures, which were associated with matrix cracks that were not deflected at fibre/matrix interfaces.

4. The orientation and the extent of matrix cracking due to thermal shock was found to depend strongly on the type of each ply (i.e. whether it was a longitudinal, a transverse
or, in the case of woven Nicalon/CAS, a matrix-rich area). Longitudinal and matrix plies contained matrix cracks perpendicular to the horizontal (length) direction, which, upon application of shocks of greater severity, increased in number but were always confined to the surface of the material. Matrix cracks due to thermal shock in transverse plies ran parallel to the horizontal, and increased significantly in length and depth at higher temperature differentials. The difference in behaviour among different plies was explained by considering a stress transfer mechanism acting between fibre and matrix as well as between different plies. By virtue of this mechanism, the energy available for cracking in longitudinal and matrix plies was accommodated by multiplication of the number of cracks, as expected by elements exhibiting 'composite' behaviour. By contrast, transverse plies showed behaviour similar to monolithic ceramics and particulate CMCs, as the energy available for cracking was consumed in extending significantly a small number of surface cracks.

5. Thermal shock was found to reduce the mechanical properties of PW woven Nicalon/CAS. However, the extent of these reductions was small and gradual with increasing shock severity. In addition, mechanical property degradation was not evident at the onset of thermal shock damage. It occurred at higher temperature differentials and coincided with the propagation of matrix cracks in transverse plies deep into the material.

6. As cracks in transverse plies were found to extend significantly and to affect the mechanical properties of the materials, it can be concluded that transverse plies are the weaker elements of the 2-D materials under conditions of thermal shock. Thus, their
incorporation should be avoided or there should be safeguards that would limit crack propagation in these plies. Such conditions seem to be fulfilled in the woven Nicalon/CAS laminate, which was found to experience much smaller damage accumulation with increasing ΔT compared with its cross-ply equivalents. The undulating nature of the plies of this material was identified as the reason for limiting the extent of thermal shock damage in this material.

7. The extent of thermal shock damage was also found to be a function of the dimensions of the material. When one of the dimensions was significantly smaller than a critical value (e.g. the thickness in (0°/90°)ₚ and (90°/0°)ₚ), matrix cracking was always confined to the surface of the material irrespective of ply, and the number of cracks was greatly reduced.

8. Analytical predictions of the onset of thermal shock fracture in UD and 2-D Nicalon/CAS CMCs with satisfactory accuracy were achieved. The analysis considered the anisotropic nature of the applied stress field as well as the presence of residual thermal stresses. A strength-based criterion, combined with a model for the effect of the biaxial nature of shock-induced stresses on the effective value of the interfacial shear stress, were sufficient to allow predictions of the thermal shock resistance of the surface of UD Nicalon/CAS that contained longitudinal fibres, as well as of the central longitudinal plies of (0°/90°)ₚ₃, (0°/90°)ₚ, and PW woven laminates. The method also allowed determination of the heat transfer conditions during fracture. A fracture mechanics-based criterion combined with a modified recent analytical result from the literature was used in the case of the transverse (end) face of UD Nicalon/CAS, as well as for the central
transverse plies of (90°/0°)_{3S}, (90°/0°), and PW woven laminates. The successful application of this approach depended on accurate knowledge of two material parameters, i.e. the relevant fracture toughness and the value of the critical dimension beyond which thermal shock resistance becomes independent of material dimensions. Based on sufficient knowledge of the critical dimension, a method was successfully devised that allowed the effect of material dimensions to be incorporated into predictions of the thermal shock resistance.

8.3. PROPOSALS FOR FURTHER WORK

1. Experimental work should be extended to CMCs of other configurations, notably those that incorporate plies with orientations other than 0° or 90°, e.g. (±45°)_{n} and quasi-isotropic laminates. This would allow a complete picture of the behaviour of fibre-reinforced CMCs under conditions of thermal shock to emerge.

2. Although the behaviour of the CMC used in this investigation can be considered to be generic, at least for dense-matrix CMCs, experimental work should be extended to the more industrially-relevant SiC/SiC laminates manufactured by CVI or melt infiltration and oxide-oxide CMCs. In addition, testing of a CMC for which CTE_{m}<CTE_{f} would be of interest.
3. The analytical work included in this thesis can be developed further in three directions:

(i) Use of a more sophisticated model for the description of the effect of thermal shock on the value of the interfacial shear stress, which would probably allow for the multi-layer nature of present-day interphases and possible effects of Poisson contraction.

(ii) Prediction of the onset of property degradation, which was shown not to coincide with the onset of thermal shock damage. This can probably be achieved by combining some of the other analytical results of Zhao et al. (2000) with a model for the rising resistance of the transverse face to crack penetration in the depth direction (R-curve behaviour).

(iii) Prediction of the onset of cracking in plies of 2-D CMCs other than the central ones. This can be accomplished by combining the gradients of two adjacent faces either analytically or graphically so that the variation of the stress reduction factor across the face thickness can be determined.

4. As analytical modelling of the condition is approaching its limits, it would be interesting to build a computational model, possibly using finite element analysis, based on the same concepts.
References


Bannister M K, Swain M V (1990), 'Thermal shock of a titanium diboride based composite', *Ceram Int*, 16, 77-83


Boccaccini A R (1998), ‘Predicting the thermal shock resistance of fibre reinforced brittle matrix composites’, *Scripta Mat*, 38(8), 1211-1217


Hencke H, Thomas J R, Hasselman D P H (1984), J Am Ceram Soc, 67, 393-


Kagawa Y (1997), ‘Thermal shock damage in a two-dimensional SiC/SiC composite reinforced with woven SiC fibers’, *Comp Sci Tech*, 57, 607-611


Ironside K I (1996), Damage in Woven Ceramic Matrix Composites, PhD Thesis, University of Surrey


