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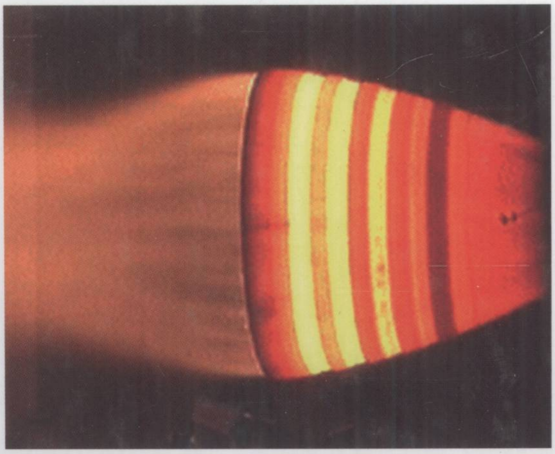
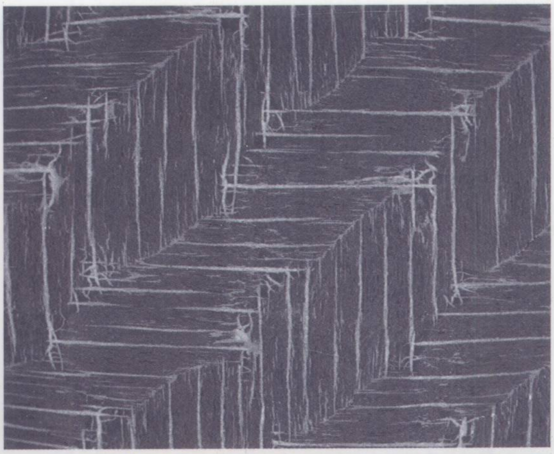
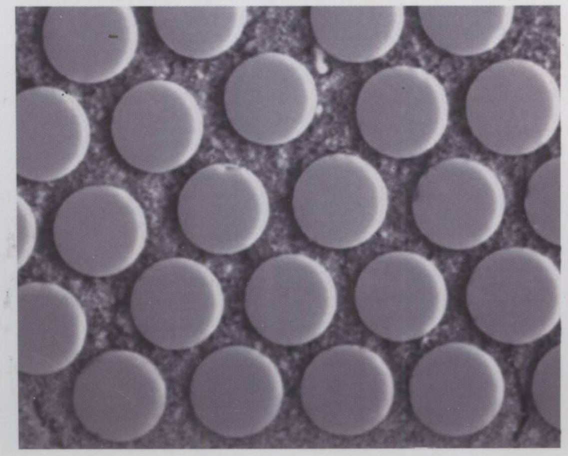
Ceramic Matrix Composites

Fiber Reinforced Ceramics and their Applications







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Foreword

Ceramic Matrix Composites (CMCs) are *non-brittle* refractory materials designed for applications in severe environments (often combining high temperatures, high stress levels and corrosive atmospheres). Compared to other structural materials (such as steels, aluminium or titanium alloys, as well as nickel-based superalloys or monolithic ceramics), they are relatively new, still mostly at the development stage but with a few well-established and promising applications in different high technology domains. We will first summarize here the main features of this new class of materials, then show what impact they have (or could have) on the development of different high tech fields and mention some important historical milestones.

The high strength potential of CMCs is directly related to the use of high strength, high modulus ceramic *fiber reinforcements* of small diameter (typically of the order of 10 μm). Covalent non-oxide fibers, such as carbon or silicon carbide based fibers, are those which presently display the best mechanical properties at high temperature (particularly in terms of creep resistance), but they are oxidation-prone. In this field, the development in Japan of the *SiC-based fiber* family, from the pioneering work of S. Yajima in the mid-1970s, which exhibits better oxidation resistance than carbon fibers, has been an important milestone. Comparatively, refractory *oxide fibers* (such as alumina and alumina-based fibers) display, by their chemical nature, excellent oxidation resistance, good mechanical behaviour at room temperature but creep even at moderate temperatures. As a result, carbon and SiC-based fibers presently are the most commonly used reinforcements in CMCs with a view to applications at high temperatures (say 1200–1800 °C). Small diameter ceramic fibers are extremely fragile and should be properly embedded in a refractory *ceramic matrix* (either oxide or non-oxide), primarily to protect the fibers and to permit load transfer from the matrix to the fibers. Fibers, with a volume fraction of the order of 40–50 %, are still the costly (but key) constituent of CMCs. *Nanoreinforcements*, such as carbon nanotubes or SiC nanofibers, are not presently used in CMCs to a significant extent, owing to handling difficulty, health considerations and cost.

Another key feature of CMCs, compared to polymer or metal matrix composites, is the fact that they are *inverse composites*, which is to say that under load it is the brittle matrix which fails first (in terms of failure strains: $\varepsilon_m^R < \varepsilon_f^R$) and at very low

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strain, typically ≈ 0.1 %. Hence, matrix cracks should be arrested or/and deflected at the fiber–matrix (FM) interface to avoid the early failure of the fibers and thus a brittle failure of the composite. This is achieved through a weakening of the FM-bonding, usually by introducing a thin layer (typically, 50 to 200 nm) of a weak material at the fiber surface, acting as a *mechanical fuse* and referred to as the *interphase*. The most commonly used interphase materials are those with a layered crystal structure (the layers being roughly parallel to the fiber surface and weakly bonded to one another to promote crack deflection). Examples are pyrocarbon (PyC) or hexagonal boron nitride (BN). Historically, they have been formed either (1)insitu in SiC (Nicalon)/glass-ceramic composites by decomposition of the fibers or/and FM interactions during composite high temperature (HT) processing, or (2) deposited by chemical vapour infiltration (CVI) from gaseous precursors, in C/SiC and SiC (Nicalon)/SiC composites. Both these processes were developed in the 1980s. When the FM bonding is weak enough, CMCs behave as *elastic damageable non-linear materials*, which is to say that beyond the proportional limit, the brittle matrix undergoes multiple microcracking under load, the cracks being deflected within (or near) the interphases, the fibers being partly (or totally) debonded and exposed to the atmosphere before the ultimate failure which commonly occurs at a strain of the order of 0.5 to 1.5 %. All these damaging phenomena take place with energy absorption and are responsible for the high toughness of the materials (a very uncommon feature for ceramics). An important milestone in this field was the pioneering work of A. Kelly and his coworkers in the early 1970s.

Improving the *oxidation resistance* of non-oxide CMCs is another important issue, particularly with a view to long duration exposures at high temperatures. In C/SiC and SiC/SiC composites, the weak points are the fibers themselves and the interphase. A first strategy is to use well-crystallized pure SiC fibers, which display good oxidation resistance, rather than SiC-based fibers (which usually contain free carbon and are poorly crystallized) or carbon fibers. In this field, the development of the so-called *stoichiometric* SiC fibers in Japan in the late 1990s was an important step. A second approach is to replace the commonly used PyC-interphase (which undergoes oxidation at temperatures as low as ≈ 500 °C) by *BN-interphase* (which displays a better oxidation resistance, at least in dry oxidizing atmospheres). A third possibility is to use *self-healing* coatings (single layer or multilayer), such as a SiC-coating containing a layer of a boron-bearing compound (such as B_4C) or a ternary Si–B–C mixture. The first role of the coating is to close the open residual porosity of the composite to impede the in-depth diffusion of oxygen. SiC-based coatings do undergo microcracking under load but oxygen diffusing along the microcracks would react with the microcrack wall to form a SiO_2–B_2O_3 healing phase. Finally, the best oxidation protection of SiC-based composites is achieved by extending the concept of crack-healing to the matrix itself which is now a *multilayered matrix* with layers of SiC, layers of sealant formers and layers of mechanical fuses, resulting in a lifetime under load exceeding 1000 hours at 1000–1200 °C. Finally, the use of oxidation-prone interphase can be avoided by utilizing a porous (and hence, relatively weak) matrix, but this approach exposes the fibers to the environment and, thus, it is better suited to oxide/oxide composites.

Processing considerations constitute another important point, the main requirement being that fiber degradation should be absolutely avoided. Hence, low temperature/pressure processing techniques are often favored. This is actually the case for the *CVI-process* and the *PIP-process* (Polymer Impregnation and Pyrolysis) where the matrix precursor is gaseous or liquid, respectively. Both are pressureless techniques involving temperatures of the order of 900–1200 °C. Further, the starting material can be a multidirectional fiber preform, e.g. a 3D-fiber architecture. These techniques yield near net shape parts (which can be of large size and complex shape) but with a relatively high residual porosity (10–15 %). In this field, an important milestone was the transfer of the CVI-process to the plant level in the 1980s for the volume production of C/SiC and SiC/SiC composites. In the so-called *RMI-process* (Reactive Melt Infiltration), the matrix is formed in situ by chemical reaction between a liquid precursor and a preconsolidated fiber preform. For SiC-matrix composites, the matrix precursor is liquid silicon (or a silicon alloy) and the fiber preform is consolidated (e.g. by PIP) with carbon, the former reacting with the latter to form the SiC-matrix. RMI is also a pressureless technique (it is conducted under vacuum). It yields near net shape composites with a low residual porosity, but it involves relatively high temperatures (1400–1600 °C for liquid silicon) with a risk of fiber degradation (unless thick fiber coatings are used), and the matrix usually contains unreacted precursor (such as free silicon). Finally, CMCs can also be fabricated according to *ceramic processing routes*. In the so-called Slurry Infiltration/High Pressure Sintering technique (SI-HPS), the reinforcement is impregnated with a suspension of matrix powder (usually a sol for oxide/oxide or a slurry for non-oxide matrix composites). After drying, the material is densified by sintering at high pressure. For non-oxide covalent ceramic powders, such as SiC-powders that display a poor sintering ability, sintering aids (such as oxide mixture forming eutectics) should be added to the slurry, the sintering conditions ($T = 1800$ °C, $P = 10$–50 MPa for SiC) remaining harsh. As a result, only very stable fibers, such as the stoichiometric SiC fibers prepared at high temperatures can be used. This technique yields composites with almost no residual porosity, high crystallinity and high thermal stability, but it is not suited to the volume production of large parts with complex shapes.

CMCs are expected to have a serious impact on the development of new technologies, as suggested by a few successful current applications. Significant *weight saving* is achieved when heavy superalloys are replaced by high strength and tough C/SiC or SiC/SiC composites in aerojet or rocket engines. Fighters are already equipped with CMC engine nozzles and could have, in the future, CMC combustion chambers. *Lifetimes* of parts working at high temperatures are improved by replacing metal alloys by CMCs. A good example is given by CMC braking systems (C/C for aircraft and C/C–SiC for cars) which exhibit longer lifetimes than their steel counterparts and better wear and friction properties at high temperatures. The use of C/C brakes, first on military fighters and then on civil jumbojet aircraft, on the basis of weight saving, braking performance and safety considerations, as well as that of C/C–SiC brakes on Formula 1 racing cars and sport cars, constitute other important milestones. CMCs can considerably extend the *temperature domain* of use of structural ceramics in many fields, such as jet engines and gas turbines (with higher yields and the possibility of reducing (or even suppressing) cooling requirement), heat exchangers and high temperature chemical engineering. Another promising new field of application could be the use of SiC/SiC composites in high temperature *nuclear reactors* (fission and fusion) for power generation, on the basis of their refractoriness, high temperature mechanical properties (creep resistance), compatibility with neutrons and low residual radioactivity after prolonged exposure to radiation.

It thus appears that CMCs – compared to metal alloys and monolithic structural ceramics – constitute a new class of materials which are well suited to applications in harsh environments. However, they are still very new and will undoubtedly require an intense effort of research. Present applications in different demanding fields suggest that they could have a bright future in the development of high technologies.

Honorary Professor, Bordeaux 1 University *R. Naslain*

Preface

Ceramic Matrix Composites (CMCs) represent a relatively new class of quasiductile ceramic materials. They are characterized by carbon or ceramic fibers embedded in ceramic matrices (oxide or non-oxide) with comparatively low bonding forces between the fibers and the matrix. These weak intefaces, in combination with a porous and/or microcracked matrix, result in composite materials which differ from all other structural materials or composites and show some outstanding properties. Their strain-to-failure is up to one order of magnitude higher than in monolithic ceramics and their low densities result in mass-specific properties which are unsurpassed by any other structural material beyond 1000 °C.

From their research beginnings about 40 years ago, the demands of space technology played the decisive role in the development of CMCs. Hot structures of limited lifetime (e.g. thermal protection systems, nozzles) in aerospace and military applications have been developed in different countries. In recent years, civil and terrestrial requirements became the driving forces and properties and manufacturing processes were consistently improved to transfer CMCs from niche applications to broader markets. Due to their high thermal stability and good corrosion and wear resistance, these composite materials are of increasing interest for long-term applications and damage-tolerant structures in different industrial sectors like ground transportation (e.g. brake and clutch systems), mechanical engineering (e.g. bearings, ballistic protections), and power generation (e.g. burners, heat exchangers).

The goals of further research and development are focused on improvements in the thermal and oxidative stability of the reinforcing fibers and on a considerable reduction of the processing costs. Reasonable costs for series productions are expected by using innovative continuously operated furnaces as they already exist for other structural (monolithic) ceramics. Also, new forming processes for the manufacture of green bodies and new hybrid processes of high reliability are necessary. Beside these fabrication approaches, novel precursors for cheaper ceramic fibers and improvements in the thermomechanical properties of short-fiber reinforced CMCs are key factors to develop CMC materials for wider application.

This textbook provides a comprehensive overview of the current status of research and development on CMCs. It presents data tables, process descriptions, and field reports, giving special emphasis to applications relevant to the respective

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topics. In this regard, the textbook begins with two chapters on fibers and textile preforms for the reinforcement of ceramic matrix composites, followed by the description of the fiber/matrix interfacial domain of CMCs. In this chapter, data on interfacial characteristics and techniques to measure these characteristics are provided. This is followed by four chapters describing the most important processes used to manufacture non-oxide CMC materials currently. This includes the manufacture of carbon/carbon, the melt infiltration of silicon into carbon/carbon composites, as well as the Polymer Infiltration and Pyrolysis (PIP) and Chemical Vapor Infiltration (CVI) processes. Two chapters on oxide-CMCs with dense and porous matrices, which are promising materials particularly in combustion environments, conclude the processing part of the book.

The following two chapters describe the microstructural modelling and testing of CMCs using different models and methods. These topics are of special interest for designing structural parts and predicting their lifetime, for example by integrating non-destructive testing methods. As all fabrication approaches have certain limitations in terms of size and shape, the following two chapters deal with machining and joining techniques to achieve CMC structures of high integrity. This is followed by chapters providing practical experiences of the application of CMC materials under extreme thermal as well as corrosive conditions. Hot structures in spacecraft and aircraft show the tremendous progress which has been achieved with respect to re-usability and lifetime of CMC structures over the last 20 years. The current stage of development in using SiC/SiC composites as future structural materials in nuclear applications is described in a separate chapter. The most attractive volume market for CMCs currently is the topic of the last chapter. Test results and experiences with high-performance brake and clutch systems equipped with disks and pads of C/SiC composites are presented, demonstrating their superior tribological behaviour in automotive and other applications.

I would like to thank all the authors for their valuable and timely contributions. I am grateful to Roger Naslain, one of the pioneers of ceramic matrix composites, for writing the Foreword of this book. Furthermore, I would like to thank Waltraud Wüst and her team from Wiley-VCH and Petra Jelitschek as well as Angelika Schwarz from my research teams in Bayreuth for their help and cooperation during the publication process.

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