Blast furnace life has also been increased through the use of SiC-based refractories.

2.4 CERAMIC MATRIX COMPOSITES

Ceramic matrix composites (CMCs) combine reinforcing ceramic phases with a ceramic matrix to create materials with new and superior properties. In ceramic matrix composites, the primary goal of the ceramic reinforcement is to provide toughness to an otherwise brittle ceramic matrix (Fig. 2.19). Fillers can also be added to the ceramic matrix during processing to enhance characteristics such as electrical conductivity, thermal conductivity, thermal expansion, and hardness. The desirable characteristics of CMCs include high-temperature stability, high thermal-shock resistance, high hardness, high corrosion resistance, light weight, nonmagnetic and nonconductive properties, and versatility in providing unique engineering solutions. The combination of these characteristics makes ceramic matrix composites attractive alternatives to traditional processing industrial materials such as high alloy steels and refractory metals. For the processing industry, related benefits of using ceramic composites include increased energy efficiency, increased productivity, and regulatory compliance. Key barriers to the broad application of ceramic matrix composites include the lack of specifications, databases, attachment concepts, in-service repair methodology, high cost, and scale-up.

Ceramic matrices can be categorized as either oxides or nonoxides and in some cases may contain residual metal after processing. Some of the more common oxide matrices include alumina, silica, mullite, barium aluminosilicate, lithium aluminosilicate and calcium aluminosilicate. Of these, alumina and mullite have been the most widely used because of their in-service thermal and chemical stability and their compatibility with common reinforcements.

Although oxide matrices are often considered more mature and environmentally stable, nonoxide ceramics—with superior structural properties, hardness, and, in some environments, corrosion resistance—are rapidly entering the marketplace. Some of the more common nonoxide ceramics include SiC, Si_3N_4, boron carbide, and AlN. Of these, SiC has been the most widely used, with AlN of increasing interest where high thermal conductivity is required and Si_3N_4, where high strength is desired.

Ceramic reinforcements are available in a variety of forms. Early ceramic composites used discontinuous reinforcements which, when combined with ceramic matrices, could be formed using conventional monolithic ceramic processes. Some of the more common discontinuous reinforcements include whiskers, platelets, and particulates having compositions of Si_3N_4, SiC, AlN, titanium diboride, boron carbide, and boron nitride. Of these, SiC has been the most widely used because of its stability with a broad range of ceramic oxide and nonoxide matrices. Discontinuous oxide ceramic reinforcements are less prevalent because of their incompatibility with many common ceramic matrices.

Modern day composites, which use the fiber in a continuous form to better optimize the structural properties, generally require more costly manufacturing processes. Continuous fiber is available in both monofilament and multifilament tow forms, with multifilament tow fiber being lower in cost on a per-pound basis and, in many cases, easier to process into complex shapes. Some of the more common continuous reinforcements include glass, mullite, alumina, carbon, and SiC. Of these, SiC fibers have been the most widely used because of their high strength, stiffness and thermal stability. Common trade names for silicon carbide fiber include Nicalon™, Hi-Nicalon™, SCS, Sylramic™, and Tyranno. For applications where temperature is lower (< 100°C) or exposure times limited, mullite fibers have been the most widely used because of their lower cost. Nextel is a common trade name for both mullite and alumina fiber. Continuous ceramic fibers are of increasing interest because of their ability to provide pseudo-ductile characteristics to otherwise brittle ceramic materials (Fig. 2.19).

Continuous ceramic fibers become especially important in large structures where the probability of processing- or in-service-induced flaws increases with part size and could result in catastrophic failure. Due to the limited commercial demand, certain grades of continuous ceramic fiber are currently available for $500–$5,000 / lb. As demand increases, continuous ceramic fiber cost is projected to approach $50–$100 / lb. When combined with a ceramic matrix, composite part cost could ultimately reach $200–$400 / lb, which then becomes cost competitive with high alloy steels on a life-cycle cost basis and with refractory metal alloys on both an acquisition and life-cycle cost basis. Today ceramic composite part costs exceed $1,000 / lb when using continuous fiber reinforcement. Discontinuous reinforced composite part cost is currently $7–$50 / lb.

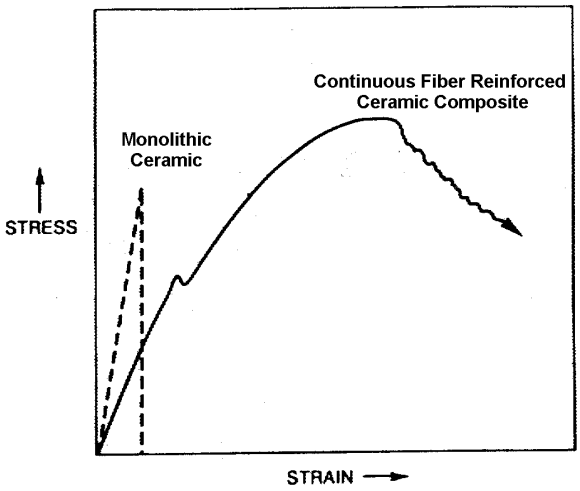

Fig. 2.19. Comparison of failure modes for monolithic ceramics and CFCCs.

Production of ceramic composites in the United States is dominated by a few large firms while small companies are involved in the manufacture of ceramic composites for niche markets. A representation of suppliers of discontinuous and continuous fiber–reinforced ceramic composites is shown in Table 2.6. In 1994, U.S. company shipments for ceramic composite materials totaled about $475 million, of which a majority was for discontinuous reinforced composites. Japanese companies are estimated to control 50% of the world market in advanced ceramics, which includes ceramic composites. Major markets for ceramic composites include cutting tools, aerospace and military, engines, wear and industrial, bioceramics, and energy related. Industrial processing applications currently make up a small fraction of the overall market but the market is growing rapidly as technology and economic barriers are overcome and user confidence builds.

The Department of Energy (DOE) has supported the development of ceramic composites from their inception because of the perceived benefits to energy efficiency, which have since been demonstrated. Early success by DOE occurred with the development and successful commercialization of whisker-reinforced ceramic composites for the cutting tool industry. In 1991, a comprehensive program was started by DOE Office of Industrial Technology to support the development of CFCCs with the goals of developing, in U.S. industry, the primary processing methods for the reliable and cost-effective fabrication of CFCCs for use in industrial applications. Specific goals include the following:

- Develop and demonstrate, at pilot scale, efficient and economic methods of processing CFCC components that are representative of the geometries and properties required to meet the performance requirements of potential end-use applications.
- Develop the necessary supporting technologies to design, analyze and test the materials processing methods and components.
- Provide representative components and relate technologies as inputs to end-user application development programs.

The remaining discussion focuses on ceramic composites considered important to the industrial processing community.

Table 2.6. Representative ceramic composite suppliers

Continuous fiber ceramic composites	Discontinuous reinforced ceramic composites
3M Company	Advanced Refractory Technologies, Inc.
Synterials, Inc.	ALANX Wear Solutions, Inc.
B. F. Goodrich	Cercom
Composite Optics, Inc.	DuPont Lanxide Composites, Inc.
Dow Corning Corporation	Greenleaf Corporation
DuPont Lanxide Composites, Inc.	
McDermott Technology, Inc.	
Techniweave, Inc.	
Textron Systems Corporation	

Discontinuous Reinforced Ceramic Composites

Discontinuous reinforced ceramic composites are produced using processes originally developed for monolithic ceramics. Processing methods commonly used include slip casting or injection molding followed by sintering to full density in a high-temperature-capable furnace. The shaping and sintering processes can also be combined using unidirectional hot pressing or hot isotatic pressing. Net or near-net shape processing can be achieved with final machining often limited to satisfying high-tolerance dimensions or surface finishes. An example of the material microstructure showing the matrix and uniformly distributed discontinuous reinforcement is shown in Fig. 2.20.

Silicon carbide whisker reinforced alumina (SiC_w / Al_2O_3) composite cutting tools have been commercialized by several vendors in the United States (Fig. 2.21). These tools have found an important niche in difficult-to-machine materials and where there is a desire for high machining rates for these materials. Examples can be found in the machining of nickel-based superalloys in the gas turbine industry. A four axis machine retrofit with SiC_w / Al_2O_3 cutting tools was used to reduce machining time of a specific operation from 3 h to 18 s. Factors that allow the increase in productivity include not only increases in cutting speeds by up to a factor of ten, but also lower wear rates and more predictable tool failure times. SiC_w / Al_2O_3 is also being used as the wear components in ceramic / metal hybrid tooling and dies in the canning industry. Key benefits include increased tool life, reduced down time, improved surface finish, lower maintenance cost, and increased production rates. SiC_w / Al_2O_3 is presently used in cupping, drawing, ironing and can necking tools.

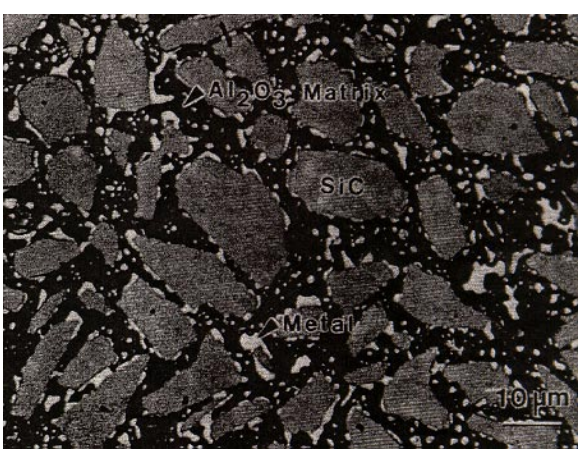

Fig. 2.20. Typical microstructure for a discontinuous reinforced ceramic composite (SiC/Al_2O_3-Al). ***S****ource:* Lanxide Corporation, Newark, Del.

Fig. 2.21. SiC_w/Al_2O_3 cutting tools. ***S****ource:* Greenleaf Corporation, Saegertown, Pa.

Discontinuous reinforced ceramics are being used in the mining and abrasives industry, where use of these ceramics has reduced downtime usually needed for replacing worn parts. The largest application has been chute liners. One product, consisting of 75% SiC + 15% Al_2O_3 + 10% Al, provides excellent resistance to dry erosion, slurry erosion, sliding abrasion, thermal shock, and chemical attack. Similar high-wear applications where discontinuous reinforced ceramics have shown improvements include liners for cyclones, pipes, and pump housings (Fig. 2.22).

Discontinued reinforced ceramics are also being considered for use in a high-pressure (1035-kPa) heat exchanger designed for use in steam reforming of methane. Advantages of ceramic composites for this application include higher temperature capability, reduced fouling, increased energy efficiency, reduced emissions and reduced reformer weight and size. Of the materials testes, a product composed of SiC + Al_2O_3 +Al proved the least vulnerable to steam attack at elevated temperatures.

Continuous Reinforced Ceramic Composites

Ceramic composites containing continuous fiber reinforcements must be processed by

Fig. 2.22. Examples of discontinuous reinforced industrial wear parts. *Source:* ALANX Wear Solutions, Inc., Newark, Del

methods that accommodate the continuous nature of the reinforcement. Typically, processing involves the formation of a fiber preform that contains an interface coating applied by chemical vapor deposition (CVD) or a particle-filled slurry process followed by impregnation with a second particle-filled slurry mix, preceramic polymer, precursor gases, molten metal, or other raw material that converts to a ceramic matrix when heated. The interface is a very thin layer (< 5 µm total thickness), can be multiple layers to achieve the desired result, and is applied to the individual ceramic filaments. The interface serves as protection for the fibers during matrix processing and as a source of debonding during crack propagation in the brittle ceramic matrix. A typical CFCC microstructure shown in Fig. 2.23 illustrates the matrix, fiber, and interface phases.

Fig. 2.23. Typical microstructure of a SiC/SiC CFCC. *Source*: AlliedSignal Composites, Inc., Newark, Del.

SiC Matrix CFCCs

SiC matrix CFCCs have been successfully demonstrated in a number of applications where a combination of high thermal conductivity, low thermal expansion, light weight, and good corrosion and wear resistance is desired. SiC matrix CFCCs—the most mature of the available CFCCs—can be fabricated using a variety of processes, fibers, and interface coatings. Fibers widely used for industrial applications where long life is desired include SiC or mullite. Processes available to fabricate SiC matrix CFCCs and the matrix composition formed include chemical vapor infiltration (SiC), polymer infiltration (SiCN, SiC), nitride bonding (Si–SiC-Si_3N_4), and melt infiltration (Si-SiC). The interface coating can be either carbon or boron nitride with a protective overcoat of SiC or Si_3N_4.

The material properties of SiC matrix CFCCs are ultimately determined by the combination of fiber, process and interface coating selected with typical values shown in Table 2.7

The CVD process is illustrated in Fig. 2.24. In this example a ceramic interface and the SiC matrix are both formed by deposition from a gas precursor at elevated temperature. Multiple cycles of the matrix-forming process are required to achieve the desired final density, with each cycle taking up to a week in many cases. The number of cycles varies from 2 to 5, depending on the part geometry and size.

The infiltration of a fiber preform with preceramic polymer or a particle slurry is illustrated in Fig. 2.25. In this example, the interface is applied onto the fiber, cloth, or preform by CVI or particle-filled slurry processes before coating with a matrix-forming second slurry or

Table 2.7. Typical material properties for a SiC matrix CFCC

Density	2.1 g/cm^3
Tensile strength	262 MPa
Tensile modulus	96 GPa
Strain to failure	0.4%
Compressive strength	450 MPa
Shear strength	34 MPa
Thermal expansion	2.7×10^{-6}/°C
Thermal conductivity	1.32 W/m°C

Source: Dow Corning Corporation

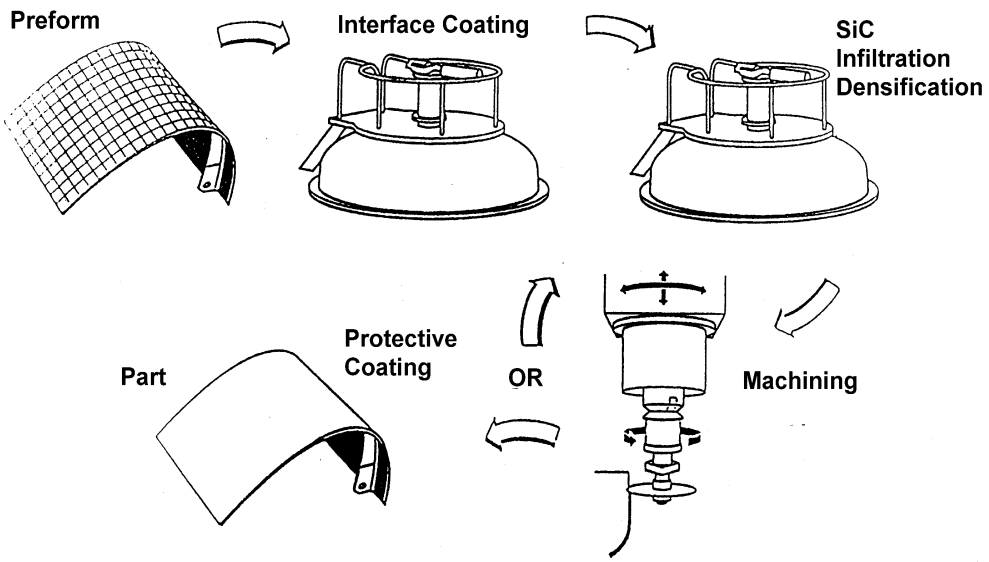

Fig. 2.24. Flowchart for the processing of CFCCs by chemical vapor deposition.

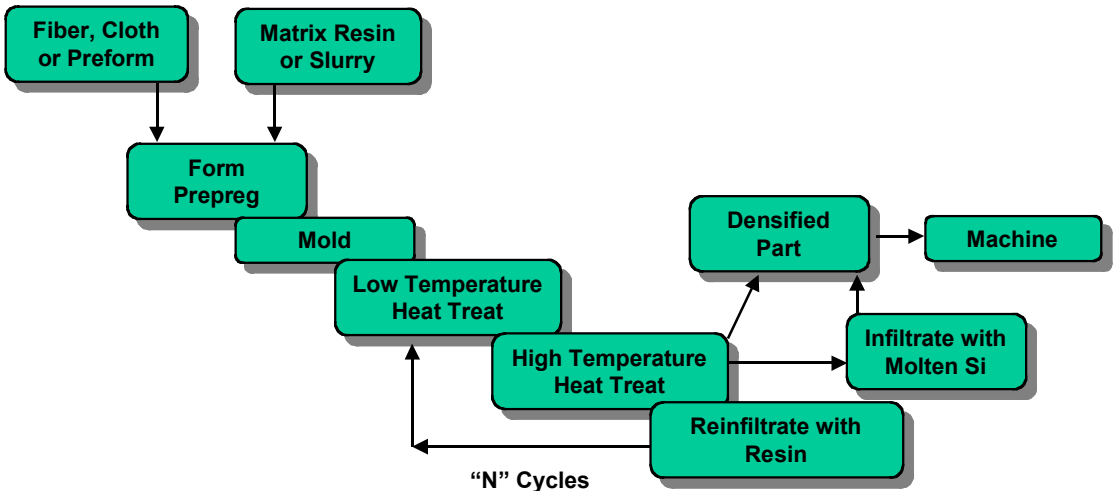

Fig. 2.25. Flowchart for the processing of SiC CFCCs by resin infiltration, melt infiltration, or nitridation.

preceramic polymer. Once coated with a polymer or slurry, the shape is formed and heat treated at low temperatures to rigidize the shape. The rigidized shape is then heat treated at elevated temperatures to convert the polymer to a ceramic matrix or bond particles in the ceramic-filled slurry. For preceramic polymers, multiple cycles are required to achieve the desired final density, with each cycle taking one to three days. The number of cycles used varies from 4 to 15 depending on the polymer and the desired final density. When using particle-filled slurries to form the matrix, porosity remaining after rigidizing the preform is either filled with molten silicon metal to form a matrix of Si-SiC or Si contained in the slurry reacts during high temperature heat treatment in nitrogen to form a matrix of Si–SiC-Si_3N_4. Variants of the melt infiltration process can also be used in which the molten silicon reacts with carbon present in the matrix to form additional silicon carbide.

Shaping to near-net dimensions occurs during preforming with final machining limited to high tolerance attachments to avoid surface fiber damage. Additional machining may be required for complex shapes and is generally performed early in the process when the composite is less dense and easier to machine. External coatings can be used to enhance environmental resistance with some of the more common environmental barrier coatings including mullite, SiC, and Si_3N_4.

Shape- and size-making capability can also be limited by the combination selected due to restrictions on available manufacturing facilities and limitations inherent in the fabrication processes themselves. Manufacturing facilities for CFCCs currently exist for parts as large as 1.5 m in diameter and 2.5 m long. Many of the technical barriers for producing SiC matrix CFCCs have been overcome, with the key remaining barrier being high cost. Applications for which SiC matrix CFCCs are being considered are summarized in Table 2.8 and are shown in Figs. 2.26–2.31. The applications are in various stages of development, with gas-fired radiant burner screens and gas turbine engine combustors well along in the product development cycle and the nearest to commercialization. SiC matrix CFCCs have operated in gas turbine engine combustor liners for 1,000 h and radiant burner screens for 10,000 thermal cycles without failure.

Oxide Matrix CFCCs

Alumina and mullite matrix CFCCs have been successfully demonstrated in applications where a high tolerance to salt corrosion, molten glass or oxidation is required in combination with the high toughness, light weight, and high-thermal-shock resistance characteristic of CFCCs. Fibers generally used for industrial applications requiring long life include SiC, mullite, or alumina and mullite matrix CFCCs. Processes available to fabricate alumina and mullite matrix CFCCs, and the matrix compositions formed, include sol-gel (Al_2O_3, Al_2O_3, SiO_2), directed metal oxidation (Al_2O_3 + Al), and chemical bonding ($AlPO_4$). The interface coating varies widely and in some cases is not required, resulting in improved thermal stability but reduced interlaminar properties.

Processes used to fabricate alumina and mullite matrix CFCCs are generally lower temperature, which results in simplified scale-up, virtually unlimited size- and shape-making capability, and reduced cost. The sol-gel and chemical bonding processes are similar to the preceramic polymer process previously described in Fig. 2.25. The directed metal oxidation processes is illustrated in Fig. 2.32. In this example, the interface is applied onto the fiber, cloth, or preform by CVI processes after forming to shape because the overall thickness of the interface makes further shaping difficult once applied. The rigidized shape containing the fiber and interface coating is next exposed to molten aluminum metal at elevated temperatures in a reactive atmosphere of oxygen to form a matrix of Al-Al_2O_3. Depending on the application, excess metal can be removed from the microstructure by a additional processing.

Material properties of alumina and mullite CFCCs are ultimately determined by the combination of fiber, process, and interface coating sizing selected, with typical values shown in Table2.9.

Applications for which alumina or mullite matrix CFCCs are being considered are summarized in Table2.10 and are shown in Figs.2.33–2.35. The applications ar e in various stages of development, with hot gas filtration being well along in the product development cycle and the nearest to commercialization.

Table 2.8. Applications being considered for SiC matrix CFCCs

Application	Conditions imposed on SiC matrix CFCC	Benefits demonstrated or perceived by the customer
Hot-gas recirculating fan	High mechanical stresses, high temperature	Increased life and temperature capability
Heat exchanger	High temperature, high thermal stresses, highly erosive and hot corrosion	Higher temperature capability and life
Gas-fired radiant burner screen	High temperature, high thermal stresses	Increased life and thermal output
Gas-fired radiant burner tubes	High temperature, high thermal stress	Increased life and temperature capability
Gas turbine engine combustion liners and tip shrouds	High temperature, hot corrosion	Reduced emissions and increased efficiency resulting from reduced cooling air, higher temperature capability
Hot-liquid filtration	Harsh chemical environment, high temperature	Increased life, higher temperature capability
Canned motor pump containment shell	Harsh chemical environment	Increased electrical efficiency through reduced eddy current drag
Gas-fired melting immersion burner tube	Harsh molten metal environment, high thermal shock	Reduced contamination, increased efficiency, improved heating uniformity
Furnace pipe hanger	High temperature, high mechanical stresses	Longer life, higher temperature capability, reduced supporting structure

Fig. 2.26. SiC matrix CFCC hot gas recirculating fan manufacturing using chemical vapor infiltration. *Source:* AlliedSignal Composites, Inc., Newark, Del.

Fig. 2.27. SiC matrix CFCC radiant burner screen manufactured using chemical vapor infiltration. *Source:* AlliedSignal Composites, Inc., Newark, Del.

Fig. 2.28. SiC matrix CFCC gas turbine engine combustor liners manufactured using chemical vapor infiltration and tip shroud manufactured using melt infiltration. ***S****ources:* AlliedSignal Composites, Inc., Newark, Del., and General Electric Power Systems, Schenectady, NY.

Fig. 2.29. SiC matrix CFCC filters manufactured using chemical vapor infiltration. ***S****ource:* AlliedSignal Composites, Inc., Newark, Del.

Fig. 2.30. SiC matrix CFCC immersion burner tubes manufactured using nitridation. ***S****ource:* Textron Systems Corporation, Lowell, Mass.

Fig. 2.31. SiC matrix CFCC furnace pipe hangers manufacured using preceramic polymer infiltration. ***S****ource:* Dow Corning Corporation, Midland, Mich.

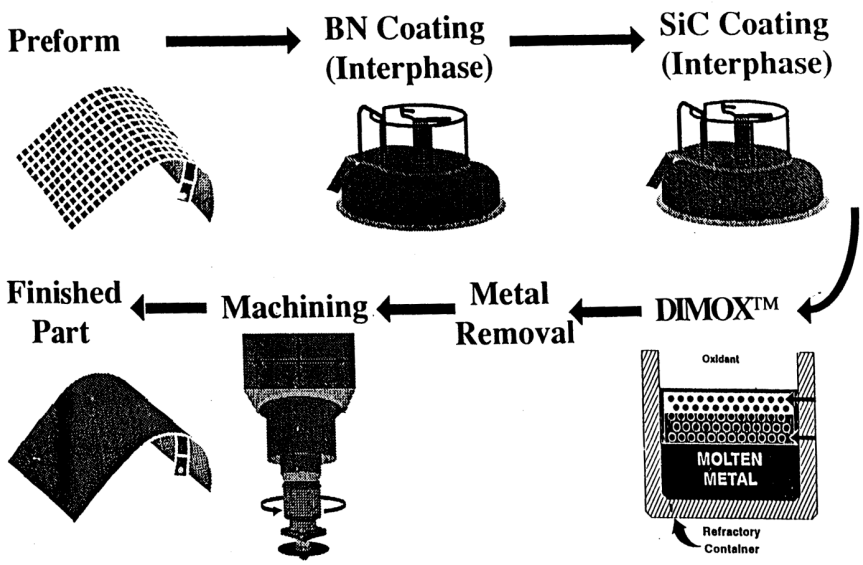

Fig. 2.32. Flowchart for the processing of Al_2O_3-Al Matrix CFCCs by direct metal oxidation.
Source: Textron Systems Corporation, Lowell, Mass.

Table 2.9. Typical material properties for an alumina matrix CFCC

Density	2.82 g/cm^3
Tensile Strength	365 MPa
Tensile Modulus	124 GPa
Strain to Failure	0.35%
Compressive Strength	270 MPa
Thermal Expansion	8.5×10^{-6}/°C
Shear Strength	15.2 MPa

Source: Composite Optics, McDermott Technology, Inc., Alliance, Ohio, and Lynchburg, Va.

Table 2.10. Applications being considered for alumina and mullite matrix CFCCs

Application	Conditions imposed on SiC matrix CFCC	Benefits demonstrated or perceived by the customer
Thermophotovoltaic burner/emittor	High thermal shock, high temperature, surface combustion	Increased efficiency, multifuel capability, maximum on/off response, increased life
Heat exchanger	High temperature, high thermal stresses, highly erosive and hot corrosion	Higher temperature capability and life
Hot-gas filtration	Harsh chemical environment, high temperature	Increased life, higher temperature capability
Gas turbine engine combustion liners and shrouds	High temperature, hot corrosion	Reduced emissions and increased efficiency resulting from reduced cooling air, higher temperature capability
Burner stabilizer	High corrosion	Longer life

Fig. 2.33. Alumina matrix CFCC burner stabilizer ring. ***S****ource:* McDermott Technology, Inc., Alliance, Ohio, and Lynchburg, Va.

Fig. 2.34. Alumina matrix CFCC heat exchanger headers. *Source:* AlliedSignal Composites, Inc., Newark, Del.

2.5 SUMMARY

In summary, advanced monolithic ceramics are being used throughout the industrial processing industry and have demonstrated superior performance to conventional materials at an affordable price. As user confidence continues to grow and as energy savings, increased productivity, and reduced maintenance are confirmed, the need has emerged for advanced ceramics with improved toughness (also known as ceramic composites). Discontinuous reinforced ceramic composites have partially filled this need, but their application is limited in both size and geometry, and their toughness is less than desired for a risk-adverse industry. Continuous fiber reinforced ceramic composites are viewed as the ultimate solution with many applications rapidly becoming commercially viable.

The following chapters discuss industrial processing applications where advanced ceramics are contributing today or could become contributors with additional product development. Industries considered are those identified by DOE's Office of Industrial Technologies as being the largest users of energy, and they include steel, aluminum, metal casting, glass, forest products, and chemical/petrochemical. Included in the discussion on potential opportunities for advanced ceramics is an overview of the character of each industry and the industrial processes currently used.

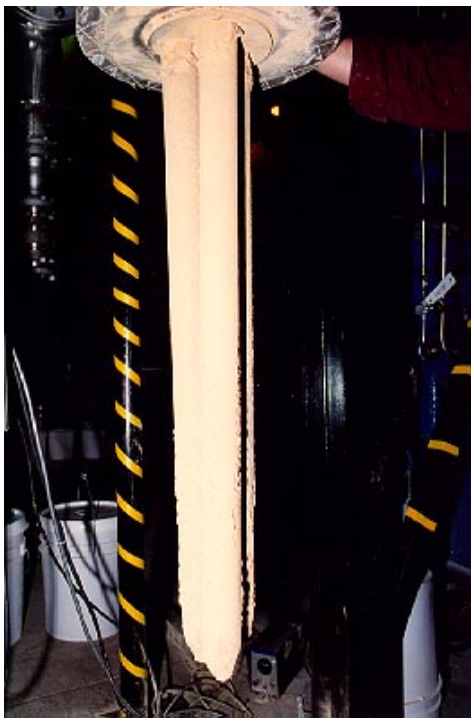

Fig. 2.35. Alumina matrix CFCC hot-gas filter. *Source:* McDermott Technology, Inc., Alliance, Ohio, and Lynchburg, Va.

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Gregory S. Corman, General Electric Corporate Research and Development
Phillip A. Craig, AlliedSignal Composites, Inc.
William Darden, Textron Systems Corporation
Richard E. Engdahl, Synterials, Inc.
Richard Goettler, McDermott Technology, Inc.
William F. Grant, Textron Systems Corporation
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Brian Seegmuller, Coors Ceramics Company
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