Energy and Environmental Research Emphasizing Low-Rank Coal -- Task 6.2 Joining of Advanced Structural Materials

Topical Report

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1.0 INTRODUCTION

In order to increase national energy self-sufficiency for the near future, energy systems will be required to fire low-grade fuels and use more efficient energy cycles than those available today. The steam cycle used at present is limited to a maximum steam temperature of 550°C and thus a conversion efficiency of 35%. To boost efficiency significantly, much higher working fluid temperatures are required. This means that subsystems must operate at much higher temperatures and therefore in much more corrosive environments than those currently used. Problems of special concern are corrosion and fatigue of directly fired turbine blades, corrosion and blinding of hot-gas cleanup filters, and corrosion and failure of high-temperature heat exchangers. To prevent failure of those structures will require the use of advanced materials, particularly ceramics, for construction. To fully utilize the potential of ceramic materials in structural applications, economical and practical methods must be developed to assemble, or join, the components of the structure. Once an effective method is found, it will be possible to construct and test large ceramic structures such as high-temperature heat exchangers and other components used in combustion and energy engineering.

Silicon carbide (SiC) is considered an attractive material for structural applications in fossil energy systems because of its corrosion and wear resistance, high thermoconductivity, and high-temperature strength. These same properties make it difficult to sinter or join SiC. Conventional sintering techniques require applying pressure and heating to temperatures near 2000°C, or the use of binders with lower melting temperatures, or pressureless sintering with the aid of carbon and boron to near full density about 2100°C.[1] The sintering temperature can be reduced to 1850°–2000°C if SiC is sintered with the addition of small quantities of Al_2O_3 and Al_2O_3 + Y_2O_3.[2-4] In addition, reaction sintering has been used by mixing Si and C with SiC powder and heating the mixture to 1400°C to cause the Si and C to react and form SiC, which bonds the aggregate together.

Work proposed for this year was to center on determining gas compositions that could be used to increase the sinterability of oxide binders and on using the binder and gas combinations to join bars of SiC, alumina, and mullite ($3Al_2O•2SiO_2$). During the course of the year the focus was shifted to SiC joining alone, because it was felt that alumina and mullite are too prone to thermal shock for use in structural applications in fossil energy systems. Because of a thermal expansion mismatch between alumina and SiC, only SiC and mullite were investigated as joining aides for SiC. Therefore, the objectives of this work evolved into examining the sintering phenomena of SiC and mullite-derived binders at and below 1500°C in various atmospheres and determining which conditions are suitable to form strong joints in monolithic SiC structures to be used at temperatures of 1000°–1400°C.

Because the number of materials to be joined was reduced, the number of joining compounds was expanded and more detailed analyses of the samples were employed than originally planned. Also, some funds were shifted to preliminary investigations of the potential to use joule heating to induce reaction sintering in the joining of SiC monoliths.

2.0 JOINING SILICON CARBIDE WITH OXIDE COMPOUNDS

2.1 Background

The principal factors determining whether or not a compound will make a good SiC joining aid are 1) sintering temperature below that of SiC but above the maximum use temperature, 2) thermal expansion coefficient close to SiC, 3) high creep resistance, 4) and ease of use in the chosen joining procedure. The joining technique requires the control of the SiC–binder interface properties since it is the region of maximum distortion and deformation.[5] Therefore, the interface must have properties that optimize fracture strength, toughness and corrosion resistance and minimize slow crack growth.

Generally, the strength of the SiC–binder interface is governed by the thermal expansion coefficient and elasticity mismatches, the fracture resistance of interface, the relative thickness of binder, and the flaw distributions in the SiC and binder layer. SiC–mullite joints offer the prospect of good high- temperature properties, since there is little difference in the coefficients of thermal expansion. However, there are still many aspects of SiC–mullite composite to be understood. One is the effect of atmosphere on sinterability and stability of the binder. Also, little information is available on high-temperature grain-boundary sliding at the SiC–mullite interface caused by a viscous glassy phase and its effect on compressive strength.

Two major factors, the densification of mullite and the high-temperature stress–strain property of mullite binder, were considered most important in predicting the properties of a SiC–mullite joint. The densification rate of mullite powders significantly increases in the presence of small amounts of liquid phase. Grain-boundary transport or diffusion appear to be the primary mechanism of densification.[6,7] Since the melting temperature of mullite is about 1830°C, an apparent density above 90% may be achieved by sintering powder compacts at temperatures above 1700°C. The sintering temperature may decrease to 1500°C if it is performed under pressures of about 355 MPa (50 kpsi).[8]

Several factors were considered in determining the best conditions to stimulate the formation of mullite–SiC joints with favorable characteristics. Mechanical properties of sintered powders depend on the morphology of powder grains. Materials with columnar (or elongated) grains demonstrate higher fracture stress[9] and creep resistance than those with circular grains. The formation of the columnar grains requires the presence of a liquid phase.[1] The addition of a small quantity of amorphous silica should therefore lower the sintering temperature of mullite and modify the morphology of the grains. Stable mullite solid solutions should form at grain boundaries if the mullite content is 85%.[11] If the silica content is too high, there may be some problems caused by the crystallization of cristobalite upon cooling.[12] Sintering of mullite under reducing conditions accelerates volatilization of silica, which may cause the intergranular phase to be enriched in alumina.[11] Also, water vapor or hydrogen in the atmosphere may create silanol groups in the silica, which may promote diffusion processes and therefore should promote sintering of SiC–mullite composites.

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2.2 Experimental

High purity mullite powder (melting temperature 1828 ± 10°C), a naturally occurring material known as mulcoa (87% mullite and 13% amorphous silica), SiC, and mixed SiC–mulcoa (70/30) were used in the sintering tests. All were sized below 30 μm. The tests were performed in air, in air and water vapor, and under reducing conditions including H_2 = 25.2 mole%, CO_2 = 10.3 mole%, and 0–30 mole% water vapor or H_2S = 1 mole%, with CO making up the balance.

Ten cylindrical pellets (1.52 cm in diameter and 1.90 cm long) formed in a hand press were sintered for 5 hr at several temperatures, then cooled slowly in the furnace, and their average compressive strengths were measured. The strain rate was 1×10^{-5} sec^{-1}. Remains of the crushed pellets were analyzed to determine their grain interface morphology and crystalline phases using scanning electron microscopy (SEM) and x-ray diffraction (XRD). Additionally, SiC with mulcoa powders (70/30) were sintered with zirconium oxide (ZrO_2) additive at 1500°C for 5 hr in air + 30% water vapor.

The most promising atmosphere and compound were then used to join bars of SiC. The blocks were 6 $\times$ 8 $\times$ 50 mm. After joining the shear strengths of the bonds were tested at elevated temperatures. Figure 1 shows a scheme of SiC blocks used for shear strength measurements.

2.3 Results and Discussion

2.3.1 The Effect of Atmosphere on the Sintering Propensities of Mullite and Mulcoa

Compressive strength development in high-purity mullite sintered at 1500°C for 5 hr in air and air + 30% water vapor is listed in Table 1. Generally, strength increases in specimens sintered in the presence of water vapor.

Figure 2 illustrates strength variation with both sintering temperature and water vapor content in air. Generally, there is a large increase of strength with water vapor content in samples sintered at 1500°C for 5 hr. The total porosity of sintered samples as estimated by automated image analysis of SEM backscattered electron images is constant at around 50%. This suggests that the strength increase may not be caused by the increased sintering resulting from enhanced reactivity of mullite particles, but is instead due to changes in the local fracture behavior of particle–particle interfaces.

To obtain additional information on interface behavior, the strength–strain relationship was analyzed. Figures 3 and 4 show the stress–strain curves for several pellets of mulcoa sintered at 1500°C in air + 30 vol% water vapor. The strength–strain curves imply that some ductility is associated with particle- free interfaces. Usually, the ductile interlayer will increase joint toughness and provide transverse crack stoppers.[13] The SEM analysis performed on polished sintered mulcoa showed that the intergranular phase is composed of silica and alumina, with about 7–17 wt% alumina and the remainder silica.

The presence of micropores in the sintered pellets modifies several physical properties, including elastic moduli, thermal conductivity and diffusivity, and dielectric constant and may also

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lead to an increase in fracture toughness.[14] Cracks and microcracks in the sintered mulcoa often formed in the intergranular phase while the strength tests were performed (Figure 5). The cracks should not form because of stresses created upon cooling as a result of the anisotropy of thermal expansion coefficients of different phases, since thermal expansion coefficients of SiC and mullite are nearly the same.

Generally, the reducing atmosphere has a small effect on strength increase in mulcoa pellets (Figure 6, Table 1). The small increase of strength under reducing conditions may be explained by the enhanced densification resulting from the formation of nonstoichiometric phases on the surface of mullite particles which, in turn, induce surface diffusion and further the densification rate. Mullite solid–solution can be represented by the formula $Al_{4+2x}Si_{2-2x}O_{10-x}$, where x is the number of oxygen atoms missing per unit cell ($0 \leq x \leq 1$).[15,16] The reducing atmosphere has an extremely low oxygen fugacity, which may further decrease the number of oxygen atoms in the structure and change both the sintering and strength parameters of the material.

2.3.2 The Effect of Atmosphere on the Sintering Propensity and Strength Development in SiC Powder

Figure 7 illustrates changes of compressive strength with sintering atmosphere and deformation temperature for SiC sintered at 1500°C for 5 hr. Water vapor and reducing atmosphere do not have a great effect on strength development in SiC. However, enhanced compressive strength is observed in samples sintered in air and deformed at room temperature and particularly at 1000°C. The enhanced strength seems to result from the mechanical properties of
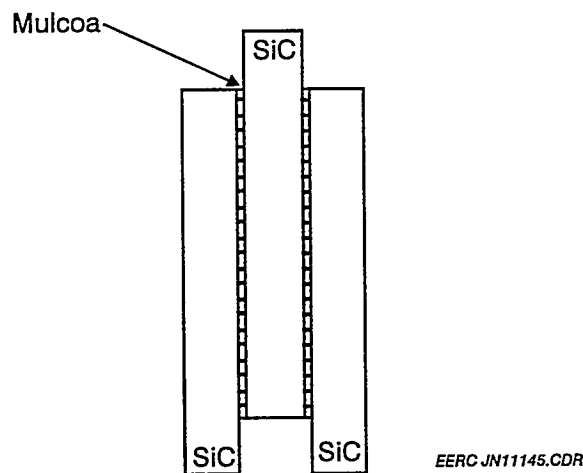

EERC JN11145.CDR

Figure 1.   Scheme of SiC bars used for measurements of shear strength.

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TABLE 1

Summary of Mechanical Properties of Mulcoa, SiC, and SiC–Mulcoa Composite

Material	Compressive Strength, MPa		Porosity, %	Atmosphere
	25°C	1000°C		
Mullite	60	62	NA[1]	Air
			NA	Air + 30% H_2O
Mulcoa	180	200	54	Air
	380	280	52	Air + 30% H_2O
	320	340	53	$H_2/CO/CO_2/H_2O$
	350	360		$H_2/CO/CO_2/H_2S$
SiC	590	920	2.2	Air
	340	420	3.5	Air + 30% H_2O
	290	310	4.8	$H_2/CO/CO_2/H_2O$
	320	330	5.5	$H_2/CO/CO_2/H_2S$
SiC + 30% Mulcoa	340	370	48	Air + 30% H_2O
SiC + 30% Mulcoa:ZrO_2, 3:1	280	360	NA	Air + 30% H_2O
SiC + 30% Al_2O_3	80	150	NA	Air + 30% H_2O
	Shear Strength			
SiC Bars/Mulcoa	200	190	NA	Air + 30% H_2O

[1] Not applicable.

the particle interfaces. XRD tests show the presence of cristobalite (SiO_2) in all samples sintered under both oxidizing and reducing conditions, which likely formed during oxidation of SiC. The total porosity of sintered samples was between 2.2% and 5.5%. Maximum compressive strength of Coors SiC, a well-sintered SiC with similar porosity (2.3%) is around 2110 MPa as determined by Coors.[17] The much lower strength of the pellets sintered at 1500°C implies that they should not be used in high stress situations but that the process may be used in the creation of weaker materials such as hot-gas particle filters.
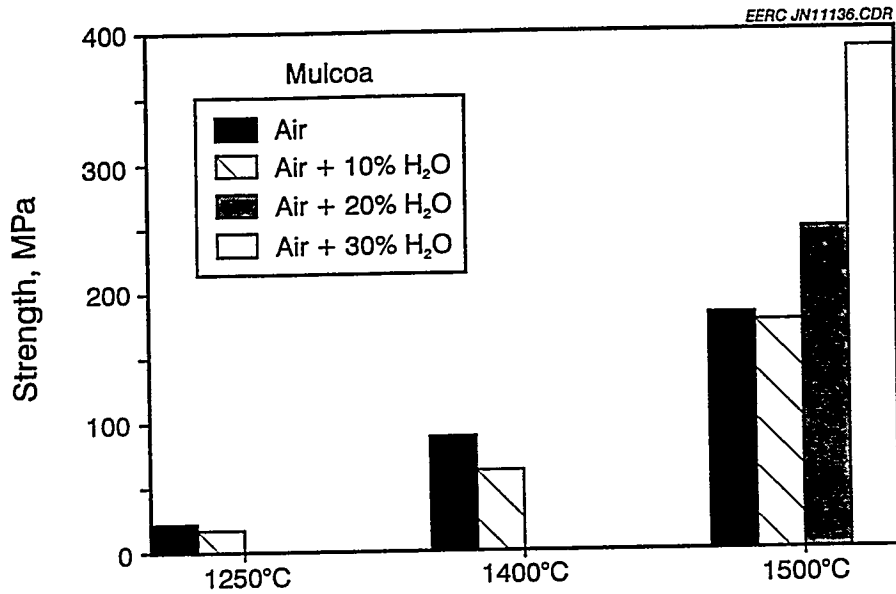
Figure 2. Strength development in mulcoa ceramic sintered in air and water vapor.

Figure 3. Strength–strain relationship in mulcoa sintered at 1500°C for 5 hr in air + 30% water vapor. The three samples were deformed at room temperature.
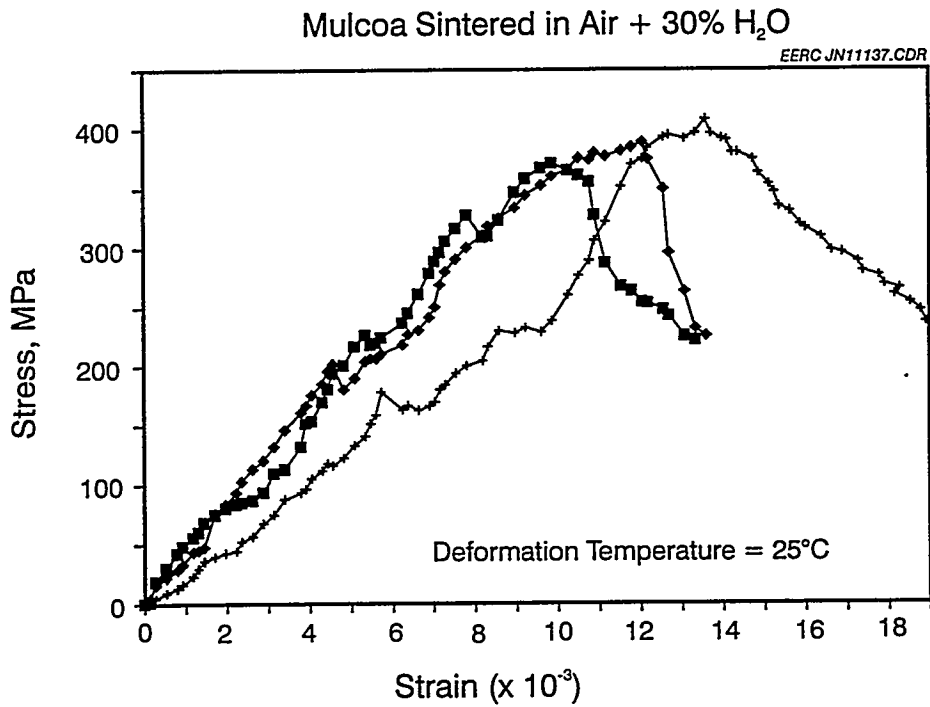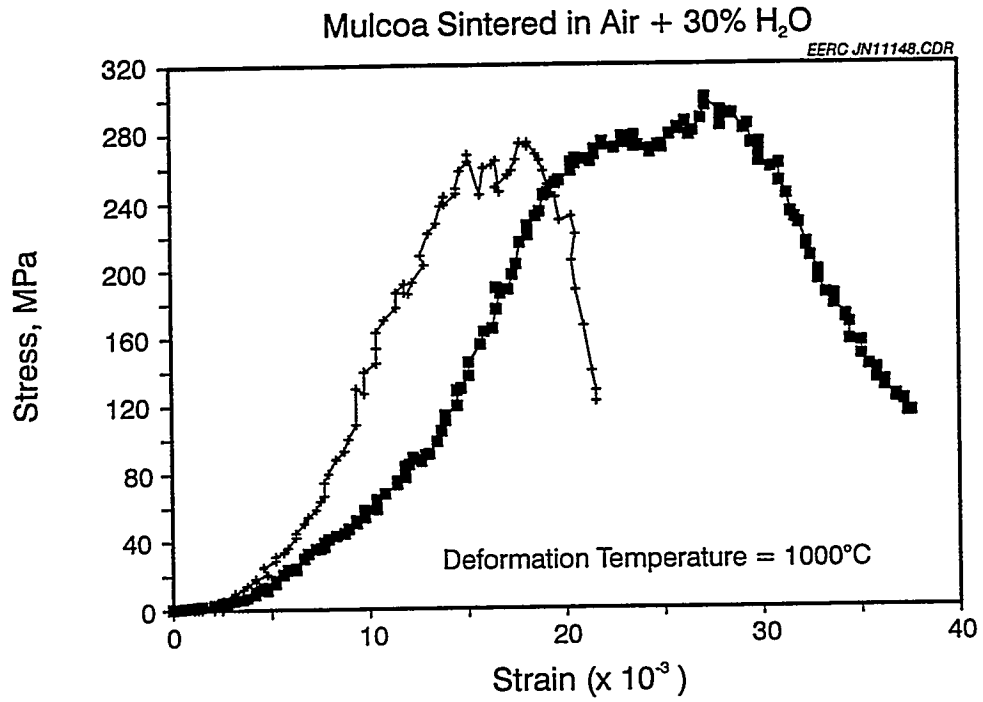
Mulcoa Sintered in Air + 30% H₂O

Figure 4. Strength–strain relationship in mulcoa sintered at 1500°C for 5 hr in air + 30% water vapor. The two samples were deformed at 1000°C.
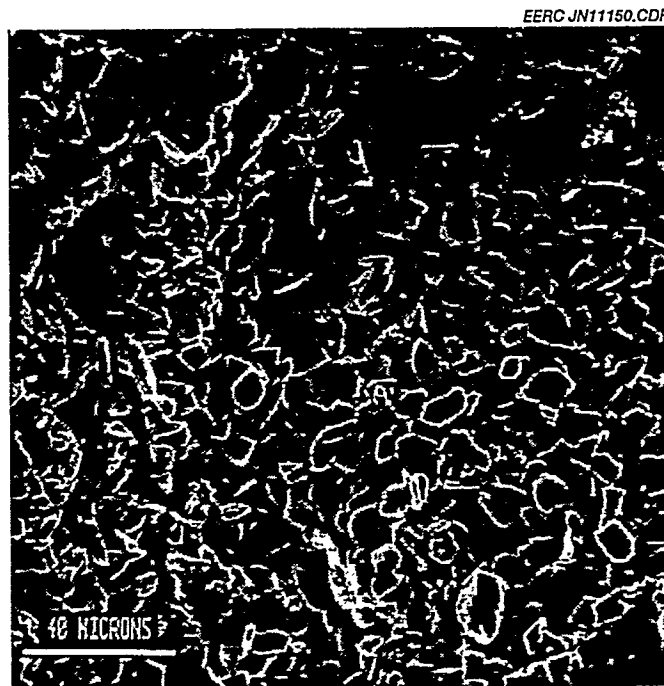

Figure 5. Microstructure of mulcoa sintered at 1500°C for 5 hr in air + 30% water vapor.
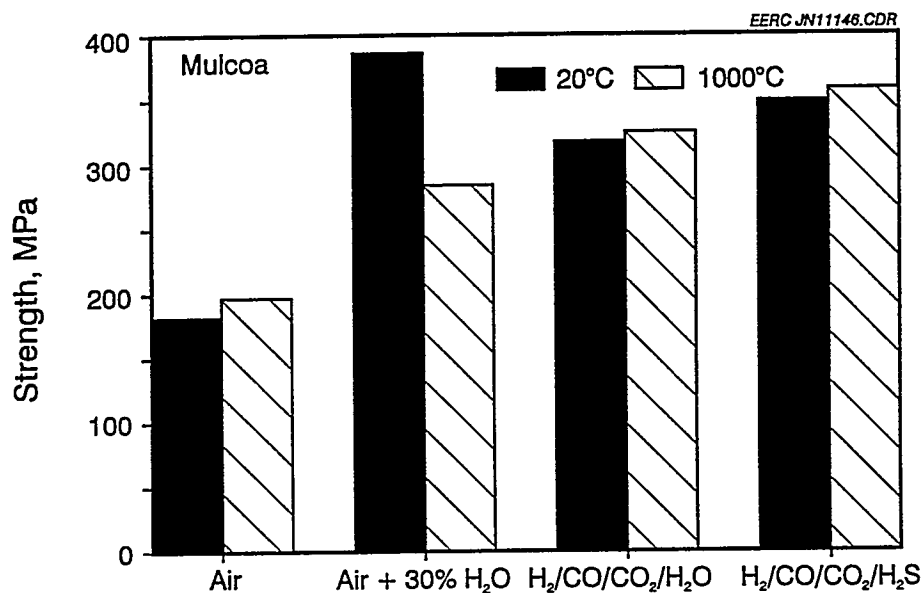
Figure 6. Strength development in mulcoa ceramic sintered in various atmospheres at 1500°C for 5 hr.

Figures 8 and 9 show that the change of Gibbs free energy with temperature for oxidation of SiC to SiO_2 and higher oxygen partial pressure over CO_2 than SiO_2, proves that silica may be formed under reducing conditions in the presence of carbon dioxide.

2.3.3 Strength Development in SiC–Mulcoa (70/30) Composite

Perhaps the most striking feature of this study is that the mechanical properties of composite pellets made from a 70/30 blend of SiC and mulcoa, sintered at 1500°C for 5 hr in air + 30% water are similar to those of the pure mulcoa pellets (Table 1). This similarity provides direct information about the role of the intergranular phase on the mechanical properties of the composite. Figure 10 illustrates the microstructure of the fractured composite, which has a total porosity of about 48%. The addition of a small quantity of ZrO_2 into the SiC–mulcoa composite does not change the mechanical properties of the material; however, XRD shows that an intermediate $ZrSiO_4$ phase forms. The formation of this phase at the surface of the material may reduce alkali-based corrosion of the material in a fossil energy application. The $ZrSiO_4$ layer would be resistant to spalling since it has a coefficient of thermal expansion very similar to that of the SiC and the mullite ($6.6 \times 10^{-6}/°C$, $5.3 \times 10^{-6}/°C$, and $5.1 \times 10^{-6}/°C$, respectively).

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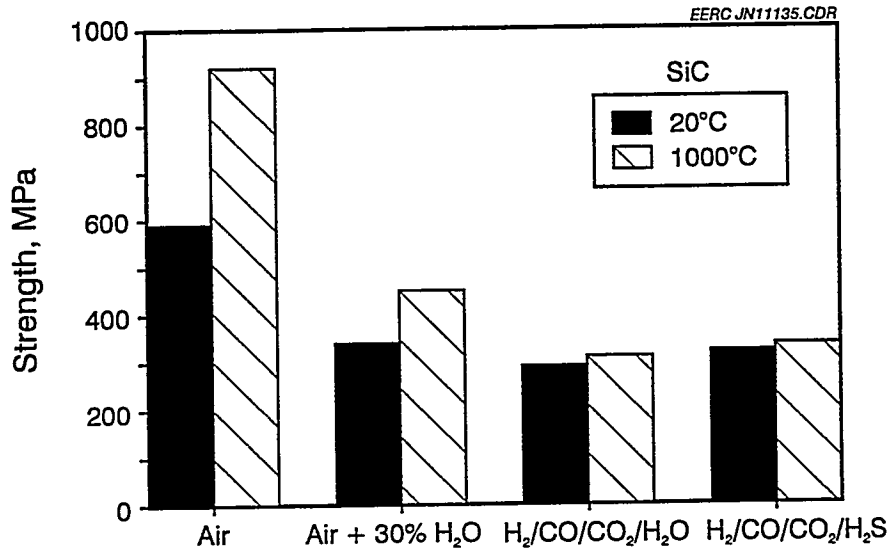Figure 7. The effect of atmosphere on strength development in SiC sintered at 1500°C for 5 hr.

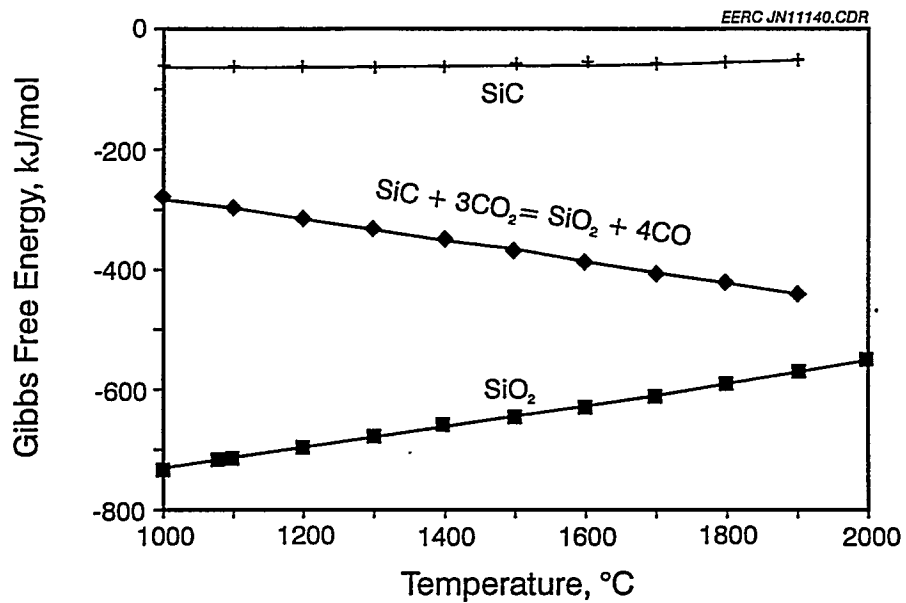Figure 8. The change of Gibbs free energy of formation with temperature for SiC, SiO_2, and SiC + CO_2.
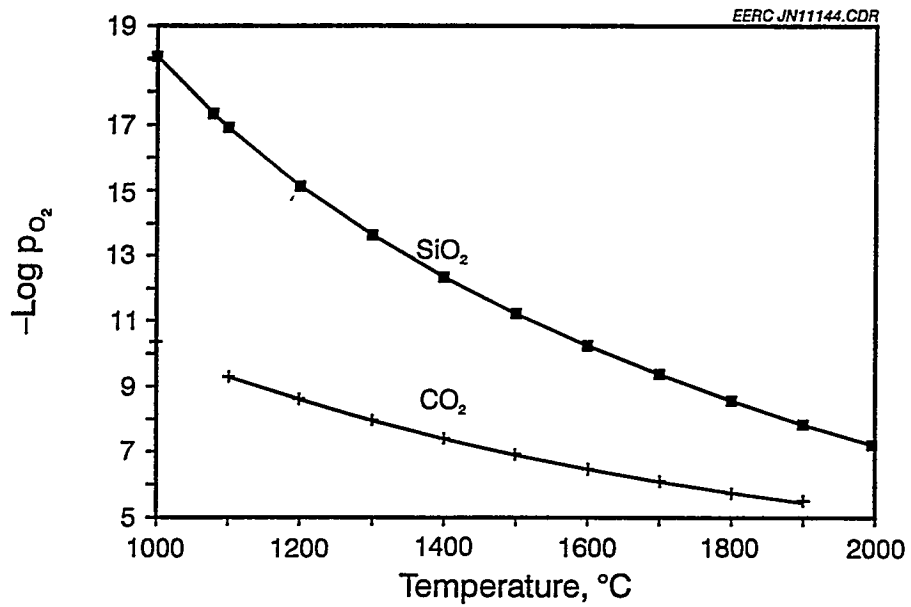
Figure 9. Partial pressure of oxygen (in −log scale) over SiO_2 and CO_2.

Figure 10. Microstructure of SiC−mulcoa (70/30) composite sintered at 1500°C for 5 hr in air + 30% water vapor.

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2.3.4 Mechanical Properties of Sintered Mulcoa and SiC–Mulcoa (70/30) Composite at 1400°C

The mechanical properties of mullite-containing composites are quite different at 1400°C as compared to the properties measured at room temperature or 1000°C. Figures 11 and 12 show the relationship between applied stress and pellet strain for mulcoa and SiC–mulcoa (70/30) composite determined at 1400°C. For mulcoa, the strength is significantly lower than that in the SiC—mulcoa composite, but shows some residual strength after fracture, as shown by the downward sloping curve after the maximum. This residual strength is important in reducing the rapidity with which a structure will fail, thereby reducing the damage caused by a failure. This residual strength or toughness is likely caused by numerous nonpropagating intergranular separations,[18] and this effect is likely to be most pronounced above 1200°C.[19] The lower strength of the mulcoa pellets at 1400°C arises from the sliding of the intergranular glassy phase.

The maximum strength of the SiC–mulcoa composite measured at 1400°C is only slightly lower than that measured below 1000°C. Also, there is more plasticity caused by grain-boundary sliding of the amorphous phase.
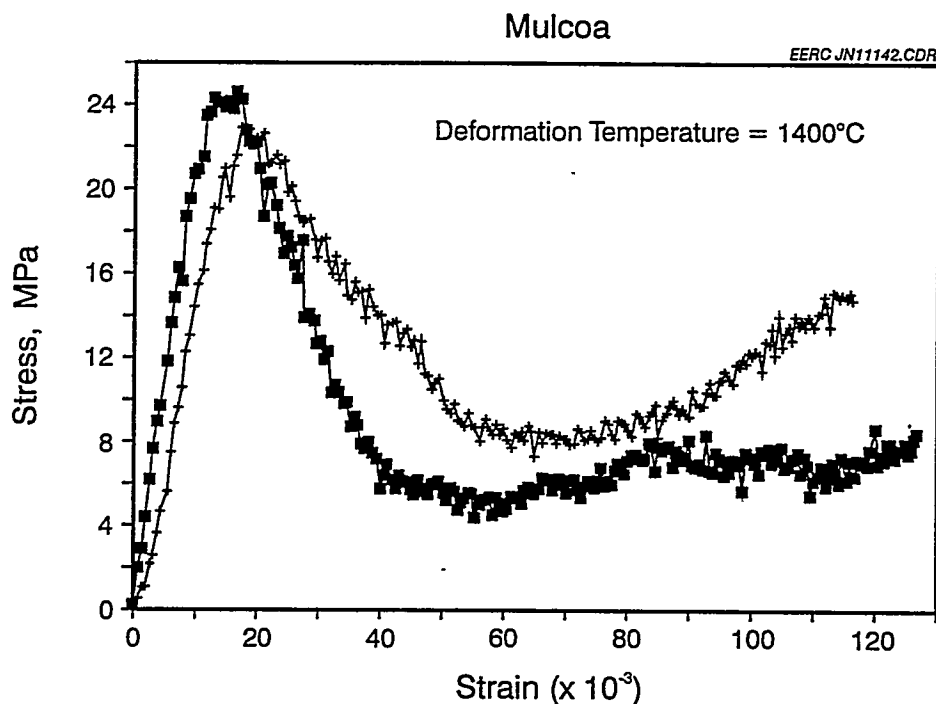

Figure 11.   Stress–strain curves in mulcoa specimens sintered at 1500°C for 5 hr in air + 30% water vapor.
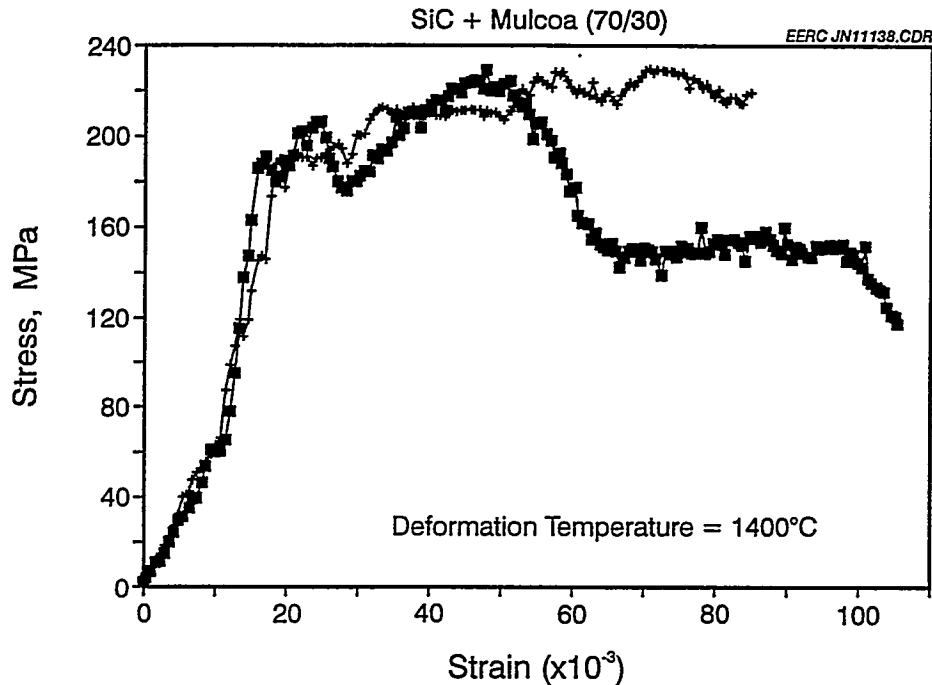
Figure 12. Stress–strain curves in SiC–mulcoa (70/30) specimens sintered at 1500°C for 5 hr in air + 30% water vapor.

2.3.5 Joining of SiC Bars with Mulcoa

Flexure test bars (6 × 8 × 50 mm) of Hexoloy SA sintered alpha silicon carbide manufactured by the Carborundum Company were joined along the 8-mm faces with mulcoa at 1500°C for 5 hr in air + 30% water vapor. The mulcoa was applied as a dry powder in a layer approximately 0.3 mm thick. No pressure was applied during sintering. The shear strengths of the joins measured at room temperature and at 1000°C are about 200 MPa (Table 1).

3.0 JOINING SILICON CARBIDE THROUGH JOULE HEATING

No matter what compounds are used in joining SiC, heat must be applied to the joint. Most often the heat comes from a secondary external source such as a furnace. Sometimes inductive heating or microwave heating is used. In all of these cases, the object needs to be surrounded by the heater. This is not a problem for smaller objects or those that are at least small in two dimensions, such as a pipe. However, for objects that are large in two dimensions, such as heat exchanger panels, such heating methods are not applicable. Therefore some preliminary work has been performed to determine whether it may be possible to heat SiC by passing an electric current through it. In such a scenario, electrodes would be connected to either side of the joint and an electric current passed through the material to cause resistive, or joule, heating. Silicon carbide is being focused on because it is an appropriate material with which to make a heat exchanger panels

and because it has a lower electrical resistance than most other ceramic materials. With proper attachment of the electrodes, and because of the high thermal conductivity of SiC, heating of the joint could be made very uniform. The method has an additional advantage in that the joint is placed in an electrical field which is known to reduce the temperature necessary for reaction between ceramic precursors, a technique known as combustion synthesis. This is an especially valuable attribute in cases where the joining is accomplished by reaction sintering.

A literature search was performed for articles on ceramic–ceramic joining written between 1986 and 1993. Very few articles were found on joining SiC, none employing joule heating. A number of articles have been published recently on the joining of silicon nitride employing joule heating, primarily by K. Okuda and others in Japan.[20–24]

In the so-called "electrical joining method" used by Okuda, welds are made by passing electric current through a joining compound between two ceramic monoliths. Heat is created in the joint by the resistance to the high voltage current. The current is applied through electrodes implanted all around the joint, and it is heated to the sintering temperature of the compound. However, ensuring even current flow so that sintering is uniform across the joint may not be possible if the joint is only accessible from one side, such as when an installed heat exchanger panel is being repaired. Therefore, we propose to use the ceramic itself as the electrode and pass current through the ceramic to the joint, assuring a much more even distribution of heat.

To ensure that the joint has the greatest possible high-temperature strength, SiC will be used as the joining material. However, SiC does not pressureless sinter below 2000°C, a temperature too difficult to obtain uniformly in a large joint. In addition, unless air is excluded, the oxidation of SiC becomes excessive above 1600°C. Therefore we propose to mix SiC powder with Si and C powders and reaction sinter the material in a manner similar to the method employed by Rabin and Moore.[25] Unlike Rabin and Moore, we intend to use Si powder mixed with the SiC and C rather than infiltrating the Si from an exterior source as they did. Using a powder means that the regions of unreacted Si are less likely to be continuous, and so resistance to coal slag corrosion should be much higher, and strength should remain high above the melting point of the silicon (1400°C), two problems associated with infiltrating the Si.[26] However, using powdered Si will cause the joining material to experience some shrinkage during sintering, so we intend to start with a more dense wafer of green material than the ceramic tape employed by Rabin and Moore.

The first step in determining the feasibility of the joining technique is to determine whether the SiC pieces to be joined can be used as the electrodes to carry the current. One of the biggest problems is obtaining good contact between the power source and the SiC, because the SiC is so hard and usually is covered by a thin, nonconducting SiO_2 layer. A number of experiments were tried, including etching the surface with hydrofluoric acid to remove the SiO_2 layer, pyrolizing the surface to create a graphite layer, melting on a layer of indium metal, and rubbing on copper and aluminum metal. However, the most effective was simply coating each end of the SiC test piece with silver paint.

Table 2 shows the resistance of a 6 × 8 × 50-mm bar of Hexoloy SA sintered alpha silicon carbide made by the Carborundum Company. The table shows that the resistance of the bar drops dramatically with temperature. For a given applied voltage, the current passing through the bar is determined by its resistance as given in Ohm's Law (Equation 1):

$$V = IR \tag{1}$$

where   V = voltage

      I = current

      R = resistance

However, the power or heat produced varies as Equation 2:

$$P = I^2R \tag{2}$$

where   P = power

Therefore, when a constant voltage power source such as a variac is used to control the current, current runaway occurs as the bar is heated. This happens because for a fixed voltage that causes heating of the sample, the resistance drops as the temperature is raised, causing the current to increase (Equation 1). However, as the current increases, the power output increases, even though the resistance drops (Equation 2), which raises the temperature, which drops the resistance, causing a further current and temperature rise. This leads to extremely rapid temperature escalations (approximately 300° to 500°C/second) causing thermal shock and breakage of the bars. Therefore, a current-limiting circuit must be constructed for the power source.

4.0   FUTURE WORK

Work and milestones are complete for calendar year 1994. In calendar year 1995, the materials work will concentrate on joining of SiC as described in Section 3.0 and on silicon nitride joining with SiAlON.

TABLE 2

Resistance of a 5-cm-long Bar of Hexoloy SA versus Temperature

Temperature, °C	Resistance, kohms
110	120
125	25
150	16
200	2
250	0.8
300	0.28

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