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# HOT CORROSION OF NICALON FIBER REINFORCED GLASS - CERAMIC MATRIX COMPOSITES: MICROSTRUCTURAL EFFECTS

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12a. DISTRIBUTION / AVAILABILITY STATEMENT  <b>Approved for Public Release, Distribution is Unlimited</b>		12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  Continuous Nicalon fiber-reinforced calcium aluminosilicate (SiC/CAS) and lithium aluminosilicate (SiC/LAS) are two glass-ceramic composites that show promise for turbine engine applications. A study was undertaken to determine the hot corrosion resistance of these two composites. The composites were coated with 3.0mg/cm <sup>2</sup> of sodium sulfate and atmospherically exposed at 900°C from 1 to 100 hours. Reactions appeared to initially occur at the exposed fibers in both systems. In the CAS system, the dissolution of the matrix occurred with the formation of CaSiO <sub>3</sub> and NaAlSiO <sub>4</sub> . The LAS system studied (UTRC200) was doped with B <sub>2</sub> O <sub>3</sub> . Apparently the boron oxide glass reacted at 900°C and formed a protective layer around the Nicalon fiber. After exposure, two additional phases (SiO <sub>2</sub> and an amorphous phase) in the LAS system were detected. A thin surface layer of molten sodium sulfate appeared to have little effect on the LAS system since exposure at the same temperature without sodium sulfate produced similar microstructures and diffraction patterns. However, when the same specimen was subjected to a hot corrosion crucible test, where the specimen is completely immersed in molten salt, it was severely attacked within one hour of exposure.			
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**KEYWORDS**

Ceramic Composites

Glass-Ceramic

Hot Corrosion

Nicalon

Silicon Carbide

Calcium Aluminosilicate

Lithium Aluminosilicate

Sodium Sulfate

## INTRODUCTION

The efficiency of Naval aircraft can be improved by increasing the working temperature of the gas turbine engine and by reducing the overall weight of the aircraft. Today's nickel-based superalloys cannot withstand the stoichiometric burn temperature\* of the fuel resulting in a less efficient burn temperature. However, some monolithic ceramics, such as  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$ , can withstand these extreme temperatures without severe oxidation or degradation in mechanical properties. Additionally, the density of ceramics is less than conventional alloys. Monolithic ceramics, however, are inherently brittle and subsequently have very limited use in turbine engines. To overcome this problem, ceramic matrix composites have been introduced with continuous fibers. The addition of the fibers increases the material's toughness so that brittle (catastrophic) failures can be avoided. It has been proposed to incorporate ceramic matrix composites into turbine engines, but limited information is available on the effects of the environment on these materials.

The environment that Navy aircraft components are exposed to is very hostile. Salts, such as  $\text{Na}_2\text{SO}_4$ , tend to deposit on the engine components and hot corrosion can occur. Hot corrosion is a chemically driven process that degrades and removes material from the substrate. This corrosion process takes place in the temperature range between the melting point of the salt and its dew point. The melting temperature of sodium sulfate is  $884^\circ\text{C}$ , and the dew point is a function of pressure. The pressure experienced in gas turbine engines may vary from 5 to 20 atmospheres<sup>1</sup> and an increase in pressure raises the dew point temperature. Therefore, hot corrosion can occur at temperatures as high as  $1200^\circ\text{C}$ , which would be above its dew point at 1 atmosphere pressure.

Continuous Nicalon fiber-reinforced calcium aluminosilicate (SiC/CAS) and lithium aluminosilicate (SiC/LAS) are two glass-ceramic composites that show promise for turbine engine applications. They exhibit high temperature oxidation resistance with a modest decrease in

\* Stoichiometric burn temperature is a complex function pressure, fuel to air ratio, and fuel type.

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mechanical properties<sup>2,3</sup>. However, the hot corrosion behavior of these glass-ceramic matrix composites is not well understood. It is imperative to understand the interactions between the engine environment and the ceramic composites. This type of information will enable glass-ceramic composites to be designed so that they can be utilized in Navy gas turbine engines. This report presents the results of a microstructural study to investigate the hot corrosion resistance of these two glass-ceramic matrix composites. The companion report, "Hot Corrosion of Nicalon Fiber Reinforced Glass-Ceramic Matrix Composites: Mechanical Properties", will address the effects of hot corrosion on the mechanical properties of these composites.

### EXPERIMENTAL PROCEDURES

#### MATERIAL

Both composites studied (SiC/CAS, SiC/LAS) consisted of 8 plies of continuous unidirectional Nicalon SiC fibers with volume fraction of fibers of 38% for CAS and 40% for LAS. The CAS and LAS composites were received from Corning Inc. and United Technologies Research Center (UTRC) respectively. The LAS system is reinforced with low oxygen Nicalon fibers in a UTRC200 matrix. The UTRC200 matrix has  $B_2O_3$  incorporated with the LAS base glass and reacts with the fiber during consolidation to form a boron/carbon interface. The addition of boron at the interface is intended to help reduce oxidation of the SiC fibers by plugging the oxygen diffusion path during matrix cracking. However, the boron additions tend to lower the maximum use temperature of the composite to 930°C. The SiC/CAS system consists of a CASII matrix reinforced with the standard Nicalon fiber and has a reported maximum use temperature of 1300°C<sup>4</sup>. The mechanical properties and composition of these composites are listed in Table I. The effects of hot corrosion on the mechanical properties of SiC/CAS composite are reported elsewhere<sup>5</sup>.

TABLE I

MECHANICAL PROPERTIES AND CHARACTERISTICS OF  
SiC/CAS AND SiC/LAS AND SiC FIBER

	SiC/CAS II	SiC/LAS	SiC
Nominal Composition	CaO Al <sub>2</sub> O <sub>3</sub> 2SiO <sub>2</sub>	Li <sub>2</sub> O Al <sub>2</sub> O <sub>3</sub> SiO <sub>2</sub>	SiC
Maximum Use Temperature	1300-1350°C	930°C	1200°C
Predominate Crystal Phase	Anorthite	B-quartz/silica solid solution	B SiC
Density, g/cm <sup>3</sup>	2.7	2.5	2.55
Elastic Modulus, Msi	18.0	19.7	28
Tensile Strength, Ksi	73	51	430
Failure Strain %	0.7	0.25	1.5

**TESTS**

The materials were cut into 1cm square specimens and coated with approximately 3.0 mg/cm<sup>2</sup> of sodium sulfate with the aid of an air brush. This coating thickness is equivalent to the expected coating on a turbine engine component after 500 hours at 900°C with a sulfur fuel impurity level of 0.05%<sup>6</sup>. Specimens were weighed and then placed into a furnace at 900°C (15°/min ramp at 1 atm) for 1-, 10-, and 100- hour exposures. Upon removal from the furnace, the specimens were weighed and an X-ray diffraction scan was performed to identify the surface phases. Optical and scanning electron microscopes were also utilized to study the surface of the composites. For comparison purposes, a set of specimens were exposed at 900°C for 100 hrs. without a salt coating.

The specimens were cut perpendicularly to the fiber axis to determine the depth of penetration of the secondary phases that formed

and to determine if fiber damaged had occurred. X-ray images from a scanning electron microscope were utilized to help establish the chemistry of the phases present.

An additional set of specimens were subjected to a molten crucible test for 1 and 10 hours at 900°C and the same analytical analysis, as mentioned above, was performed to help determine degradation mechanisms. The specimens in this group were completely immersed in the molten salt during the entire exposure. The specimens and environmental exposures is given in Table II. All of the specimens were exposed to 900°C for varying lengths of time.

TABLE II

## SUMMARY OF SPECIMENS AND EXPOSURE CONDITIONS

MATRIX	REINFORCEMENT	SALT	
		EXPOSURE	TIME OF EXPOSURE (Hrs)
CAS	SiC	Film	1
CAS	SiC	Film	10
CAS	SiC	Film	100
CAS	SiC	None	100
CAS	None	Film	100
CAS	SiC	None	100
CAS	SiC	Crucible	1
CAS	SiC	Crucible	10
LAS	SiC	Film	1
LAS	SiC	Film	10
LAS	SiC	Film	100
LAS	SiC	None	100
LAS	SiC	Crucible	1
LAS	SiC	Crucible	10

## RESULTS AND DISCUSSION

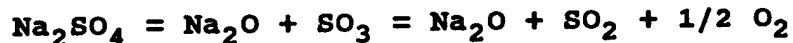
## SiC/CAS

A micrograph of the as-received SiC/CAS composite is presented in Figure 1a. It can be seen that the silicon carbide fibers are exposed on the specimen's surface. After a 1-hour heat treatment at 900°C, a reaction took place at the exposed fibers, as seen in Figure 1b. Analysis of the X-ray profiles presented in Figure 2, indicates the presence of two new phases: calcium silicate ( $\text{CaSiO}_3$ ) and sodium aluminum silicate ( $\text{NaAlSiO}_4$ ). This was further verified by X-ray image analyses of the surface and of the cross section as seen in Figures 3 and 4. The calcium rich phase is depleted of sodium and aluminum while the other phase is rich in Al and Na. Therefore, a possible reaction sequence is as follows:

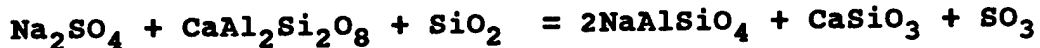
Initially fibers exposed to the surface are oxidized:



Sodium sulfate forms sodium oxide by the following reaction at elevated temperatures:



Finally, the residual silica in the matrix in conjunction with the silica from the fiber oxidation, reacts with the salt and matrix:



Gas evolution, which may be  $\text{SO}_3$ ,  $\text{SO}_2$ ,  $\text{O}_2$  or  $\text{CO}$ , forms bubbles in the newly formed phases Figures 1b and 5.

The X-ray diffraction patterns of samples tested for 1-, 10-, and 100- hours are similar and indicate that the same reaction products have formed. The specimen exposed for 1 hour had the same phases present as the longer exposures (10 and 100 hr.) but the relative intensities of the peaks were not the same. This indicates that phases formed during hot corrosion continue to form and crystallize with time producing a narrow high amplitude peak. The surface morphology can be seen in a scanning electron micrograph, Figure 5. The newly formed phases, from the exposure, are very brittle and numerous cracks have formed on the surface, Figures 5 and 6. Examination of the cross-sections of the 100-hour-exposed specimens revealed that the matrix material has been

attacked by the molten salt. The elemental profile obtained from an energy dispersive spectrometer indicates that the fibers exposed on the surface of this specimen are severely attacked, Figure 4. The sulfur map did not indicate a gradient near the surface. This indicates that residual sodium sulfate was not present.

The X-ray diffraction data obtained from the noncoated specimen (exposed for 100 hours at 900°C) indicated that no new surface phases formed during the heat treatment. This can also be seen from the X-ray map of the cross-section of this specimen, Figure 7. The surface morphology of the uncoated specimens was similar to the as-received specimen except for the degradation of the reinforcing SiC fibers that were initially exposed to the surface.

In summary, the SiC/CAS composite exhibited little surface deterioration in a salt-free environment up to 900°C. However, the salt coated ( $\text{Na}_2\text{SO}_4$ ) specimens behaved differently. As mentioned above, two new crystalline phases were formed on the composite surface due to salt attack. Also it appears that the silica that normally protects the silicon carbide fibers from further oxidation reacts with the salt and takes part in the decomposition of the matrix by the proposed equation above. This results in the complete degradation of the fibers at the specimen's surface. This finding agrees with the work of Tressler et al<sup>7</sup> and of Henager et al<sup>8</sup>, in that SiC is very susceptible to molten salt ( $\text{Na}_2\text{SO}_4$ ) attack.

A molten crucible test was performed on the composite (SiC/CAS). This is a severe hot corrosion test that has been used in the past to evaluate turbine engine components. It should be noted that since the specimen is completely immersed in molten salt, the partial pressures of the constituents, most notably oxygen and  $\text{SO}_3$ , are different when compared to the salt coated hot corrosion specimens mentioned above. Therefore, it is expected that the thermodynamics and kinetics of the reactions change, and subsequently different reactions may occur. Sodium aluminosilicate ( $\text{NaAlSiO}_4$ ) was detected on the 10-hr. specimen, but calcium silicate did not form. The surface of the specimen was more severely attacked when compared to the coated specimens Figure 8. This

agrees with the work performed by D.W. McKee et al.<sup>9</sup> on hot corrosion of sintered SiC, in that at very low partial pressure of O<sub>2</sub>, the SiC corroded very rapidly because the protective SiO<sub>2</sub> layer fluxed into the melt. This shows the importance of the selection of the test method. In other words, it is imperative to simulate the engine environment as closely as possible. Bianco et al.<sup>10</sup> completed similar work with the hot corrosion of cordierite. They, however performed burner rig test and determined that a good correlation exists between burner rig tests and thin coating furnace test. Therefore, it is not recommended to use crucible tests for hot corrosion testing.

The matrix material (CASII), without the SiC fiber reinforcement, was also exposed at 900°C for 100 hours with and without a salt deposit. The purpose of this study was to determine the influence of the SiC fibers on the hot corrosion process of the composite. The exposure at 900°C for 100hr. did not change the surface chemistry of the CASII material. However, the salt coated specimen (900°C/100hr) produced NaAlSiO<sub>4</sub> and possibly (Na<sub>.8</sub>Ca<sub>.1</sub>)<sub>2</sub>SO<sub>4</sub>. This indicates that the surface chemistry of the composite is influenced by the presence of the SiC fibers during hot corrosion.

#### SiC/LAS

A comparison study between an uncoated and salt coated specimen was also undertaken with the LAS system at 900°C for 100 hours. The surface morphologies of these specimens after exposure were similar. The 1-, 10-, and 100-hour-exposed specimens, it appears that an amorphous phase, possibly boron oxide, leached from the matrix, Figure 9. The initiation site of the reaction appears to be localized at the exposed fibers. It is postulated that the fiber initially oxidizes and forms SiO<sub>2</sub> and additionally, the Boron reacts to form B<sub>2</sub>O<sub>3</sub> on the surface of the specimen.

The addition of sodium sulfate did not promote formation of additional phases. This was determined by comparing the two specimens

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with a 100-hour heat treatment (coated and uncoated specimens). The X-ray diffraction profiles of these two specimens were similar. The surfaces of the exposed specimens are shown in figures 9a and 9b. Note that the surfaces are very porous and numerous cracks are observed. X-ray maps of the cross-sections of these specimens indicate that Na diffused into the matrix from the exposed surface, and Mg which is a constituent of the matrix, diffused to the surface, Figure 10. The Mg rich phase may possibly be  $Mg_2SiO_4$ , but has not been detected with X-ray diffraction techniques.

This SiC/LAS composite system behaved differently when it was exposed to a molten crucible test. The composite was severely attacked, in that the specimen dissolved in the salt ( $Na_2SO_4$ ) after 10 hours at  $900^\circ C$ . After a one hour exposure, in the crucible test, the specimen was intact, but the surface was severely attacked, figure 11. Therefore, it has been shown that thin film hot corrosion results differ from crucible test results of the same material. It has been suggested by N.S. Jacobson et al.<sup>11</sup>, that oxygen potential at the melt/specimen interface of a crucible test may be unrealistically low. This in turn alters the kinetics of the system and does not simulate what may be expected in a turbine environment.

### CONCLUSIONS

From the results of this study, the following observations are made.

#### SiC/CAS

- 1 The calcium aluminosilicate matrix is damaged by molten sodium sulfate.
- 2 The dissolution of the matrix initiates at the exposed fibers and forms  $CaSiO_4$  and  $NaAlSiO_3$ .

SiC/LAS

- 1 The deposition of a thin layer of molten sodium sulfate apparently has very little effect on the surface morphology or composition after exposure at 900°C.
- 2 The composite is severely attacked when immersed in molten Sodium Sulfate for more than 10 hours at 900°C
- 3 A brittle phase forms on the surface of the specimen which initiates at the exposed fibers.

FUTURE WORK

None of the specimens studied, excluding the molten crucible specimens, had any detectable residual sodium sulfate present after the heat treatment. This indicates that all of the sodium sulfate may have reacted within the first hour of testing. Further investigation in this area is needed and may entail recoating the specimens with salt during the test. This would more closely simulate the conditions experienced in turbine engines. Burner rig tests will also be performed on these glass-ceramic composite systems.

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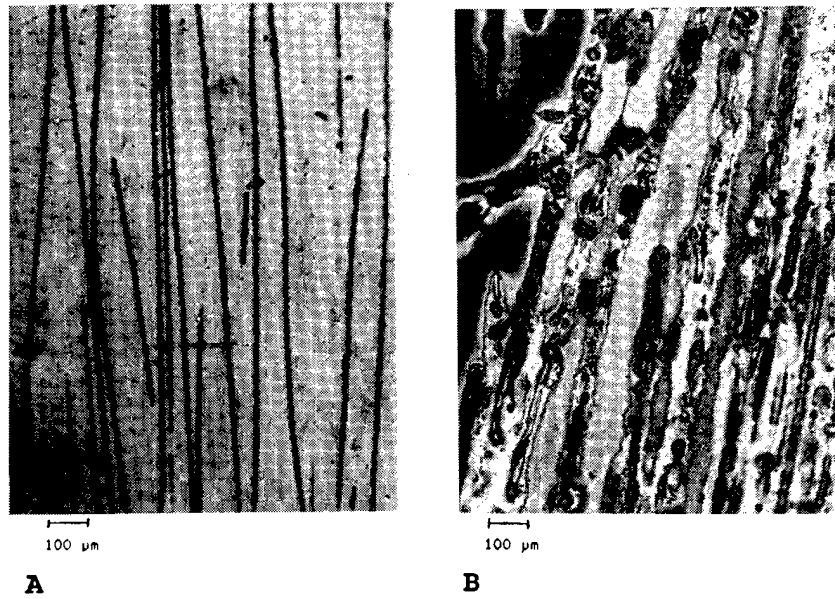


Figure 1) (A) As received SiC/CAS composite. Note the exposed fibers on specimen surface. (B) Same as (A) after 1hr. exposure at 900°C with  $\text{Na}_2\text{SO}_4$  coating.

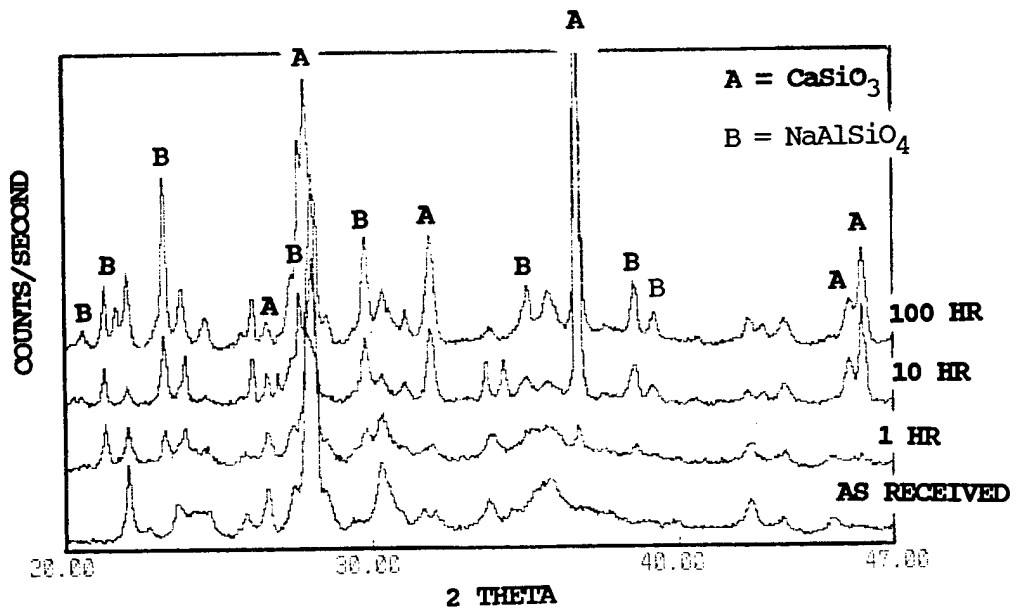


Figure 2) X-ray diffraction pattern of SiC/CAS composite. Exposures were at 1-, 10- and 100-hours at 900°C with  $\text{Na}_2\text{SO}_4$  coating.

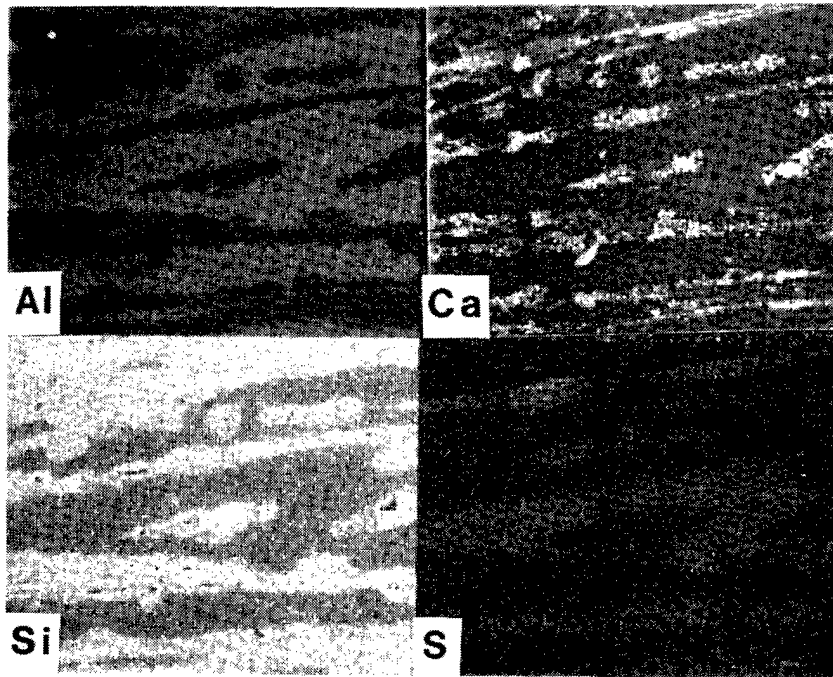


Figure 3) X-ray images taken of the surface of the SiC/CAS composite which was coated with  $\text{Na}_2\text{SO}_4$  and exposed at  $900^\circ\text{C}$  for 1 hr.

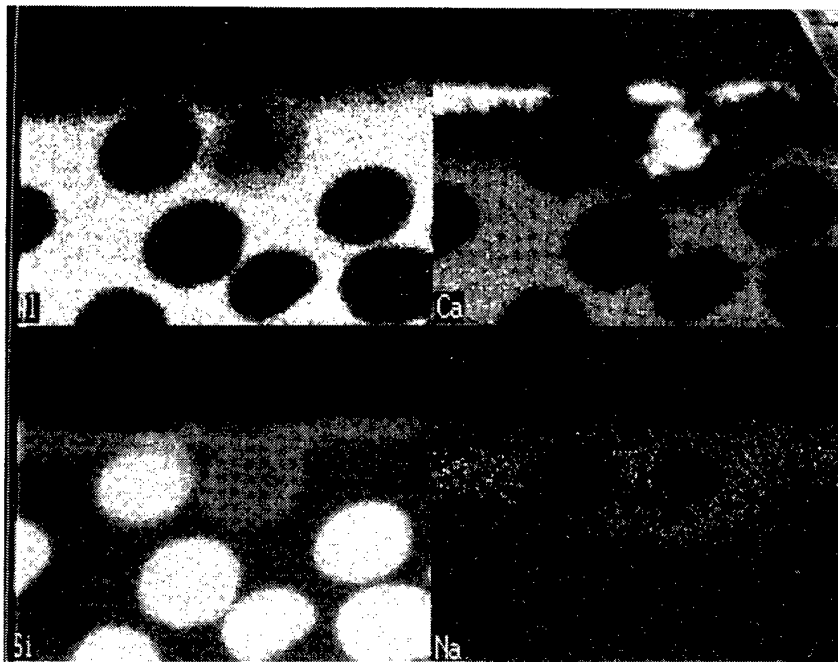
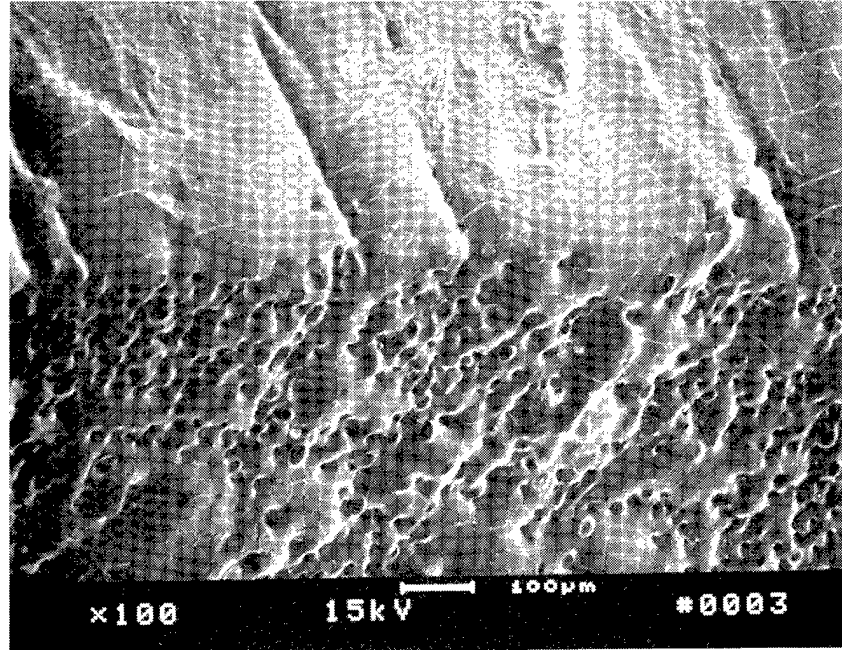
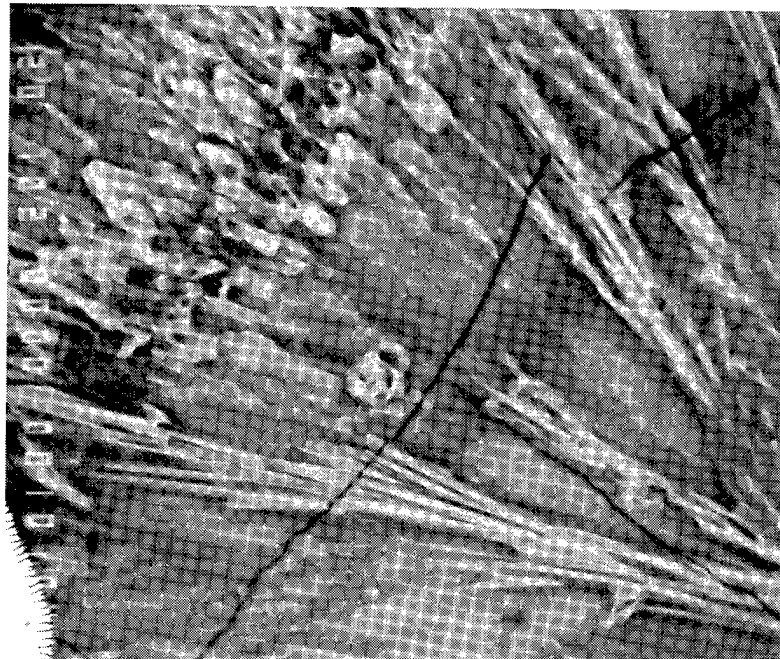


Figure 4) Elemental map taken of the cross-section of the SiC/CAS composite which was exposed to a salt coating at  $900^\circ\text{C}$  for 100 hrs.



**Figure 5)** SEM image of SiC/CAS composite which was salt coated and exposed for 10 hr. at 900°C. Note the surface cracks and pores.



**Figure 6)** Backscattered SEM micrograph of SiC/CAS composite that was coated with  $\text{Na}_2\text{SO}_4$  and exposed at 900°C for 100 hrs.

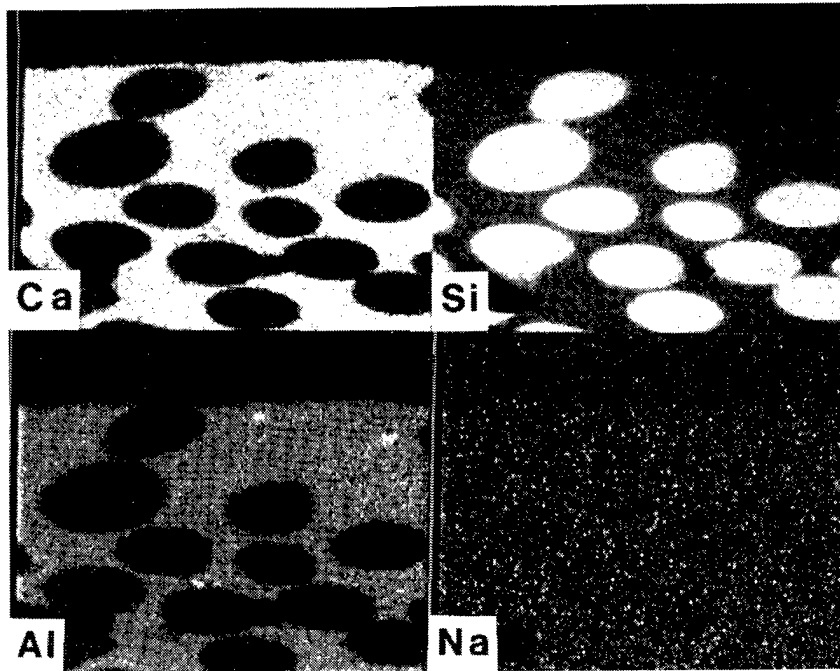


Figure 7) Elemental map taken of the cross-section of the SiC/CAS composite which was exposed at 900°C for 100 hrs. Note that a reaction zone as seen in figure 4 is not present.

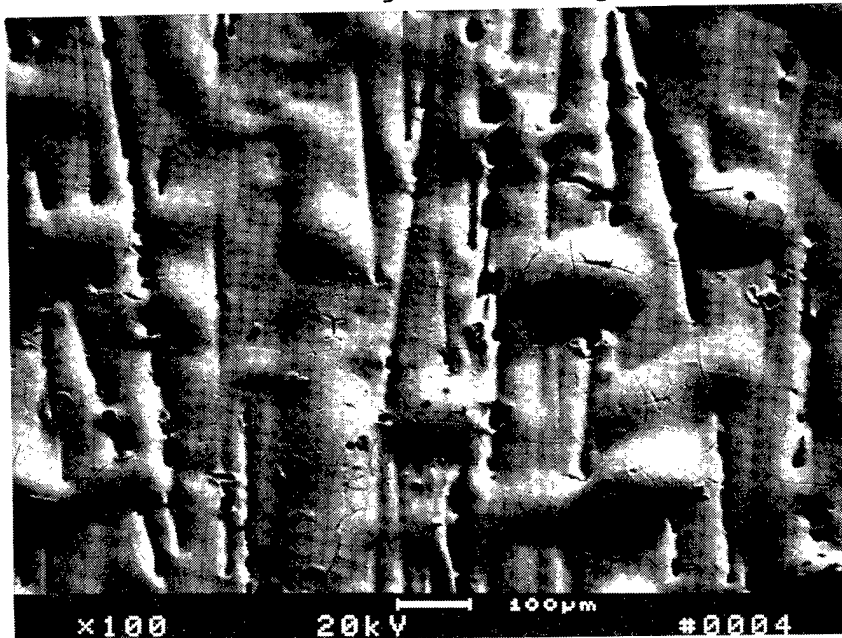
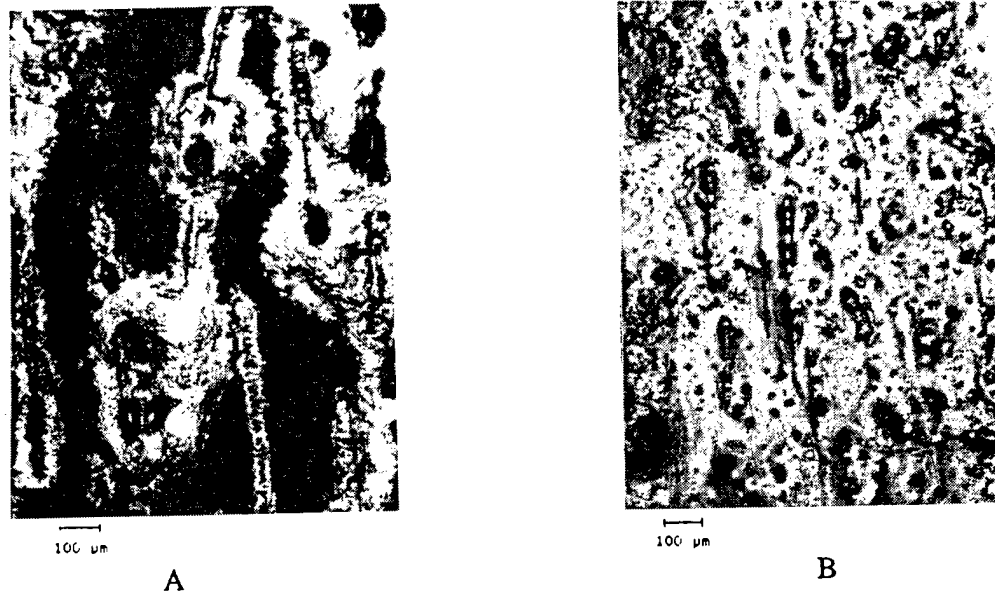
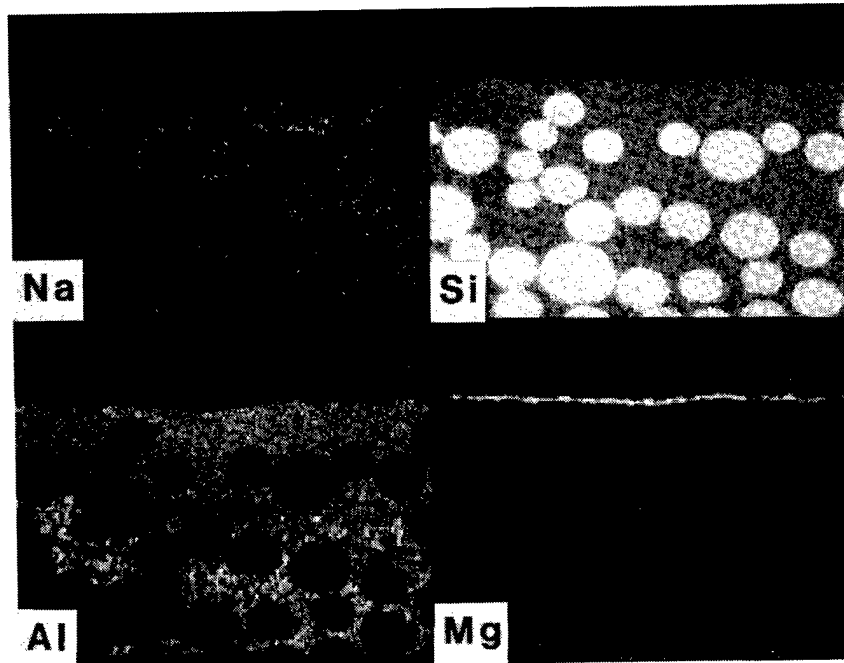


Figure 8) SEM micrograph of the SiC/CAS composite which was exposed to a molten crucible test for 1hr at 900°C. Note the severity of surface attack, and the formation of an amorphous phase.



**Figure 9)** (A) SiC/LAS composite coated with  $\text{Na}_2\text{SO}_4$  and exposed for 1 hr at  $900^\circ\text{C}$ . (B) Same as (A) but exposed for 100hr.



**Figure 10)** X-ray map of cross-section of SiC/LAS composite that was exposed at  $900^\circ\text{C}$  for 100 hours with a salt coating.

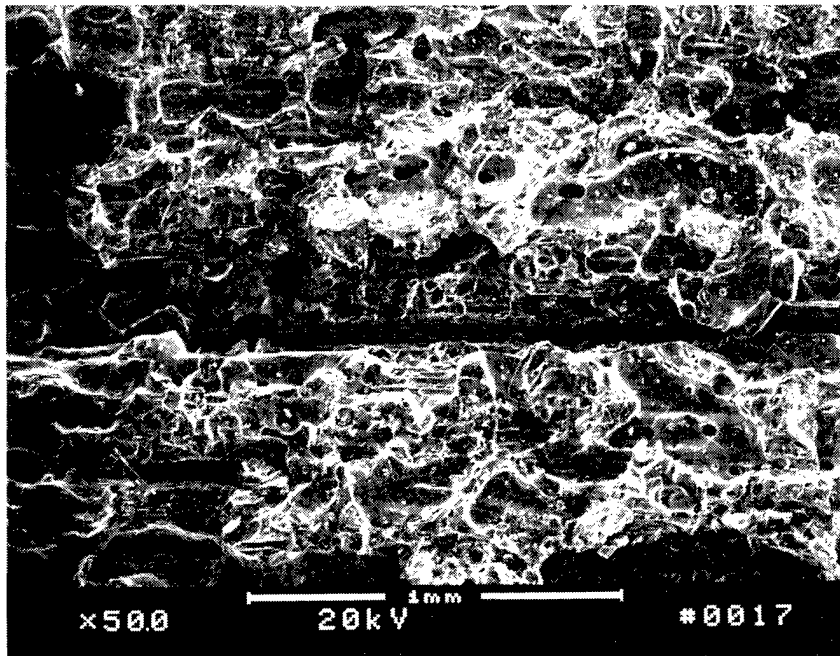


Figure 11) Surface of SiC/LAS composite that was exposed in a molten crucible test for 1 hour.



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