Proc. of SPIE Vol. 2855, High Heat Flux Engineering III, ed. A M Khounsary (Nov 1996) Copyright SPIE

Chemical vapor deposited SiC for high heat flux applications

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ABSTRACT

Important properties and applications of CVD SILICON CARBIDE^{TM} with particular emphasis on high heat loads are reviewed. Data on the mechanical and thermal properties of CVD-SiC as function of temperature are presented. Further, the effect of different high temperature treatments on flexural strength of CVD-SiC is discussed and the thermal shock resistance of CVD-SiC is compared to other competing materials. Finally, several high heat flux applications in the areas of optics, semiconductor processing and wear parts are discussed.

Key Words: Silicon Carbide, CVD-SiC, Optics, Laser Optics, RTP Rings

1. INTRODUCTION

SiC is a good material for high heat flux applications due to its many attractive properties such as high value of thermal conductivity and flexural strength, low value of thermal expansion coefficient and excellent resistance to thermal shock, oxidation and chemicals[1-18] . SiC is a wide band gap material (band gap = 2.2 - 2.86 eV) with good theoretical transmission in the wavelength region 0.5 - 6μm, it can be used for both reflective and transmissive applications. High heat flux applications may arise in several different areas such as optics (windows and domes for high speed missiles and lasers, synchrotron and high energy laser mirrors), semiconductor processing (susceptors and rings for rapid thermal processing (RTP)) and wear parts (optics molds).

The properties of SiC depend considerably upon the specific method used to produce it. Five basic types of SiC are currently available: viz., hot pressed, sintered, siliconized or reaction bonded, single crystal and chemical vapor deposited (CVD). SiC made by sintering and hot pressing SiC powders or by the reaction bonding process is usually a porous and/or multiphase material. These forms of SiC usually possess lower values of thermal conductivity, flexural strength and thermal shock resistance. Further, these SiC neither transmit very well in the visible and infrared regions, nor do they produce a very smooth surface on polishing. Fabrication of an optical figure from these latter forms of SiC require an overcoat (cladding)of CVD SiC, Si or any other suitable material.

Single crystal SiC (undoped) provide the best possible thermal properties of SiC. However, single crystal SiC is quite expensive and difficult to produce in large sizes. The hexagonal form of single crystal SiC has been produced in small sizes for semiconductor applications, and is currently available commercially. The single crystal β-SiC, which is cubic and isotropic, is not readily available.

Chemical vapor deposited SiC is a potentially superior material for high heat flux applications. By varying the process parameters, SiC properties can be optimized for either reflective or transmissive optics applications[1-2] . This change in transmission does not significantly affect the thermal properties of SiC. CVD-SiC is a theoretically dense, highly pure polycrystalline material which is free from voids and micro-cracks. Further, the CVD process is scaleable and is capable of producing near net shape and size parts[3-7]. Monolithic SiC parts upto 1.0-m in diameter has been successfully produced. Heat exchanger channels designed to actively cool mirrors have also been successfully fabricated in SiC optics used for synchronous systems and high energy lasers. The CVD-SiC process can be further scaled to yield multi-meter size parts. Large scaling capability reduces cost and makes CVD-SiC components cost effective in comparison to other competing materials. Finally the CVD process is reproducible. This reproducibility has been demonstrated statistically by plotting important properties of CVD-SiC on Statistical Quality Control (SQC) charts. An important consequence of reproducibility and homogeneity of CVD-SiC is that a fabrication process can be developed to yield parts of consistent quality and finish from batch to batch.

Table 1 shows a comparison of important properties of different forms of SiC. We see that CVD-SiC has properties superior to all other forms of SiC except the single crystal SiC which is not readily available in bulk form.

Table 1: Comparison of important properties of different forms of SiC

Material	Density	Thermal Conductivity ($Wm^{-1}K^{-1}$)	CTE 20-1000°C (K^{-1} x 10^{-6})	Elastic Modulus (GPa)	Polishability (A RMS)
CVD-SiC	3.21	330	4.0	466	≤ 3
Single Crystal SiC	3.21	490	---	---	≤ 3
Reaction Bonded SiC	3.1	120-170	4.3	391	≥ 20
Hot Pressed SiC	3.2	50-120	4.6	451	≥ 50
Sintered SiC	3.1	50-120	4.5	408	≥ 100

In this paper, CVD-SiC use for high heat flux applications is discussed. In Section 2 that follows, important properties of CVD-SiC relevant for high heat flux applications are presented. In particular, the temperature dependence of thermal conductivity, thermal expansion coefficient, heat capacity, flexural strength, elastic modulus and bulk absorption coefficient are included. In Section 3, the thermal shock resistance of CVD-SiC is discussed. High heat flux applications are discussed in Section 4. These applications have been selected from areas of optics, semiconductor processing and wear parts. Finally, conclusions are included in Section 5.

2. CVD-SIC PROPERTIES

The CVD process was used to produce two different types of SiC; opaque and transparent SiC. Most properties of these two forms of SiC are the same. The main difference is in visible-infrared transmission. The transparent SiC has high transmission in the 0.5 - 6 μm region while the opaque material exhibits significant amount of scattering which makes it unsuitable for transmissive optics applications. Both types of CVD-SiC are fabricated by the pyrolysis of methyltrichlorosilane in excess H_2 in a hot wall CVD reactor. The process conditions used for the opaque SiC are: substrate temperature = 1350°C, furnace pressure = 200 torr, H_2/MTS molar ratio = 4-7 and deposition rate = 1-2 μm/min. The CVD process conditions for the transparent SiC are: substrate temperature = 1380-1470°C, furnace pressure = 2-25 torr, H_2/MTS ratio = 10-30, and deposition rate = 0.2-1 μm/min. Further, addition of 5%-20% HCl was determined to be beneficial in enhancing the transmittance of transparent SiC. Additional details about the CVD-SiC process are given in references 1-18.

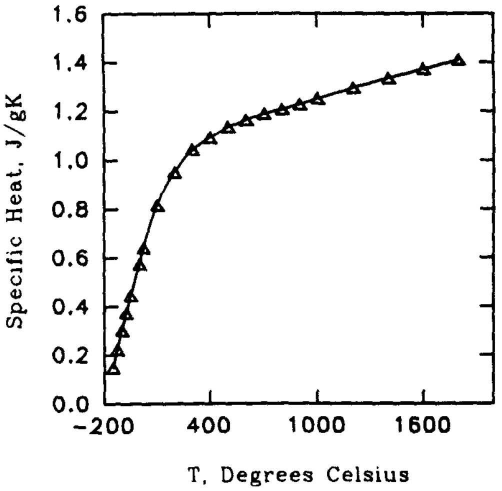
CVD-SiC produced at Morton Advanced Materials under the trade name CVD SILICON CARBIDE™ has been extensively characterized for important physical, optical, mechanical, thermal and electrical properties. These properties are reported in references 2, 7-9. All these measurements essentially show that CVD-SiC is a theoretically dense, highly pure, β-phase polycrystalline material possessing superior mechanical, thermal, optical and physical properties of interest. Table 2 lists some important properties of CVD-SiC.

For high heat flux applications, it is important to know the properties of CVD-SiC as function of temperature. Figure 1 shows the specific heat of CVD-SiC as a function of temperature in the temperature range -150 to 1800°C. The specific heat values were measured using Netzsch differential scanning calorimeter upto 1200°C at Purdue university. Values at and above 1400°C were extrapolated. These specific heat values are typical of polycrystalline SiC.

Figure 2 shows the thermal conductivity of CVD-SiC as function of temperature in the range of -150°C to 1800°C for two samples which were taken from two different areas of the furnace. Thermal diffusivity was measured using the laser flash technique at Purdue university, and then, from these data, thermal conductivity was calculated using the density value at room temperature (d = 3.207 g cm^{-3}) and heat capacity data as a function of temperature. At -150°C and 1800°C, the thermal diffusivity values were not measured but extrapolated. Both samples in Figure 2 show essentially the same thermal conductivity values indicating that CVD-SiC is homogeneous. Further, the thermal conductivity peaks at 485 $Wm^{-1}K^{-1}$ near 173K and the curve shows a T^{-1} dependence above 200°C as exspected for SiC [23].

Table 2: Important Properties of CVD-SiC

Property	Average Value	
Color	Dark gray (opaque SiC) Yellow (transparent SiC)	
Crystal Structure	FCC Polycrystalline, β-phase Randomly oriented (opaque SiC) Highly Oriented <111> (transparent SiC)	
Average Grain Size (μm)	5-10	
Optical Transmittance, 0.6-5.6 μm (0.5-mm thick)	0%	(opaque SiC)
	> 40%	(transparent SiC)
Attenuation Coefficient (cm^{-1})		
@ 0.6328 μm	> 100	(opaque SiC)
	6.9	(transparent SiC)
@ 3 μm	>60	(opaque SiC)
	2.2	(transparent SiC)
Density ($g\ cm^{-3}$)	3.21	
Vickers Hardness (1 Kg load)	2540	
Fracture Toughness, K_{IC} ($MN\ m^{-1.5}$)	3.1	(opaque SiC)
	2.2	(transparent SiC)
Elastic Modulus, GPa	466	
Flexural Strength, MPa	470	
Weibull Parameters		
Modulus, m	11.45	
Scale Factor, MPa	462	
Trace Element Impurities (ppmw)	3.2	
Coeff. of Thermal Expansion @ 293K ($10^{-6}\ K^{-1}$)	2.2	
Thermal Conductivity @ 27C ($W\ m^{-1}\ K^{-1}$)	330	(opaque SiC)
	214	(transparent SiC)
Heat Capacity ($Jkg^{-1}K^{-1}$)	640	
Electrical Resistivity (ohm-cm)	1-50	(opaque SiC)
	4.5×10^{4}	(transparent SiC)
Dielectrical Constant (35-50 GHz)	136	
Dielectrical Loss (35-50 GHz)	75	
Loss tangent	0.55	
Thermo-optic Coefficient, dn/dT ($10^{-6}\ K^{-1}$)		
@ 2-4 μm	37	
Refractive Index		
@ 633 nm	2.635	
@ 1152 nm	2.576	
@ 1523 nm	2.566	



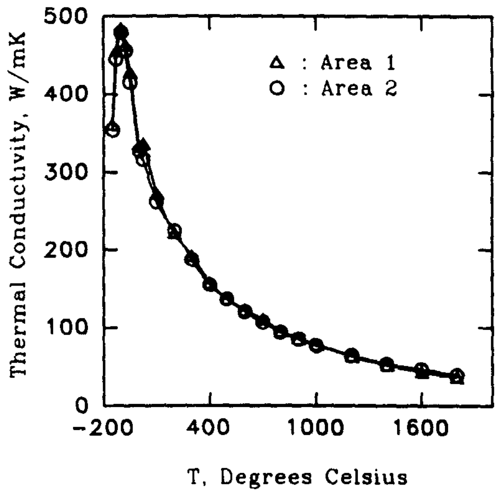


Figure 1. **Specific heat as function of temperature for CVD-SiC.**

Figure2. **Thermal conductivity as a function of temperature for two samples of CVD-SiC.**

Figure 3 shows the thermal expansion coefficient (CTE) of CVD-SiC as function of temperature in the range of 133K to 1273K. These measurements were made using differential dilatometer supplied by Theta Industries, Inc. We see that CTE decreases rapidly at low temperatures and remains relatively constant at high temperature. This behavior is typical of crystalline materials such as CVD-SiC.

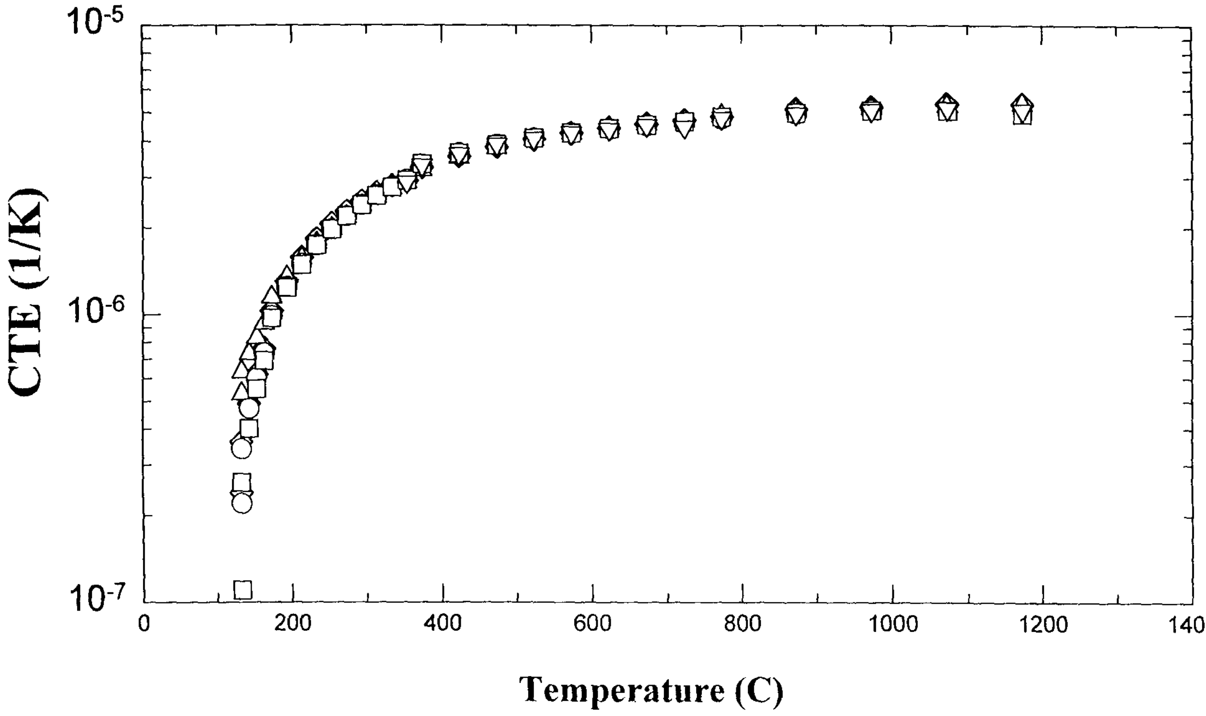


Figure 3. **Thermal expansion coefficient as a function of temperature for CVD-SiC**

The flexural strength of CVD-SiC as function of temperature in the range of 79K to 1723K are shown in Figure 4. The flexural strength measurements were made using an Instron mechanical tester at two different facilities, Morton Advanced Materials and University of Dayton Research Institute. For these data, all the beams were prepared with a surface finish of ~ 0.5 μm RMS. The flexural strength data of Figure 4 shows that CVD-SiC strength increases with temperature. This effect has been observed previously for CVD-SiC and is attributed to small plastic deformation that occurs at crack tips at higher temperatures.

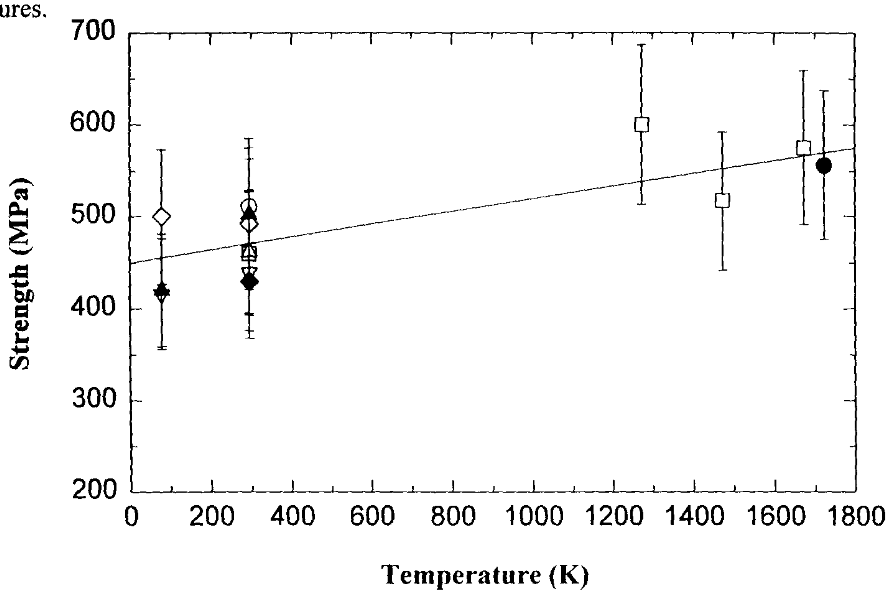


Figure 4. Flexural strength (four point loading) of CVD-SiC as function of temperature

The elastic modulus of CVD-SiC as a function of temperature is shown in Figure 5. The elastic modulus was measured at University of Dayton Research Institute using a Grindo Sonic, MK3 (J.W. Lemmens, Co). From Figure 5 we see that the sonic modulus decreases by only 10% when the temperature increases from room temperature to 1500°C.

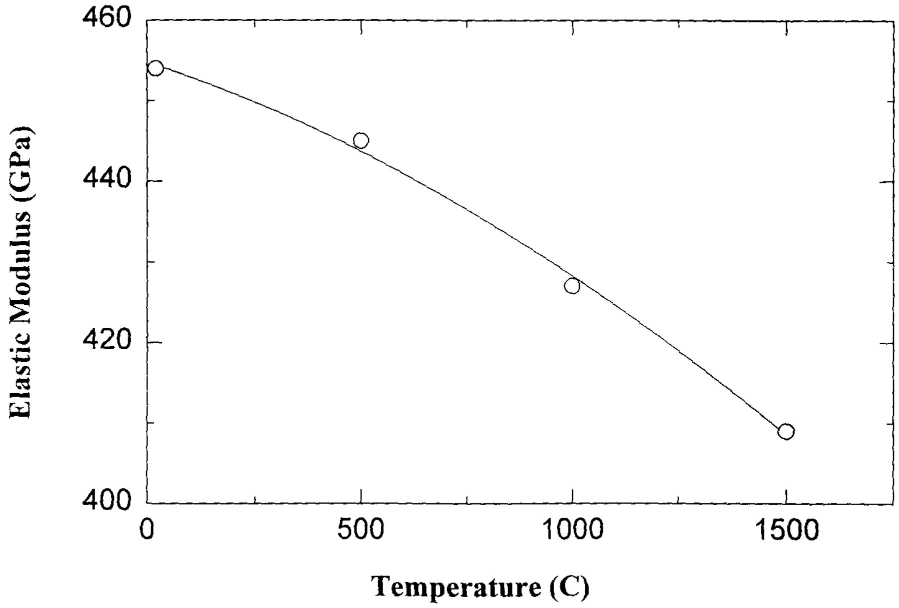


Figure 5. Elastic (sonic) modulus of CVD-SiC as function of temperature

The transparent SiC was characterized at Johns Hopkins University for absorption coefficient, emittance and dn/dT where n refers to the refractive index of CVD-SiC[19]. Figures 6-8 show these results. Figure 6 shows the absorption coefficient of transparent SiC for two different temperatures, 291K (lower curve) and 912K (upper curve) as function of wavelength in the range of 2.5 - 6.67 μm. The data are shown by the dotted line while the multiphonon model calculatios are shown by the solid lines. We see that as the wavelength decreases, the absorption coefficient also decreases. At wavelength < 3 μm, the four phonon band is visible.

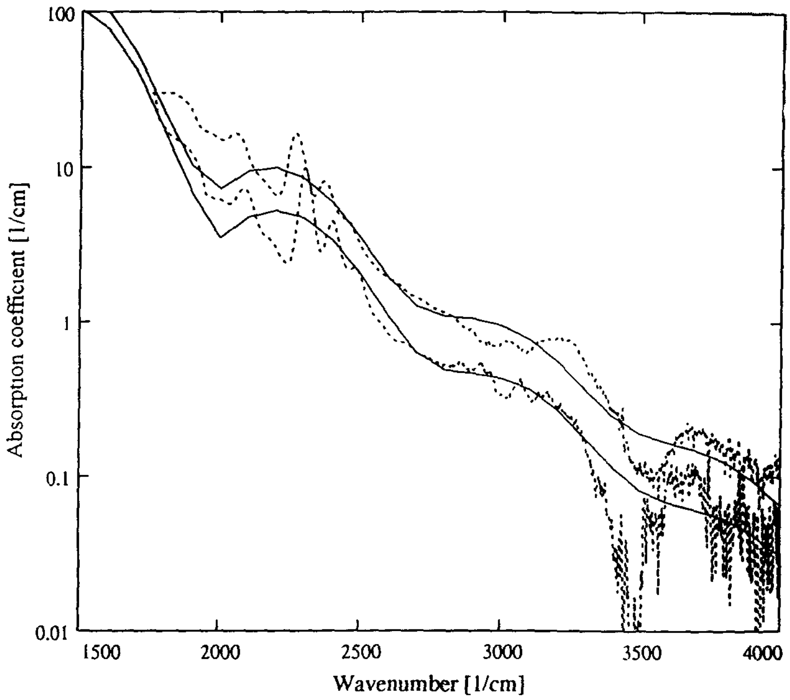


Figure 6. Absorption coefficient of transparent SiC as function of wavelength at two different temperatures, 291K (lower), 912K (upper). Data (dotted lines) are compared with multiphonon model[19].

The emittance of 1-mm thick sample of CVD-SiC is computed based upon the above absorption coefficient and is plotted in Figure 7 as function of wavelength. The mean emittance at 4-5 μm is about 0.3 at room temperature and 0.5 at 815K. However, in the 3-4 μm range, the mean emittance is about 0.05 at room temperature and about 0.1 at 815K. These data show that transparent SiC is a good candidate material for high heat flux optics applications, particularly at wavelength < 4 μm.

The dn/dT for transparent SiC as function of wavelength in the region of 2 - 4 μm is shown in Figure 8. We see that dn/dT value is fairly constant in this wavelength range and equal to about 37 x 10^{-6} K^{-1}.

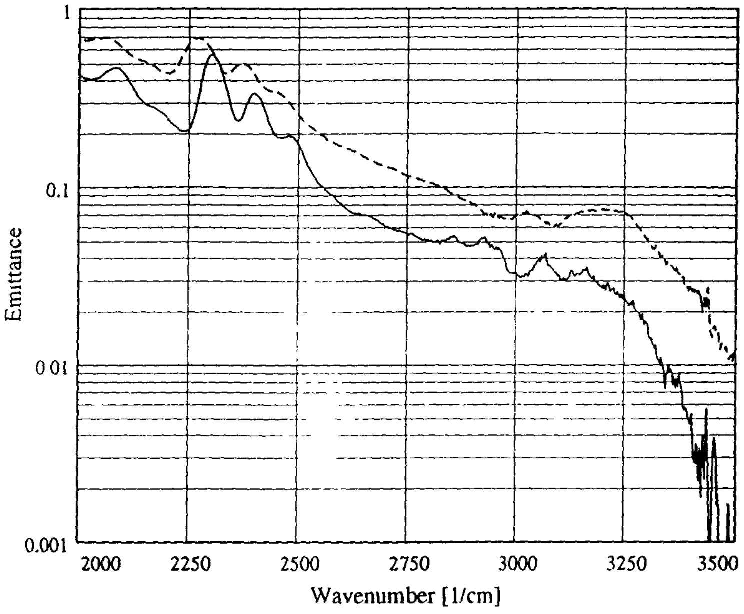


Figure 7. Calculated emittance for transparent SiC at 291K (solid line) and 912K (dashed line). The material thickness was assumed to be 1-mm.

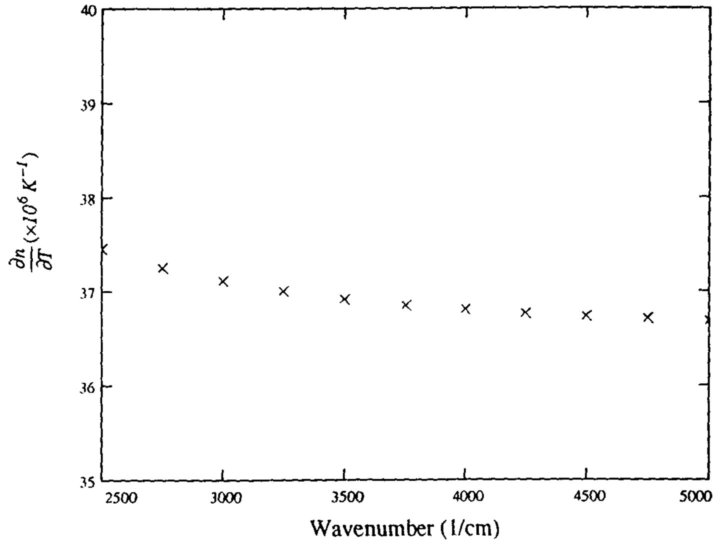


Figure 8. Thermo-optic coefficient, dn/dT of transparent SiC

3. THERMAL SHOCK RESISTANCE

For high heat flux applications, it is important that the material exhibits good resistance to thermal shock. The thermal shock parameter, R is defined as $\sigma\kappa(1-\nu)/\alpha E$, where σ is the flexural strength, κ is the thermal conductivity, ν is the Poisson's ratio, α is the thermal expansion coefficient, and E is the elastic modulus. This parameter provides a relative indication of thermal shock resistance of materials when Biot number, $hL/\kappa \leq 1$[20]. Here h is the heat transfer coefficient and L is the characteristic dimension, which could be thickness of the window or dome. Table 3 compares the thermal shock parameter of CVD-SiC with that of several competing materials[20-21]. We see that CVD-SiC has a thermal shock parameter value which is significantly greater than those of all other materials except diamond. However, diamond is a relatively expensive material and is difficult to polish and obtain in large sizes. Further CVD diamond grown in thick layers exhibits growth in grain size with thickness which results in considerable thermal conductivity variation along the thickness of the material. Overall, CVD-SiC is a preferred material for high thermal shock resistance.

CVD-SiC was also subjected to different thermal treatments consisting of thermal shock by water quench, and annealing at 1000°C in N_2 and vacuum[9]. The flexural strength of CVD-SiC increased by 37% - 53% when it was heated to a temperature range of 600°C-1000°C and quenched in water or annealed in flowing N_2. In the case of thermal shock by water quenching, the increase started at 600°C and became relatively constant above 800°C. This increase was attributed partially to an increase in compressive residual stresses and partially due to healing of machining flaws[9].

Table 3: Comparison of thermal shock resistance of some important optical materials

Material	Flexural Strength σ (MPa)	Elastic Modulus E (GPa)	Poisson's Ratio ν	Thermal Conductivity κ ($Wm^{-1}K^{-1}$)	Thermal Expansion Coefficient α (10^{-6} K^{-1})	Thermal Shock Parameter R
CVD-SiC	470	466	0.21	330	2.2	119.5
Sapphire	400	380	0.27	24	8.8	2.1
Spinel	160-190	190	0.26	14.6	8.0	1.2-1.39
ALON	300	315	0.24	12.6	7.8	1.02
Yttria	116	164	0.3	14	7.1	0.94
MgF_2	100	115	0.3	16	11.0	0.89
Diamond	2000	1050	0.16	2000	0.8	4000
GaP	100	103	0.31	97	6	10.8
GaAs	60	86	0.31	53	6	4.3
CVD-ZnS	103	75	0.29	16.7	7	2.3

4. HIGH HEAT FLUX APPLICATIONS

High heat flux applications of CVD-SiC can be found in several different areas such as optics, semiconductor processing and wear parts . Some of these applications are still in the developing stage. The details of these applications are discussed below:

4.1 Optics applications

CVD-SiC is an attractive material for fabricating both transmissive and reflective optics components. The transparent form of CVD-SiC can be used as windows and domes in the visible and infrared in severe environments associated with high speed missiles, combustion, space and laser systems. Of particular significance is its use in laser welding systems and free electron lasers operating at a wavelength of 1.06 μm. Transparent SiC can also be used for fabricating high temperature luminous sources and is a good candidate material for electronic packaging applications because of its high electrical resistivity and thermal conductivity. The transparency of the material can be used to provide optical connection on the electronic chip.

For reflective optics applications, CVD-SiC has been used to fabricate lightweight mirrors, x-ray grazing incidence mirrors, optics standards, and optics baffles. The CVD-SiC mirrors are used in surveillance, high energy lasers, laser radar systems, synchrotron x-ray and VUV telescopes, large astronomical telescopes and weather satellites. A few examples of high heat flux applications of CVD-SiC in optics are discussed below:

Table 4 summarizes the results of high power CW Nd:Yag laser irradiation of a sample of transparent SiC for laser welding applications. The Yag laser was passed through a fiber optics cable to produce a spot size of 750 μm. The sample thickness was 0.54-mm. The input power was varied in the range 55-550 watts. We see that even at very high energy densities the was no appreciable degradation in the transmittance of transparent SiC. Further, after laser irradiation was completed, no visible damage to transparent SiC sample was observed. In comparison, other competing materials such as sapphire, ALON, CLEARTRAN^{TM} and quartz did not survive the extreme thermal shock.

Table 4: CW Nd:Yag laser irradiation results

Laser wavelength = 1.06 μm **Spot size = 750 μm** **Sample thickness = 0.54 mm**

Input Power (W)	On Time (s)	Power Density (KW cm^{-2})	Energy Density (KJ cm^{-2})	Output Power (W)	Transmittance (%)
55	5	12	60	34	62
58	5	13	65	37	64
82.5	5	19	95	52	63
290	5	66	330	177	61
550	3	125	375	--	--

In order to provide a proper thermal control of the optics for high heat loads, heat exchanger channels or patterns can be fabricated on the backside of the CVD-SiC faceplate. These patterns can be fabricated directly in the CVD chamber by near net shape replication process. Since in a CVD process, the deposition occurs on a substrate layer by layer on the molecular scale, the patterns are replicated down to very fine details. This replication of fine features was demonstrated in CVD-SiC on the Thermally Controlled Tertiary Mirror (TCTM) program[22]. Figure 9 shows a picture of such replication in CVD-SiC. This picture shows a heat exchanger pattern consisting of posts and crosses. The depth of this pattern is about 1-mm, the thickness of crosses is 0.25-mm, the diameter of posts is 0.75-mm and the spacing between center lines of two adjacent posts is 1.25-mm. This replication was performed on graphite. From Figure 9 we see that fine features of heat exchanger pattern are replicated precisely. There was no evidence of any rounding, pits, holes or voids on the surface. The cross-section of the replicated structure indicated that the sharpness of the replication was maintained throughout the depth of the structure.

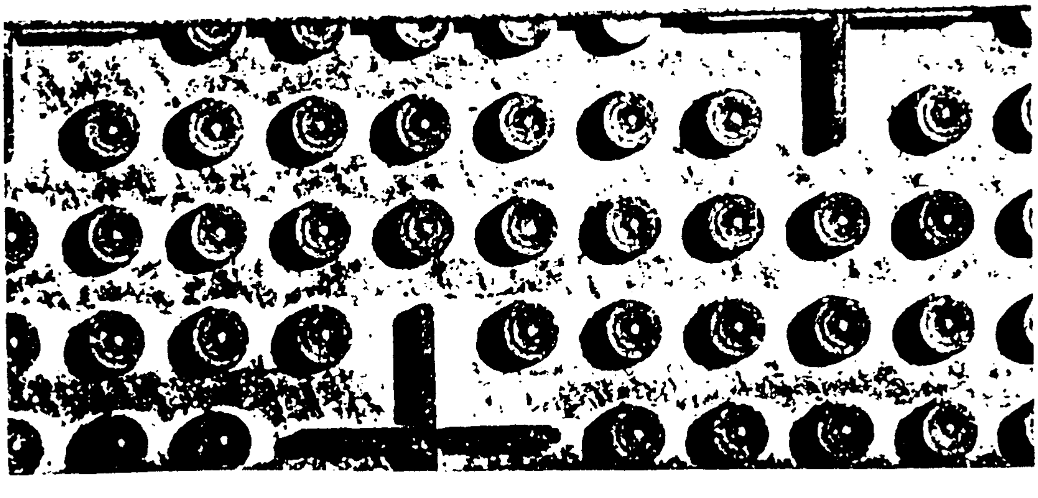


Figure 9. Heat exchanger pattern replicated during CVD-SiC process. Post diameter is 0.75-mm; crosses are 0.25-mm wide.

4.2 Semiconductor processing applications

The superior properties of CVD SILICON CARBIDE^{TM} make it an attractive candidate material for semiconductor processing applications. These applications include different wafer support components such as susceptors, lifting parts, plates, and flow controls for such processes as RTP, CVD, ion implant, etching, lithography, and dry, vapor-phase and wet cleaning. However, here we will concentrate on high heat flux applications which include susceptors and rings for processes such as RTP epi, and CVD; cantilevers and wafer carriers for oxidation or diffusion furnaces; and electrodes for plasma etching.

A silicon (Si) epitaxial reactor requires very high temperatures and extreme cleanliness. The severe temperature excursions, particularly in the rapid thermal processors have a tendency to crack or flake the SiC coating on the currently used graphite susceptors. Consequently, bulk CVD-SiC susceptors provide an attractive alternative. In addition, the high stiffness to weight ratio of CVD-SiC allows the susceptor to have low weight and good surface flatness. This low mass coupled with high thermal conductivity keeps the heat ramps rapid and contributes to temperature uniformity over the wafer. The CVD-SiC susceptors also do not readily degrade during hot HCl cleaning cycles, permit tight susceptor tolerances due to a close thermal expansion match with Si, generate fewer particulates, and can be thermally cycled many more times than other competing materials. The excellent machinability and process reproducibility of CVD-SiC ensures that the parts are fabricated to the same shape with consistent high quality.

In addition to susceptors, slip rings constitute another critical element in RTP, RTCVD, and RTEpi reactors. Such a ring surrounds the wafer and is usually slightly offset from its plane. The slip rings serve to make the radial temperature profile more uniform in the wafer. Without a slip ring, the edge of the wafer leads the rest of it in temperature during ramping, while in steady-state, there is an edge loss. CVD-SiC provides definite advantages over other materials because of its high thermal conductivity, predictable absorption and emission characteristics, higher purity and lower particle generation.

Semiconductor furnaces often employ high temperatures for oxidation or diffusion and use support components such as cantilevers, wafer carriers, support tubes and paddles. Currently used cantilevers are heavy, conduct heat poorly, and have a coefficient of thermal expansion that is quite different from silicon. Quartz is relatively fragile, produces more particulates, cannot withstand HF in wet processing, and has high sodium content. For all these reasons, CVD-SiC offers a better alternative. The transparent form of CVD-SiC provides not only a perception of high purity, but also the capability to see the wafers without taking them off the support components. Currently, efforts are being made to fabricate the above components from CVD-SiC.

Because of its low resistivity of about 1-50 ohm-cm, CVD-SiC conducts electricity and is used for electrodes in plasma etch systems. Electrodes made from CVD-SiC do not degrade readily and last for a long time in the hostile plasma environment. This reduces equipment downtime and makes this material very competitive in terms of cost of ownership

4.3 Wear applications

CVD-SiC has been successfully used as a substrate material for making optics molds because of its high value of thermal conductivity, elastic modulus, and flexural strength, and its resistance to abrasion, scratch, oxidation and chemicals. CVD-SiC provide more uniform temperature over the whole surface of glass or plastic optics, thus minimizing residual stress during optics cooldown. The significant advantages of CVD-SiC are particularly realized when large area optics molds are used. Further, CVD-SiC molds have long life, and have been successfully used for fabricating hundreds of optics parts from one mold.

5. SUMMARY AND CONCLUSIONS

CVD-SiC is a good material for high heat flux applications due to its superior thermal, mechanical, optical and physical properties, excellent high temperature property retention, and its high resistance to thermal shock, oxidation, abrasion, wear and corrosion. CVD-SiC thermal shock resistance is significantly better than most other competing materials. The flexural strength of CVD-SiC increased by 37-53% when it was heated to a temperature range of 600-1000°C and quenched in water or annealed in flowing N_2 or vacuum.

High heat flux applications of CVD-SiC can be found in many different areas such as optics, semiconductor processing and wear parts. In optics, the applications consists of windows and domes for high speed missiles and lasers, and mirrors for high energy lasers and synchrotron x-ray systems. Sophisticated heat exchanger patterns can be fabricated in the CVD-SiC by the replication process directly in the CVD chamber. In semiconductor industry, currently the applications are focused around furnace support components such as susceptors and RTP rings. However, as workers become more accustomed to CVD-SiC use, applications in other areas will expand. Finally, in wear parts, the primary application of CVD-SiC is in optics molds.

6. ACKNOWLEDGEMENTS

The authors would like to thank Steve Daignealt of SSG, Inc. for providing information about Itek TCTM program and Michael Thomas of Johns Hopkins University, Applied Physics Laboratory for providing data about transparent SiC.

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