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Compatibility of SiC and SiC Composites with Molten Lead

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Abstract

The choice of structural material candidates to contain Lead at 1000°C are limited in number. Silicon carbide composites comprise one choice of possible containment materials. Short term screening studies (120 hours) were undertaken to study the behavior of Silicon Carbide, Silicon Nitride, elemental Silicon and various Silicon Carbide fiber composites focusing mainly on melt infiltrated composites. Isothermal experiments at 1000°C utilized graphite fixtures to contain the Lead and material specimens under a low oxygen partial pressure environment. The corrosion weight loss values (grams/cm² Hr) obtained for each of the pure materials showed SiC (monolithic CVD or Hexoloy) to have the best materials compatibility with Lead at this temperature. Increased weight loss values were observed for pure Silicon Nitride and elemental Silicon.

For the SiC fiber composite samples those prepared using a SiC matrix material performed better than Si₃N₄ as a matrix material. Composites prepared using a silicon melt infiltration process showed larger corrosion weight loss values due to the solubility of silicon in lead at these temperatures. When excess silicon was removed from these composite samples the corrosion performance for these material improved. These screening studies were used to guide future long term exposure (both isothermal and non-isothermal) experiments and Silicon Carbide composite fabrication work.

1. Introduction

Ceramic-matrix composites and those with continuous ceramic fiber reinforcement offer advantages over classical ceramic materials for improving thermal cycle resistance, corrosion enhancements, and mechanical properties. Applications for these ceramic composite materials include rocket motors, jet engines, space vehicles, brake disks, and orthopedic appliances. This wide range of applications (thermal, structural, and environmental) reflects the versatility and “engineerability” of these materials [1]. Silicon Carbide (SiC) and Silicon Carbide fiber matrix composites are a class of these ceramic matrix composite materials which provide improved material strength and fracture properties at elevated temperatures. These composite materials have good corrosion resistance (in oxygen environments) and have low activation characteristics which make their use in radiation environments attractive. SiC ceramic composites are one of three main candidates for structural materials in various fusion power programs and blanket systems around the world [2,3,4]. In GEN IV reactor systems and other energy conversion applications, the high application temperature of SiC composites allows the use of high temperature coolants (molten metals and salt eutectics) which holds the promise for higher energy conversion efficiency (>50%) [5].

SiC composites are multilayered structures consisting of a choice of SiC fiber, a SiC fiber coating, a matrix material to “glue” the fibers together, and in some cases a seal coating layer placed over the machined component. Several reports in the literature have studied SiC composite compatibility in Pb-17%Li eutectic but at temperatures not exceeding 800°C [6,7]. Limited data is available for

exposures of these composite materials in molten lead and at temperatures greater than 800°C.

A screening study was undertaken to study the material compatibility of several SiC fiber composites with molten lead at 1000°C. The pure components which make up the matrix portion of these composites were also evaluated under these experimental conditions to determine their compatibility as well. In addition, an attempt was made to investigate the contributions of machining to a composite's compatibility with molten lead at 1000°C.

2. Experimental Procedure

2.1 Materials

The SiC composite samples consisted of several types each with different SiC fiber materials and matrix components. All composite samples were machined from larger plates to a 1" x1" square shape (except for the SiC_f/SiC composite from Honeywell which was circular) with a 0.125" centering hole in the sample for attachment to the test vessel.

The first set of composites (SiC_f / SiC / Si) were of a liquid silicon melt infiltrated composite composed of Tyranno SA SiC fibers (cloth with #8 Hardness Satin weave) or HiNicalon Type S SiC fibers (cloth with plain weave) supplied by GE power systems. Both composites were prepared in a similar fashion with the composite preforms infiltrated with a SiC particle slurry. Final densification of the composite was carried out by silicon melt infiltration not exceeding 1430°C. The fiber contents of the finished composites were 40 and 37% respectively.

The as received composite material, for both fiber types, contained excess elemental silicon on one or more of the surfaces. To study the compatibility of the fiber and internal matrix, two one inch square samples of each composite were machined to remove the excess silicon from the surface. Once the excess silicon was removed, the fiber weave pattern of the composite could be easily seen. In addition, this machining also revealed small void areas on the inside of the composite (Figure 1). Two additional samples of the HiNicalon Type S fiber composite were further seal coated (in house) with CVD SiC. One sample was seal coated in the “as received” condition and the other sample had the excess silicon machined from the surfaces (showing the fiber weave pattern) before seal coating. The thickness of the seal coat layer was determined to be 50 - 60 microns in depth. These machined composite samples (as well as the CVD coated samples) will be used to investigate the effects of machining a composite on its compatibility with lead since the machined area is nearly the total surface area exposed to the lead.

The second composite ($\text{SiC}_f / \text{Si}_3\text{N}_4$) was prepared using CG Nicalon SiC fibers with a Si_3N_4 matrix material provided by COI Ceramics. The manufacturing process used for the composite provides material which is not fully dense but has a porosity of 5 percent. More detailed information was not available for this composite sample.

The third composite ($\text{SiC}_f / \text{SiC}$) was prepared by Honeywell Advanced Composites. The composite consisted of HiNicalon SiC fiber weave cloth which had a pyrocarbon coating applied to the surface. Repeated cycles of CVI SiC were applied to the perform as the matrix with the final volume percent of fiber being 36% .

In addition to the composites, pure samples of sintered alpha SiC (Hexoloy SA) from Carborundum, Monolithic Chemical Vapor Deposited SiC supplied by Morton, Si₃N₄ from Advanced Ceramics, and single crystal Silicon (Aldrich), were also studied to understand the nature of the pure matrix material in molten lead.

2.2 Experimental Procedure

All samples were cleaned in a 1% nitric acid (75-85°C) solution, deionized water, isopropyl alcohol, and dried under vacuum before initial weights were obtained for each sample. Each sample was loaded into a test vessel made from high purity graphite. The test vessel was composed of two tapered 40 mL crucibles one being inverted onto the other with machined surfaces to make a tight seal. A graphite retention pin was also prepared to fit inside the test vessel to hold the sample below the level of the molten lead during each experiment. Each graphite test vessel contained 150 g of high purity lead (99.9995%, Aldrich Chemical Co.) for each exposure.

Four test vessels were placed onto a tray which slid into a 3 inch OD x 4ft long aluminum oxide furnace tube house in a three zone furnace. The tray with the samples was placed into the center zone of the furnace (12" in length) which was maintained at $1000 \pm 5^{\circ}\text{C}$ over the length of that zone for the experimental exposure. The samples were heated (150°C) under a vacuum (0.02 atm) for 24 hours to outgas the experimental set-up and to also check for possible air leaks within the tube furnace. Once this vacuum treatment was complete, a cover gas of purified argon was maintained in the furnace tube during the remainder of the experimental run. The gas flowed at a rate of 200cc/min ($\pm 10\text{cc/min}$) and was continually

monitored for its moisture content by an online hygrometer. Typical readings from the hygrometer indicated a very low level of moisture entering the tube furnace during each run ($-65^{\circ}\text{C} \pm 5^{\circ}\text{C}$ dew point). Any oxygen ingress to the system was judged to be minimal over the course of each experimental run.

The furnace would be heated ($10^{\circ}\text{C}/\text{min}$) to the experimental temperature and then maintained at that temperature for 120 hours. The furnace was then cooled (under continued argon flow) to room temperature for removal of the test vessels. The graphite vessels were transferred to an inert atmosphere glovebox for re-melting of the lead (at approximately 400°C) and removal of the material sample.

Each sample was cleaned of residual lead using a 1% nitric acid solution (80°C) for 30 minutes. The sample was rinsed in deionized water, dried, and weighed before being immersed into fresh dilute acid solution. This process was repeated until the difference in weights between any two cycles was less than 0.5mg of each other. The final weight was obtained using a five place balance and the weight loss associated with each sample was obtained by comparison to the starting weight. Any oxides which formed on the samples during the exposure were not removed from the sample and are included in the final weights for each piece.

In addition to the weight loss value for each sample ($\mu\text{g}/\text{cm}^2\text{ Hr}$), X-ray diffraction, Scanning Electron Microscope (SEM), and low magnification optical analysis of the surfaces were carried out before and after exposure to lead for each sample.

3. Results

Table 1 contains the corrosion results for all samples exposed to molten lead at 1000°C. A value for the uncertainty (1σ) in the weight loss data is also included in the table to indicate the error associated with each measurement due to the low weight loss for some samples.

3.1 SiC_f / SiC composite (HiNicalon SiC fiber)

The corrosion weight loss for this sample listed in Table 1 is very low and is within the uncertainty of the measurement for this material. The visual appearance of the sample is visually identical to that observed before exposure of the sample to molten lead. There does not appear to be any attack on the exposed SiC fibers or SiC matrix material from exposure to lead at 1000°C.

Chemical analysis by Glow Discharge Mass Spectrometry (GDMS) of the lead from this exposure showed less than detectable levels of silicon or carbon to be present in the lead.

3.2 SiC_f / Si₃N₄ (CG Nicalon SiC fiber)

Figure 2 shows the surface condition of this sample after exposure to lead at 1000°C. The SiC fiber structure can clearly be seen in the figure and an optical comparison to the untested sample indicates some level of matrix attack has occurred. The corrosion data listed in Table 1 indicate that this sample gained weight due to lead exposure. No lead was detected on the surface of the sample after being cleaned suggesting that some amount of lead had penetrated the composite. This material was not fully dense and did have some degree of porosity

in the as received form. To confirm lead penetration an X-ray computed tomography scan of the sample was performed with one cross sectional view shown in Figure 3. The presence of lead in the interior portion of the sample is confirmed by the bright signal emanating from the center of the sample. Other locations within the composite sample also showed a similar response. It appears that once the lead has penetrated the composite it is protected from the cleaning solution, thus yielding a positive weight gain for the sample. Chemical analysis of the lead sample by GDMS did show above detectable levels of silicon in the lead.

3.3 Melt Infiltrated SiC_f / SiC / Si

3.3.1 Tyranno SA SiC fiber Composite (as received)

A cross sectional analysis of the “as received” sample (Figure 4) shows that the final composite sample is somewhat unsymmetrical in nature, with one side of the composite being greatly enriched in silicon. Initial XRD analysis of the “as received” sample surfaces showed elemental silicon, with both alpha and beta SiC present. The beta form was determined to be the dominant phase present.

Upon exposure of the “as received” sample to molten lead the silicon rich side of the sample experienced non-uniform corrosion and became pitted in random locations (Figure 5). The SiC fiber weave pattern can be seen at the bottom of the pits which were formed, along with a covering of fine grain green particles (Figure 6). The nature and shape of these particles can be seen in the SEM picture shown in Figure 7. XRD analysis of the interior regions of the pits where these particles are located showed them to be composed exclusively of two forms of alpha SiC, Moisanite-6H and Moisanite-15R, with the former being the dominant phase. The exterior surface regions of the sample around the pits were also analyzed by XRD and were shown

to be composed of elemental silicon and beta SiC (Moisanite-3C) which was also seen on the original material.

3.3.2 Tyranno SA SiC fiber Composite (surface milled)

A sample of the “as received” material was machined to remove the excess surface silicon present on the composite. The final machined sample clearly shows the fiber weave pattern on both sides of the sample (Figure 1). In addition, small void areas are also apparent on the interior portion of the sample where the melt infiltration process failed to provide the molten silicon. XRD analysis of the general machined surface area indicated the same phases to be present but now with only a trace of elemental silicon. This is not surprising since the molten silicon infused into the interior spaces of the composite during the densification phase of manufacturing and these areas should be few in number.

Exposure of this sample to molten lead did not produce any pitting on the sample surface or any visible attack of the SiC fibers themselves. The only indication of any attack is seen in small areas around fiber intersections where fine grain green particulate material has formed (Figure 8). XRD analysis of these areas containing the green material showed it to be the same as that found in the other post exposure samples of this composite (alpha SiC; Moisanite-6H and Moisanite-15R, with the former being the dominant phase). XRD analysis of areas next to those which contain the green material showed the areas to be composed of beta SiC and minor amounts of carbon and SiO₂.

3.3.3 Hi-Nicalon Type S SiC fiber Composite (as received)

This composite sample is more symmetrical with a uniform excess layer of elemental silicon on both sides of the sample. Initial XRD analysis of the “as received” sample surfaces showed elemental silicon, and both alpha and beta SiC to be present, with beta being the dominant phase.

Exposure of this sample to lead at 1000°C produced the same type of non-localized attack (pitting) on the silicon surfaces as that show with the as received Tyranno SA fiber composite discussed above. The bottom of the pits which formed on the surface also contained a fine green particulate material which upon XRD analysis was confirmed to be alpha SiC (Moisanite-6H and Moisanite-15R, with the former being the dominant phase). The exterior surface regions of the sample around the pits were also analyzed by XRD and were shown to be composed of elemental silicon and beta SiC (Moisanite-3C) and small amount of SiO₂.

3.3.4 Hi-Nicalon Type S SiC fiber Composite (milled sample)

A sample of the “as received” material was machined to remove the excess surface silicon present on both sides of the composite. The final machined sample also clearly shows the fiber weave pattern on both sides of the sample and is similar to the picture shown in Figure 1. AS with the pervious milled sample, this machined composite also exposed a number of small void areas on the internal portions of the composite. XRD analysis of the machined surface indicated the same phases to be present as seen for the previous milled sample.

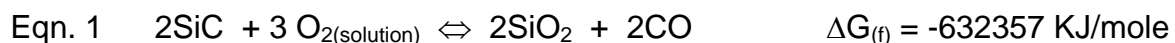
Exposure of this sample to molten lead also did not produce any pitting on the sample surface or any visible attack on the SiC fibers themselves. The only indication of any attack is seen in small areas around fiber intersections where fine grain green particulate material has formed (similar to the milled sample of TYranno fiber composite). XRD analysis of these areas containing the green material showed it to be the same as that found in the other post exposure samples of this composite (alpha SiC; Moisanite-6H and Moisanite-15R, with the former being the dominant phase). XRD analysis of areas next to those which contain the green material showed the area to be composed of beta SiC and minor amounts of carbon and SiO₂.

3.3.5 CVD SiC coated Hi-Nicalon Type S SiC fiber Composites

Exposure of the two samples which were seal coated with CVD SiC showed only a small amount of corrosion (weight loss value from Table 1) with no visible sign of attack from the molten lead. XRD analysis of the exposed samples showed a small amount of SiO₂ to be present on the surface and no change to the type of SiC (only beta SiC both before and after exposure) present.

4. Discussion

The intent of this study was to understand the materials compatibility of the various SiC composites in molten lead without the presence of additional corrosion factors. One major factor is the presence of oxygen both dissolved in the lead and also present in the cover gas of the experimental set-up which could diffuse into the molten lead. At 1000°C the reaction between SiC and oxygen is very thermodynamically favorable as indicated by equation 1.



The argon cover gas was purified by passage through a reactive metal purifier and a column of graphite pieces held at 650°C. These measures were taken to reduce the oxygen and moisture content of the cover gas to as low a level as possible. On-line hygrometer readings of the inlet gas gave dew point readings of -55 to -65°C which corresponds to a partial pressure of oxygen (through moisture dissociation) in the cover gas of 7.4×10^{-10} atm (assuming a gas temperature of 700°C).

The use of graphite vessels to hold the lead (and samples) allows for a further reduction in the dissolved oxygen available to interact with the SiC samples. If kinetics are assumed to be non-limiting at this temperature, the thermodynamic partial pressure of oxygen needed to form CO at 1000°C is 9.3×10^{-19} atm.

Therefore, the graphite vessel will purify the lead of dissolved oxygen to a level which corresponds to this partial pressure of oxygen. Initial evaluations of the purified lead purchased for these experiments indicated oxygen to be present in the lead to a level of 10-20 ppm as determined by inert fusion analysis (LECO oxygen and carbon analyzer), ESCA, and GDMS. Experimental confirmation for this value for oxygen content was provided by exposure of a duplicate sample of pure monolithic SiC sample (CVD from Morton) to the lead but without graphite being available to purify the molten lead. An aluminum oxide test vessel and aluminum oxide retention pin were used for this exposure. Aluminum oxide is very thermodynamically stable under these experimental conditions and is not expected to contribute to the oxygen content of the lead. Without graphite available to react with the oxygen present in the lead, the oxygen will be available to react with the

sample causing a greater weight loss of the sample. When the sample disk was removed from the lead (1000°C, 50 hrs) and cleaned of residual material, there was SiO₂ on the surface. Once this layer was removed (1% HF @ 80°C) the weight loss for this sample was larger ($7.3 \times 10^{-6} \pm 3.9 \times 10^{-7} \text{ g/cm}^2 \text{ Hr}$) than for an identical sample exposed to lead using the all graphite vessel. This difference in weight loss can be equated to an amount of oxygen needed in the lead at the start of the experiment to develop this weight loss value and was calculated to be approximately 13 ppm oxygen present in the lead.

Since graphite is used to control the oxygen content of the lead, carbon solubility also needs to be understood as a possible factor which could influence the corrosion data from these experiments. Due to equilibrium considerations, the presence of dissolved carbon in the lead at 1000°C could suppress the solubility/corrosion data values formulated for these SiC composite samples. Extrapolating the high temperature solubility data from Ruff and Bergdahl [8], an estimate can be made for the solubility of carbon in molten lead at 1000°C to be 0.25 at% or 145 ppm. No additional analytical efforts were made to confirm this value but it will be assumed that carbon is present in the lead under these experimental temperatures. The effect of dissolved carbon on the corrosion performance of these composites is included in the weight loss values obtained for these samples. Repeat experiments using similarly purified lead, without the presence of dissolved carbon, may produce higher corrosion values.

Of the three classes of SiC composite samples studied the SiC_f / SiC composite showed the least amount of attack by the molten lead. The weight loss for this

sample was within the error associated with the weight loss value. In addition to the SiC_f / SiC sample, the two samples of pure SiC material also shows very good compatibility with molten lead at 1000°C. The weight loss data for CVD monolithic beta SiC was $8.9 \times 10^{-7} \pm 1.8 \times 10^{-7}$ g/ cm² Hr and that of the sintered alpha Hexoloy SA was $6.25 \times 10^{-6} \pm 1.09 \times 10^{-7}$ g/ cm² Hr. The Hexoloy SA material is not as fully dense as that of CVD material (3.1g/cc compared to 3.2g/cc) and does have a small amount (0.4%) of boron added as a binder in the sintering process. The behavior of both forms of pure SiC materials further supports the good compatibility (with lead) of a SiC matrix in a composite material.

The remaining two composite samples (SiC_f / Si₃N₄ and SiC_f / SiC / Si) did show some level of attack from the molten lead. It is possible that limited solubility of the matrix material may be the reason for the observed weight loss. For both of the silicon infused composite samples (SiC_f / SiC / Si), the corrosion performance can be linked to the excess silicon present on the surface. Comparison of the weight loss values for the "as received" samples to that of pure silicon show values which are close to one another. This together with the selective attack on the silicon enriched surface and lack of attack on the milled samples points to the solubility of silicon in lead to be the main reason for the weight loss observed with this sample.

Limited experimental data exist for the high temperature solubility of silicon in molten lead. Solubility experiments by Thurmond and Kowalchik [9], and Kirkwood and Chipman [10] over the temperature range of 1050-1420°C measured a solubility of 0.029 - 0.45 wt% for silicon in molten lead. Using the weight loss data from the pure silicon sample, an approximate solubility of 0.013wt% silicon in lead can be obtained which is consistent with their data. Conversion of the actual weight lost for each of

the two as received silicon infused composites were closer to 0.022 wt% indicating that additional corrosion factors maybe also be present with these composite samples.

The different distributions of SiC crystal structures found in the pit areas compared to those present in the “before exposure “ condition is interesting. There appears to be an enhancement of the alpha SiC relative to that of the beta SiC after exposure to the lead. Both forms of SiC are expected to be present in these samples (as confirmed by XRD analysis) due to the nature of the manufacturing process and the densification temperatures [11]. It is not clear at this time whether there is selective attack on the beta form of SiC compared to that of alpha form in these composites, or if the distribution of the two forms throughout the composite is non-uniform. Further studies would be needed to understand this observation.

Using the two milled composite samples, there appears to be no visual evidence of attack to the HiNicalon Type S or Tyranno SA SiC fibers themselves in either of the two composite samples. Tyranno fibers can contain a small amount of titanium (1.5%) as a high temperature enhancement for the fiber. Titanium has an increased solubility in lead above 725°C due to the formation of Ti_4Pb complexes which draw the titanium from the material and tie it up in solution as the complex. However, GDMS analysis of the lead failed to show any detectable titanium in the post test lead.

The weight loss evaluation of the SiC_f / Si_3N_4 composite sample is less straightforward due to the weight gain for this composite sample. A possible contribution

from the manufacturing process variability and lower final densification of this composite may have caused the composite to experience a weight gain from exposure to lead. If the porosity developed due to exposure to the lead (enhanced solubility), then some loss of the matrix material, in this case Si_3N_4 , needs to occur. A sample of pure silicon nitride exposed under similar conditions produced a small weight loss value of $2.6 \times 10^{-6} \pm 9.16 \times 10^{-7} \text{ g/cm}^2 \text{ Hr}$. The amount of corrosion on the Si_3N_4 sample is also in agreement with the other pure materials exposed to lead in these experiments. Optical evaluation of the $\text{SiC}_f/\text{Si}_3\text{N}_4$ composite does show some degree of roughening of the composite surface after exposure compared to the initial surface condition of the sample. However, the corrosion performance of this composite was most likely due to contributions from the manufacturing process and not totally a matrix solubility issue.

The results of the CVD SiC seal coating for the composites were as expected. An order of magnitude improvement in the corrosion weight loss values for the silicon infused composite samples was realized by minimizing the contact (effectively lowering the solubility) of elemental silicon to the molten lead. A much smaller improvement value was seen for the milled sample which was CVD coated. This may indicate that the machining contribution to a composite's materials compatibility with molten lead is smaller than the contributions made by the matrix's stability toward the medium.

5. Conclusion

Isothermal testing of various silicon carbide fiber reinforced composite samples has been studied in molten lead at 1000°C for 120 hours. The best performance (based

on weight loss) was for the composite which utilized SiC fibers and a SiC matrix (SiC_f / SiC composite material). Samples of a silicon melt infused composite did show increased weight loss due to the limited solubility (in lead) of the excess silicon present in the composite. Optical evaluations of machined edges and surfaces after exposure did not show the presence of any additional pores caused by the lead exposure or any attack on the SiC fibers used to form the composites. Machining contributions to a composite material's compatibility with molten lead appears to be a smaller contribution than that made by the matrix's stability toward the medium.

Manufacturing variabilities and lower densification of the SiC(f)/Si₃N₄ composite did not allow for a complete evaluation of this composite in regards to molten lead compatibility.

Seal coating (with SiC) of a composite appears to be effective in minimizing corrosion due to solubility concerns but might not be needed to protect against machining damage done to a composite. A more complete study would be needed to further investigate the role of machining damage to a composites overall compatibility with molten lead at 1000°C.

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Table 1

Corrosion Weight Loss Data for SiC Composites and Matrix Components

(1000°C , 120Hrs)

Sample	Area Exposed (cm ²)	Initial weight (g)	Weight Change (g)	Corrosion g/sq cm Hr	Uncertainty ± g/sq cm Hr
Monolithic SiC	3.62	1.13165	-0.00034	8.9 E-7	1.8 E-7
Hexoloy SA SiC	6.09	2.18512	-0.00457	6.25E-6	1.09E-7
Si ₃ N ₄	3.64	0.97369	-0.00123	2.6 E-6	1.50 E-7
Elemental Silicon	11.56	2.81757	-0.01918	1.38 E-5	5.05 E-8
SiC _f / SiC	1.74	0.23904	-0.00010	4.4-7	3.1E-7
SiC(f) / Si ₃ N ₄	16.94	5.23372	+0.01531		
SiC(f)/SiC/Si Tyranno SA fiber	6.37	5.7254	-0.03172	4.14 E-5	9.16 E-8
SiC(f)/SiC/Si Tyranno SA fiber milled	13.82	4.72664	-0.00837	5.04E-6	4.22E-8
SiC(f)/SiC/Si HiNicalon (S) fiber	14.2	4.8738	-0.03498	2.05 E-5	4.10 E-8
SiC(f)/SiC/Si HiNicalon (s) fiber milled	13.54	3.75433	-0.00922	5.67 E-6	4.30 E-8
Sealed coated SiC(f)/SiC/Si as recieved	12.34	4.13258	-0.002495	1.68 E-6	4.70 E-8
Sealed coated SiC(f)/SiC/Si milled	11.64	3.28647	-0.00131	9.38 E-7	5.00 E-8

Figure 1

SiC_f / SiC / Si HiNicalon Type S Fiber (Milled surface)

Weave pattern of fibers are shown after machining the composite surface. Small void areas are now visible near the fiber junctions.

(The center hole is for the graphite retention pin in the test vessel)

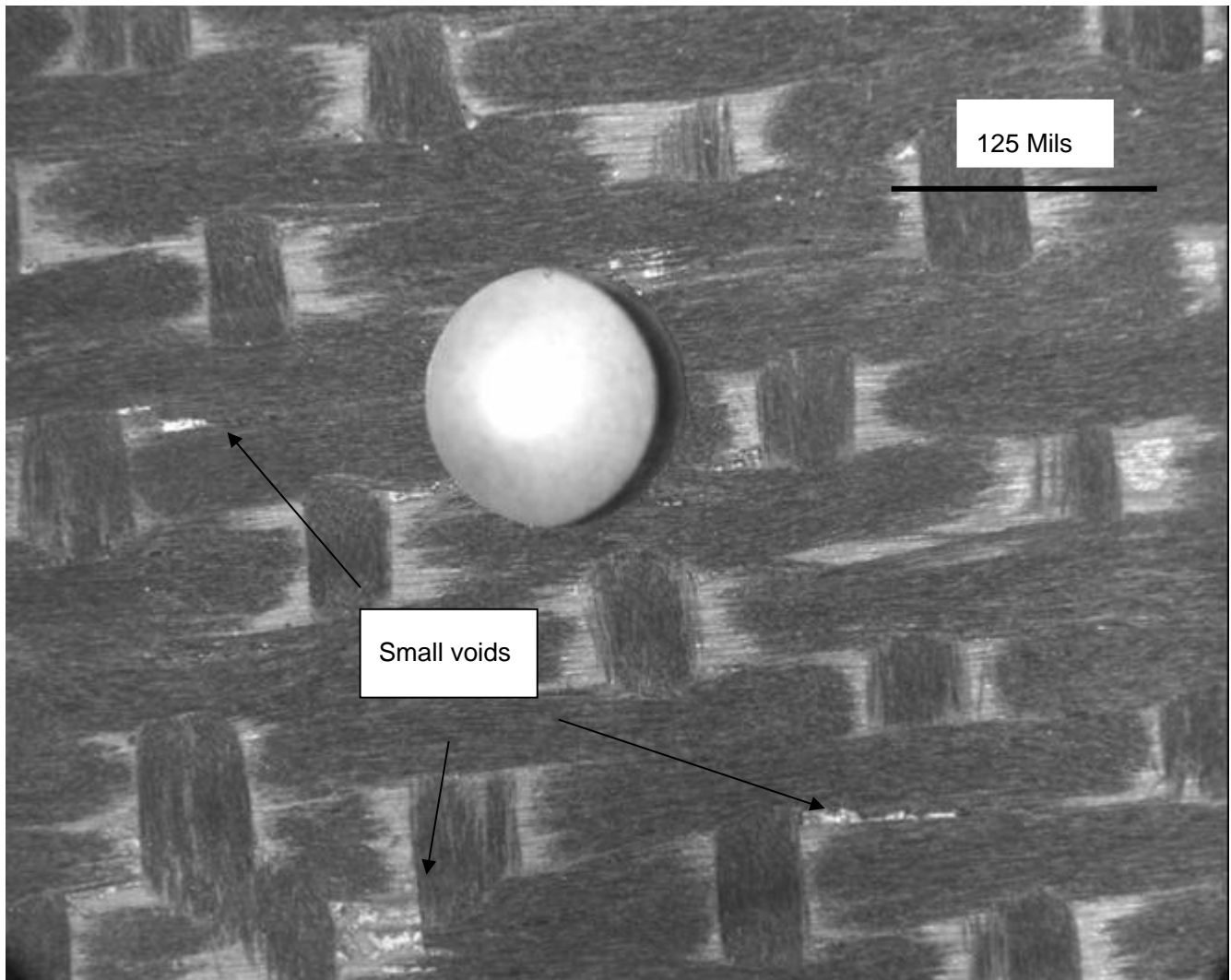


Figure 2

$\text{SiC}_f / \text{Si}_3\text{N}_4$ Composite

Exposed to molten lead at 1000°C for 120 Hour

SiC fiber weave pattern is now visible on the surface of the composite.

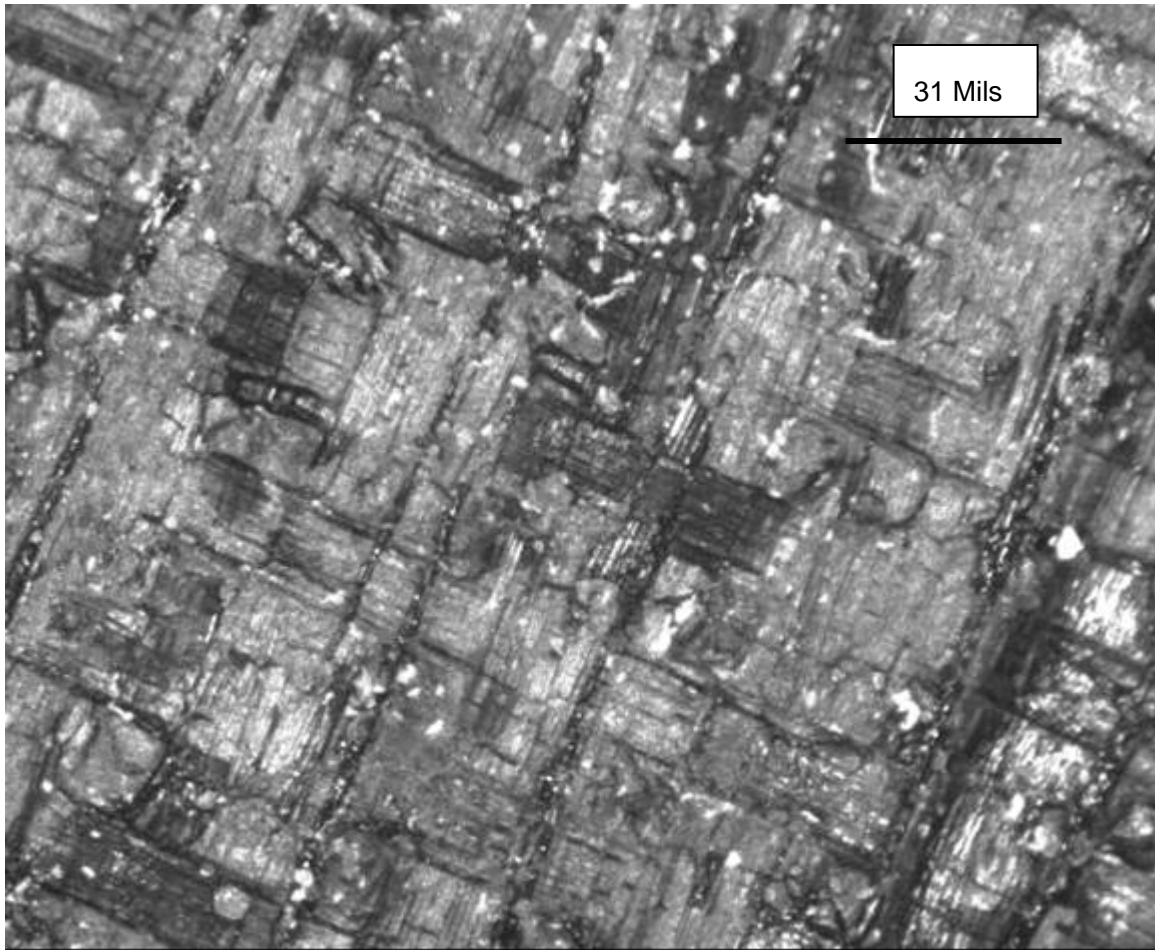


Figure 4

Cross Sectional view of SiC_f / SiC / Si Composite Tyranno SA fiber

Excess silicon layer is seen at the top of the cross section shown in the figure.

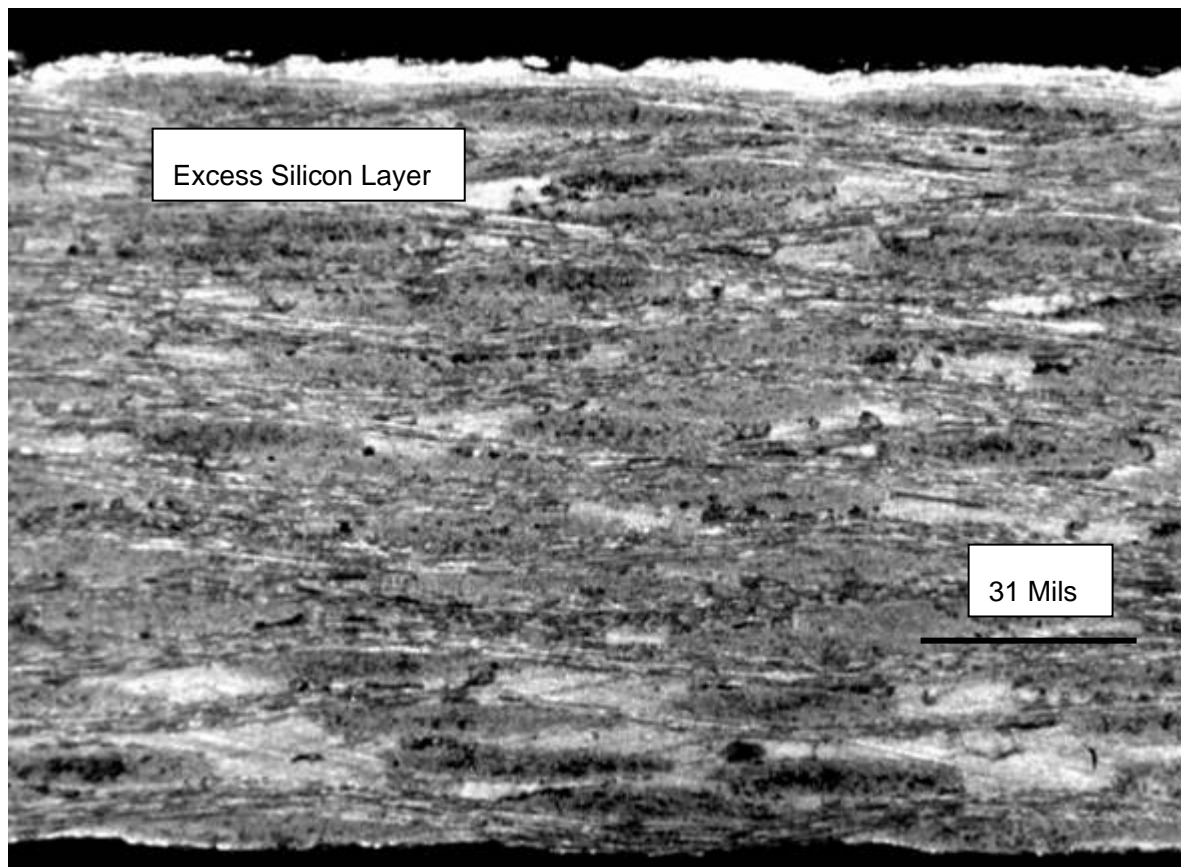


Figure 5

SiCf / SiC / Si Composite Tyranno SA fiber

Exposed to molten lead at 1000°C for 120 Hour

Surface shown is of the excess silicon side of the composite now showing random
areas of attack from the lead.

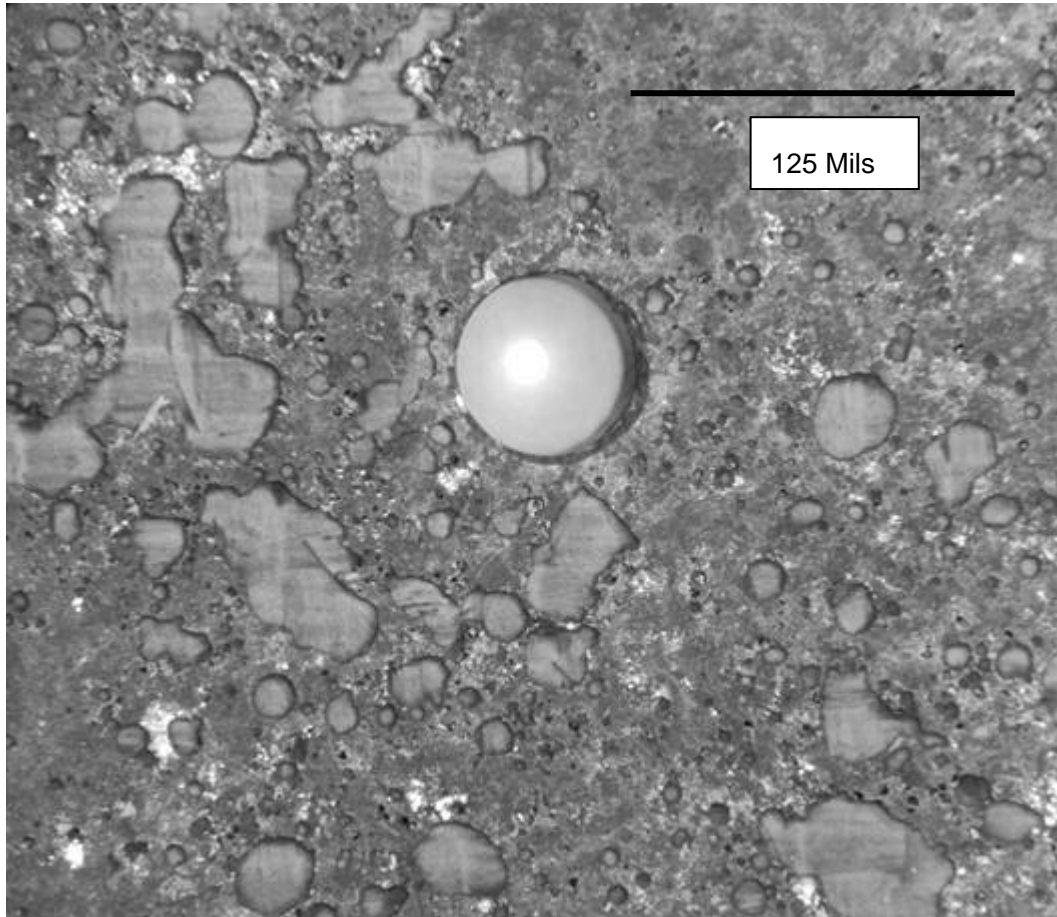


Figure 6

Inside surface of pit SiC_f/ SiC / Si Composite Tyranno SA fiber

1000°C, 120 Hour Exposure

Enlarged view of one pit area from the previous figure showing the SiC particles now present at the base of the pits in addition to the fiber weave pattern.

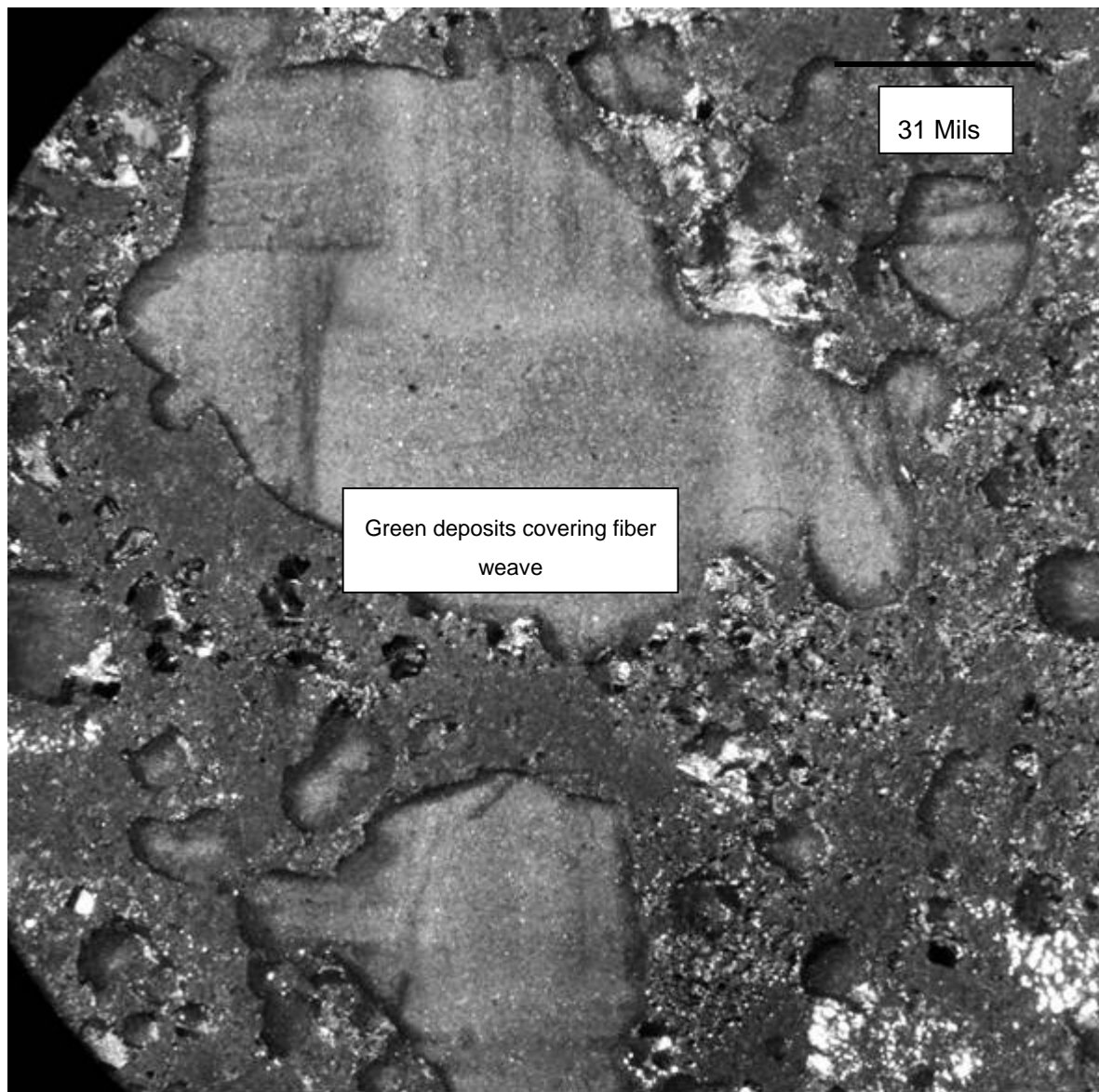


Figure 3

Cross Sectional XRD Computed Tomography Scan

$\text{SiC}_f / \text{Si}_3\text{N}_4$ CG Nicalon Fiber Composite, 1000°C, 120 Hours

(Highlited Square is the signal from Lead)

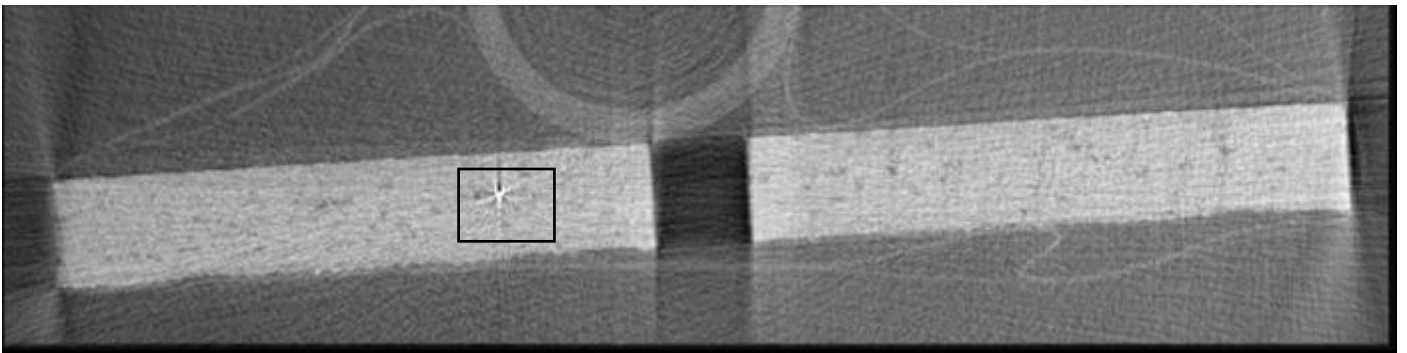


Figure 7

SEM Picture of Pit Interior of Fine Grained SiC Partcles (3000x Mahnification)

SiC_f / SiC / Si Composite Tyranno SA fiber

Exposed to molten lead at1000°C for120 Hours

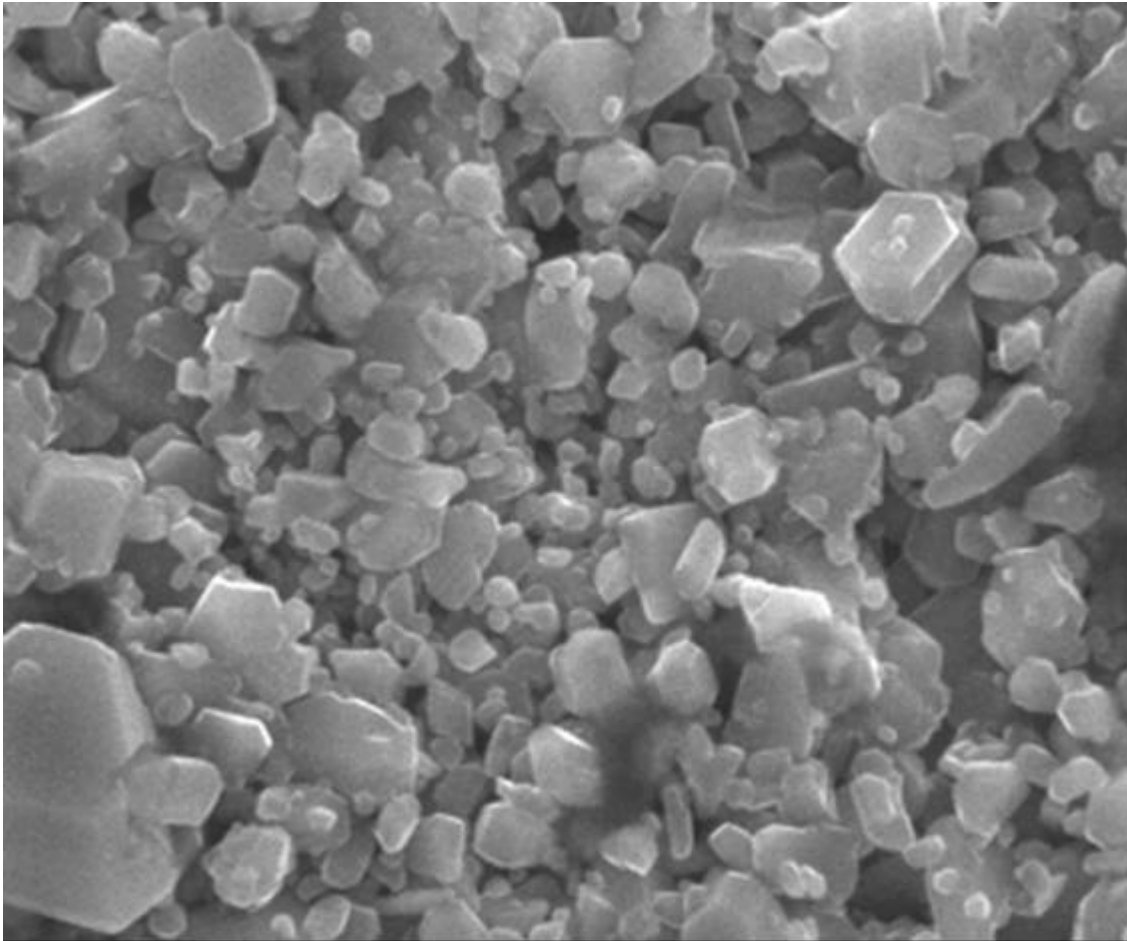


Figure 8

SiCf / SiC / Si Composite Tyranno fiber (Milled Surface)

Exposed to molten lead at 1000°C for 120 Hours

The highlighted box shows one area where green SiC deposits can be seen at the fiber intersections. Other spots are also seen at various locations on the composite surface.

