Chapter 16 Ceramic Matrix Composites (CMCs) for Aerospace Applications

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Abstract Ceramic materials have excellent properties, but are brittle and the strengths are highly variable. Particulate reinforcements give isotropic properties, but only marginal improvement in toughness. Continuous reinforcements improve the ceramic materials both in terms of fracture toughness as well as strength variability. The processing of ceramic matrix composites and improving the required properties with the available reinforcements is an emerging technology that is finding new critical applications.

Keywords Ceramic matrix composites \cdot Processing \cdot Properties \cdot Applications

16.1 Introduction

Ceramic materials, by virtue of their inherently high melting points, low density, good chemical inertness, good stiffness and high hardness, are considered to be candidates for extending performance limits beyond those offered by metallic materials. However, their inherent brittleness and poor strength reliability have inhibited the widespread use of ceramic materials for structural applications. In an effort to overcome these basic problems, considerable progress has been made during the past four decades in both compositional and microstructural design of ceramics, and this is well-summarized in the monograph edited by Professor Walter Krenkel [\[1](#page-16-0)].

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The deployment of composite technology is the principal methodology to achieve vastly improved mechanical properties and structural integrity, as is unequivocally advocated by several handbooks/monographs/edited volumes $[1-17]$ $[1-17]$ $[1-17]$ $[1-17]$ as well as overviews [\[18](#page-17-0)–[38](#page-17-0)] and comprehensive technical papers [[39](#page-17-0)–[59](#page-18-0)]. All of these data sources point to the fundamental fact that three major entities, namely the matrix, reinforcement and the interface are responsible for determining the characteristics and properties of a particular composite material.

The matrix is the major continuous phase in which the reinforcement is uniformly distributed. Depending upon the type of matrix, composite materials can generally be classified into four different categories, namely polymer matrix composites (PMCs), ceramic matrix composites (CMCs), metal matrix composites (MMCs) and intermetallic matrix composites (IMCs). This chapter discusses the second category, CMC materials.

16.2 CMC Constituents

16.2.1 Ceramic Matrices

Major advantages of ceramic matrices over polymer and metal matrices are their higher melting points, higher hardness, lower coefficient of thermal expansion (CTE) and better chemical inertness. The range of ceramic matrices is vast. Oxides like Al_2O_3 , MgO, ZrO₂, Mullite, and Spinel, and non-oxides like SiC, Si₃N₄, TiC, B_4C and Ti B_2 belong to this category. High temperature structural silicides like $MoSi₂, WSi₂$ and TiSi₃ are also being considered as potential matrix materials.

Even though the major emphasis in CMCs centres on the comparatively cheaper and abundant oxide matrices $(Al₂O₃$ in particular), other potential matrix systems, e.g. Mullite, SiC, SiAlON and MoSi₂ are also being examined for their potential as structural materials. Table [16.1](#page-2-0) provides comparative thermal stability data for some common oxides and non-oxides [\[4](#page-16-0)], since such data primarily decide the choice of CMCs for high temperature applications in different environments.

Discontinuously reinforced ceramic matrix (DRCM) composites are CMCs reinforced by ceramic particulates, platelets, whiskers or short fibres. The reasons for success of these composites as structural ultrahigh temperature (UHT) materials are their remarkable isotropic properties, amenability to conventional ceramic powder processing techniques, and the availability of comparatively low-cost and high-volume production. The physical and mechanical properties of various common ceramic matrix materials are compiled and presented in Table [16.2](#page-2-0) [[7\]](#page-16-0).

A series of early investigations indicated that continuous fibre reinforcements in alumina can impart high stiffness and better tensile properties as compared to most other CMCs [\[2](#page-16-0), [6](#page-16-0), [9](#page-16-0), [22](#page-17-0)–[24](#page-17-0), [30](#page-17-0), [34](#page-17-0), [48](#page-18-0), [54\]](#page-18-0). However, this approach has several limitations: expensive and complicated processing techniques; problems related to oxidation and reaction of fibres with the matrix material at elevated temperatures;

Properties/materials	Melting point $(^{\circ}C)$	Short term maximum use temperature $(^{\circ}C)$	Maximum use temperature $(^{\circ}C)$ in air for no significant creep or loss of properties	
Oxides		(In air)		
Alumina	2060	1950	1500	
Zirconia (stabilized)	2680	2300	1500	
Mullite	1810	1700	1400	
Spinel ($MgA1_2O_4$)	2135	1800	1400	
Magnesia	2800	2000	1400	
Non-oxides		(In inert atmosphere)		
Silicon carbide	2400 (dissociates)	2000	1450	
Boron carbide	2420	1300	800	
Titanium carbide	3050	1400	750	
Silicon nitride	1800 (dissociates)	1500	1200	
Titanium diboride	2880	2400	1000	

Table 16.1 Comparative thermal stability data for some of the most common ceramic materials $[4]$ $[4]$

Table 16.2 Comparative properties of various ceramic matrix materials [[7\]](#page-16-0)

Material	Density (g/cm^3)	Melting point (° \mathbf{C}	Hardness (GPa)	Fracture toughness $(MPa\sqrt{m})$	Thermal conductivity $(W/m^{\circ}K)$ at 1273 ^o K	Coefficient of thermal expansion $\times 10^{-6}$ (/ \circ K) at 1273° K	Electrical resistivity Ω m at $298^\circ K$
$A1_2O_3$	3.97	2050	18	3.2	6	8	$>10^{15}$
MgO	3.24	2800	12	2.6	τ	9	10^{12}
ZrO ₂	6.10	2720	12	8.0	3	13	10^{12}
Mullite	2.80	1810	13	2.1	5	6	10^{13}
SiC	3.21	$2400^{\rm a}$	21	4.2	40	5	\approx 1
Si ₃ N ₄	3.20	1900^{a}	16	5.1	15	3	10 ⁴
TiB ₂	4.50	2800	22	5.2	25	6	10^{-5}
MoSi ₂	6.25	2100	12	4.1	20	8	2×10^{-5}
TiC	4.90	3050	25	4.2	30	\mathbf{Q}	10^{-4}
AIN	3.26	2300	13	2.7	50	6	2
TiN	5.40	3090	18	4.1	30	9	5×10^{-5}
BN	3.50	3000^a	32	3.1	35	6	10^{10}
B_4C	2.50	2420	25	3.2	15	6	0.5

a Dissociates

poor resistance of polycrystalline fibres to creep deformation; considerable degradation of the continuous polycrystalline fibres due to grain growth at temperatures above 1250 °C; and fragmentation behaviour of the polycrystalline fibres during high pressure—high temperature consolidation processing, such as hot pressing. Because of these inherent problems, and also because discontinuous reinforcements are easier to process and more available, discontinuous reinforcements are preferred to continuous reinforcements for toughening brittle ceramic matrices.

Although various alumina matrix composites have been developed over the past few years, major attention has been focussed on discontinuously reinforced alumina matrix (DRAM) composites using SiC fibres. Because of the potential of this $A1_2O_3-SiC_w$ system, it has become one of the most important members of the DRAM composite family. Some others are $A1_2O_3-SiC_p$ (particulate), $A1_2O_3$ -TiB, $A1_2O_3$ -TiC, $A1_2O_3$ -TiN, $A1_2O_3-SiC_{p1}$ (platelet), $A1_2O_3-B_4C$ and $A1_2O_3$ -Graphite systems.

16.2.2 Ceramic Reinforcements

Most of the ceramic reinforcements based on oxides, carbides, nitrides and borides have already been found to be suitable reinforcements for incorporation into brittle ceramic matrices. Amongst these reinforcements, SiC is of the highest interest as a reinforcement material because of its relatively high modulus, low coefficient of thermal expansion (CTE), low density, good hardness, and availability in various forms ranging from different sizes of particulates to fine single crystal whiskers, multi-filament tows and relatively coarse monofilaments.

Reinforcements are generally available in the form of continuous fibres, short fibres, whiskers, platelets and particulates, see Fig. 16.1. Continuous fibres are

Fig. 16.1 Schematic diagram showing different types of CMCs—namely, particulate, discontinuous and continuous fibre-reinforced CMCs

unidirectionally oriented polycrystalline material and are available in the form of monofilaments or multifilaments. Monofilaments, such as Boron, Borsic and SiC, with diameters of about $100-150 \mu m$, are made by the chemical vapour deposition (CVD) technique. Multifilaments such as Nicalon (SiC), Sumitomo $(A1₂O₃)$ and Carbon fibres are made by pyrolysis of organometallic compounds in the form of tows (bundles of a few thousand 3–10 μm diameter fibres) or two/three-dimensional weaves of the tows. Short fibres such as Saffil and Kaowool are physically similar to multifilaments except for the shorter length.

Whisker-reinforced CMCs employ SiC and $Si₃N₄$ defect-free single crystals with lengths of the order of 0.2–250 μm and aspect ratio (length to diameter ratio) in the range of 2–250. Among the commercially available types, SiC whiskers have the greatest potential for improving the properties of brittle ceramic matrices, owing to their better reinforcing properties. Secondly, commercial grade SiC whiskers of high purity have been successfully produced by various routes, e.g. (i) thermal decomposition or hydrogen reduction of organic silicon compounds, (ii) hydrogen reduction of a gaseous mixture of silicon halides and hydrocarbons, (iii) recrystallisation of sublimed silicon carbide, (iv) supersaturation technique in the molten phase of silicon alloys, and (v) reaction between $SiO₂$ and C with a catalyst and reaction between Si and a hydrocarbon in the presence of H_2S .

Continuous fibre-reinforcement possesses the advantage of superior properties like stiffness and strength as compared to the discontinuous reinforcements (particulates, short fibres, etc.), but cannot impart isotropic properties and is unamenable to near-net-shape forming techniques. On the other hand, discontinuous reinforcements offer isotropic properties and the amenability to be processed by conventional ceramic powder processing techniques.

Chawla [\[9](#page-16-0)] has recently reviewed the major commercially available continuous fibre reinforcements and their suitability for different matrix systems. Effects of various reinforcements such as SiC, $SiC_{pl}(platelet)$, $SiC_w(whisker)$, TiC, B_4C , TiB₂, B_4C_{2pl} (platelet), B_4C_w (whisker), $A1_2O_{3pl}$ (platelet), Graphite, TiN, ZrB_{2pl} (platelet), SiAION and Diamond on different ceramic matrix systems have been studied extensively. For the majority of such CMC systems, improvements in mechanical properties have been achieved as compared to their unreinforced counterparts. The properties of some of the commonly available ceramic reinforcements are summarized in Table [16.3](#page-5-0) [[3\]](#page-16-0).

16.2.3 Interfaces

Performance of a composite material depends critically on the nature of the matrix/ reinforcement interface, as well as the interfaces between the matrix grains and the reinforcement particles. The interfaces play a crucial role in the load transfer between the matrix and the reinforcement as well as in interactions between cracks and reinforcements. Moreover, physical properties like thermal conductivity, coefficient of thermal expansion (CTE) and dimensional stability depend on the

 CMC_e $\frac{1}{2}$ \tilde{f} $\frac{1}{2}$ ÷. ϵ the \ddot{f} \div Ă

nature of the interface. It is thus desirable to have a clear understanding of the interfacial characteristics of different types of ceramic matrix composites and consequently to be able to tailor these for optimum performance of CMCs.

Although the primary role of the interface in CMCs is to transfer load from the matrix to the reinforcement, its nature primarily depends on the chemical reaction, wetting, and bonding between the matrix and reinforcement. Interfacial bonding can be categorized as mechanical and chemical. Mechanical bonding could arise due to mismatch of CTE or elastic modulus between the matrix and the reinforcement. It is comparatively a weaker bonding, but it is efficient in load transfer when the applied force is parallel to the interface. Interfacial bonding of this type can give rise to substantial toughening effects for brittle ceramic matrices via fibre pull-out/bridging mechanisms. On the other hand, chemical bonding due to one or more chemical reactions yields a much stronger interface between the matrix and the reinforcement. However, if a brittle reaction product is formed in the process, this may be a serious detrimental factor for the performance of a composite. It must be noted that although several studies of the influence of interface characteristics have been reported with many promising applications [[14,](#page-17-0) [42,](#page-18-0) [45](#page-18-0)], these effects are not yet fully understood; hence this topic is being further researched.

16.3 Toughening by Fibre Reinforcement/Crack Bridging

The toughening of ceramics (which is also more generally discussed in Chap. [18](http://dx.doi.org/10.1007/978-981-10-2134-3_18) of this Volume) by fibre reinforcement is most effective in CMCs, and is discussed in detail here. First we note that even the toughening of monolithic ceramics can be a complex process, with several mechanisms contributing simultaneously, see Fig. [16.2](#page-7-0). Bearing these comments in mind, the fracture process of a CMC adds additional complexities, which are illustrated schematically in Fig. [16.3](#page-7-0).

Different micromechanisms of fracture take place in three fracture regions: (i) the process zone ahead of the crack tip; (ii) at the crack tip itself; and (iii) in the crack wake. For CMCs the most important result is crack tip shielding owing to crack bridging. These mechanisms include some or most of the following [[30,](#page-17-0) [32,](#page-17-0) [40\]](#page-17-0), which by themselves might seem to be detrimental, but in combination with others become beneficial:

- 1. Local increase in the stress level with the application of external loading.
- 2. Development of compressive residual stresses at the crack tip.
- 3. Crack extension in the compressive stress zone.
- 4. Relative displacement of matrix/ interface elements.
- 5. Matrix microcracking, leading to matrix failure (with or without significant crack path meandering, i.e. crack deflection and/or branching).
- 6. Disbonding of matrix/fibre interface (with or without significant frictional forces).
- 7. Fibre pull-out and fibre breakage in the crack tip process zone.

- 8. Frictional sliding of the fibres along the matrix/fibre interfaces.
- 9. Loss of residual strain energy.

The overall result is significant energy dissipation through frictional events in the wake and process zones, acoustic emission and fibre disbonding, pull-out and breakage. Contributions from these stages of crack tip and fibre-reinforcement interactions, with or without the contributions from matrix fracture events, have led to significant increases in the fracture resistance [\[40](#page-17-0)].

Several constitutive laws and quantitative treatments of the fracture toughness enhancements by crack bridging are available in the literature and are summarized by Anthony G. Evans [[30\]](#page-17-0). A closer look at these studies suggests that the mode I fracture toughness of any particular material that exhibits crack bridging, measured

Fig. 16.2 Schematic of important toughening mechanisms in monolithic ceramics. After [[33](#page-17-0)]

in terms of the critical fracture energy (G_{Ic}) , varies considerably with (i) interface disbond toughness (τ_i/τ_f) the ratio of initial and final fibre sliding stresses), (ii) fibre characteristics, namely the length (2d, where d is defined in Fig. [16.3\)](#page-7-0), radius (R) and volume fraction (f), (iii) misfit strain between the matrix and the fibre (ϵ_{ii}^T) , (iv) frictional coefficient at the disbonding interface (μ) , (v) fibre strength (S) and matrix toughness (significant for strongly bonded interfaces), and (vi) fibre sliding stress (τ) and pull-out length (h_p), where the value of τ usually varies inversely with h_p . This large number of variables and parameters makes predictions of CMC fracture toughnesses very difficult.

CMCs toughened by fibre reinforcement are also called "Inverse Composites", since in these materials the brittle matrices fail before the fibres. Since the governing principle is that the failure strain for the matrix is less than that of the reinforcing fibres, matrix cracks have to be arrested and/or deflected at the fibre/matrix interfaces to avoid premature failure of the fibres. This is the only possibility for effective fibre-reinforced toughening.

16.4 Processing of CMCs

Processing of particulate and short fibre composites is carried out by a conventional powder metallurgy (P/M) process, since the reinforcements are essentially small-dimensioned. The ceramic and reinforcement powders are blended with suitable additives. The powder can be cold pressed in a die followed by sintering or hot pressing. Another method involves mixing the blend of ceramic and reinforcement powders with a binder and shaping the mix by extrusion or injection moulding, followed by debonding and sintering. A general schematic is shown in Fig. [16.4](#page-9-0).

CMCs reinforced with continuous fibres use a reinforcement skeleton or a preform woven into a porous solid block of fibres that has to be filled with a ceramic matrix. The preform can be woven/arranged in a solid block in a variety of forms, the most common being 3-D and 4-D (D-dimension) configurations. The pores are micron-sized and only a liquid or vapour can infiltrate and deposit the ceramic matrix within the preform.

There are four common methods adopted for the densification of the composite: reactive liquid infiltration (RLI), chemical vapour infiltration (CVI), polymer impregnation pyrolysis (PIP) and the Sol-Gel process. The processing methods are specific to certain composite systems:

1. RLI is used to process carbon fibre-reinforced silicon carbide composites (C-SiC). This process takes advantage of the high reactivity of carbon and liquid silicon. The preform is partially densified with carbon and infiltrated with molten silicon by capillary action under controlled conditions in a furnace. As the silicon infiltrates, it reacts to form silicon carbide. Figure [16.5](#page-9-0) gives a schematic of the RLI process.

Fig. 16.4 Flow chart for the processing of ceramic matrix composites

Fig. 16.5 Schematic of silicon melt infiltration in a reactive liquid infiltration (RLI) process. The crucible shown here contains liquid silicon, and the preform is essentially a carbon long fibre structure that is gradually infiltrated by silicon, which after reaction transforms to SiC

2. CVI involves the infiltration and reaction of two or more chemical species in the vapour state to form the desired ceramic matrix in the preform. The process is carried out in highly controlled temperature gradient conditions in a furnace. This method may yield any type of ceramic and is most popularly used for the processing of C–SiC or SiC–SiC composites. The chemical used is methyl trichlorosilane, which yields SiC. The CVI process is very slow and takes several months of continuous operation to yield a product. However, the composite properties are excellent. Figure [16.6](#page-10-0) shows a schematic of the CVI process.

Fig. 16.6 Schematic figure of thermal gradient CVI processing

- 3. PIP involves the use of a ceramic precursor polymer to first impregnate the porous fibre preform, followed by pyrolysis to form the ceramic. As the polymer pyrolyses, it leaves porosity and the impregnation process has to be repeated many times until the required density of the composite is achieved. This process uses pyrolysis of a polycarbosilane polymer to form a SiC matrix. A schematic of the process is shown in Fig. [16.7.](#page-11-0)
- 4. Aqueous sols of oxide ceramics like alumina and silica can be used to infiltrate porous fibre preforms, followed by gelling and removal of the aqueous medium by drying. This process can be repeated many times until the required composite density is achieved.

16.5 CMCs Properties

The material behaviour of fibre-reinforced CMCs is more similar to that of metals than monolithic ceramics, in that the strength does not depend on the volume of the part or structure (size effect). Hence reliable large CMC structures can be realized without increasing the failure risk. However, the availability of reliable data for material properties is limited. Additionally, the published values cannot be

Fig. 16.7 Schematic of the polymer infiltration pyrolysis (PIP) process

compared directly, owing to different evaluation methods and a lack of information about material composition and manufacturing details. These caveats should be borne in mind when consulting Tables [16.4](#page-12-0) and [16.5](#page-13-0), which provide a rough guide to the properties of some CMCs: these data cannot be used as design data without consulting the material manufacturer.

The mechanical properties are investigated on samples loaded parallel to the fabric or fibre layers. High temperature properties are determined in an inert gas atmosphere for carbon fibre based CMCs, and in ambient air for SiC/SiC material. The variations of the values in Tables [16.4](#page-12-0) and [16.5](#page-13-0) originate from the standard material variants, based on different fibre types, fibre volume contents and matrix composition.

The mechanical properties of CMCs are strongly influenced by the fibre/ matrix bonding. Therefore these materials tend to show similar ultimate strengths and failure strains when highly compatible fibre coatings are used. However, for melt infiltrated C/C—SiC materials where the carbon fibres are processed as supplied (i.e. no costly fibre coatings), the tensile and flexural strengths are significantly lower.

		CVI		LPI		LSI	
		C/SiC	C/SiC	C/SiC	$C/C-SiC$	$C/C-SiC$	C/SiC
Manufacturer		SNECMA (SPS)	MT Aerospace	EADS	DLR	SKT	SGL
Density (g/cm^3)		2.1	$2.1 - 2.2$	1.8	$1.9 - 2.0$	>1.8	\overline{c}
Porosity $(\%)$		10	$10 - 15$	10	$2 - 5$		\overline{c}
Tensile strength (MPa)		350	300-320	250	80-190	$\overline{}$	110
Strain to failure $(\%)$		0.9	$0.6 - 0.9$	0.5	$0.15-$ 0.35	$0.23 -$ 0.3	0.3
Young's modulus (GPa)		$90 - 100$	$90 - 100$	65	$50 - 70$	$\overline{}$	65
Compression strength (MPa)		580-700	$450 - 550$	590	$210 -$ 320	$\overline{}$	470
Flexural strength (MPa)		500-700	450-500	500	$160 -$ 300	$130-$ 240	190
ILSS (MPa)		35	$45 - 48$	10	$28 - 33$	$14 - 20$	$\qquad \qquad -$
Fibre content (vol.%)		45	$42 - 47$	46	$55 - 65$		
Coefficient of thermal expansion $(10^{-6}K^{-1})$	Ш	3 ^a	3	1.16^{d}	$-1-2.5^{b}$	$0.8 - 1.5^d$	-0.3
	\perp	5^{a}	5	4.06 ^d	$2.5 - 7b$	$5.5 - 6.5^d$	$-0.03-$ 1.36 ^e
Thermal conductivity (W/mK)	ı	$14.3 - 20.6^{\mathrm{a}}$	14	$11.3-$ 12.6^{b}	$17.0-$ 22.6°	$12 - 22$	$23 - 12^{f}$
	T	$6.5 - 5.9$ ^a	$\overline{7}$	$5.3-$ $5.5^{\rm b}$	$7.5 -$ 10.3°	$28 - 35$	-
Specific heat (J/kgK)		620-1400	-	$900 -$ 1600 ^b	$690 -$ 1550		

Table 16.4 Typical material properties of fabric and UD crossply (0°/90°; EADS) based C/SiC and C/C—SiC materials, depending on the manufacturing method—chemical vapour impregnation (CVI), liquid polymer impregnation (LPI) or liquid silicon impregnation (LSI)

l and ⊥ = fibre orientation; ^aRT-1000 °C; ^bRT-1500 °C; ^c200–1650 °C; ^dRT-700 °C; ^e200–1200 ° $C; ^{f}20-1200$ °C

At temperatures up to 1200 °C, and in an inert gas atmosphere, the mechanical properties of C/C—SiC are slightly higher than at room temperature, similar to the behaviour of C/C materials. However, at temperatures above 1350 °C in a vacuum, a certain decrease of tensile strength was observed.

N.B: The lifetimes of carbon fibre based CMCs in air is limited above 450 °C, owing to oxidation of exposed fibre ends and exposure of fibres owing to matrix cracking. Thus despite the fact that a SiC matrix and surface coatings significantly increase oxidation stability, C/SiC and C/C-SiC materials are not usable for long-term applications, e.g. in gas turbines.

16.6 Aerospace Applications

There are numerous actual and potential applications of CMCs in the aerospace arena. Most are for high temperature oxidizing environments pertaining to aeroengines and re-entry space vehicles.

Because of the sensitivity of carbon to oxidation, ceramic matrices have been developed since the middle of the 1970s to replace carbon in order to obtain materials capable of long-term resistance to high thermal fluxes and mechanical loads in oxidizing environments. C–SiC or SiC–SiC composite materials are preferred for these applications. These materials offer good strength (about 300 MPa) at room temperature, and a non-brittle behaviour, with an enhanced failure strain of about 0.5% .

The feasibility of different aeroengine parts, hot gas valve parts, thermal structures and thermal protection systems (TPS) of re-entry vehicles based on carbon ceramic or ceramic—ceramic materials, has already been demonstrated, see Figs. 16.8, [16.9](#page-15-0) and [16.10.](#page-15-0)

Fig. 16.8 SiC–SiC and C–SiC composites for aeronautical applications

Fig. 16.9 C-SiC composites for thermal protection systems (TPS)

Fig. 16.10 SEPCARBINOX flaps for Rafale M88-2 engine

16.7 Summary

In designing ceramic matrix composites, due consideration is to be given for selecting the matrix, ceramic reinforcement and the interface. Each of them plays a vital role in deciding the final properties of the composite. Processing of discontinuously reinforced composites is relatively easy and has already found a number of applications. Complicated processing like chemical vapour infiltration is currently required to make SiC fibre reinforced SiC composites with excellent high temperature properties. Also, this process is slow and takes several days to months to make components. Alternative processing techniques as well as improved fibres need to be developed. At present the new fibre reinforced composites like C–SiC and SiC–SiC are replacing earlier materials in critical aerospace applications. With further developments and innovation in processing, they should find wider application.

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