

## WEAR AND REACTIVITY STUDIES OF MELT INFILTRATED CERAMIC MATRIX COMPOSITE

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### ABSTRACT

As interest grows in the use of ceramic matrix composites (CMCs) for critical gas turbine engine components, the effects of the CMCs interaction with the adjoining structure needs to be understood. A series of CMC/material couples were wear tested in a custom elevated temperature test rig and tested as diffusion couples, to identify interactions. Specifically, melt infiltrated silicon carbide/silicon carbide (MI SiC/SiC) CMC was tested in combination with a nickel-based super alloy, Waspaloy, a thermal barrier coating, Yttria Stabilized Zirconia (YSZ), and a monolithic ceramic, silicon nitride (Si<sub>3</sub>N<sub>4</sub>). To make the tests more representative of actual hardware, the surface of the CMC was kept in the as-received state (not machined) with the full surface features/roughness present. Test results include: scanning electron microscope characterization of the surfaces, micro-structural characterization, and microprobe analysis.

### INTRODUCTION

Ceramic matrix composites (CMCs) have decades of development and testing efforts behind them [1-4]. With this background and key material attributes, CMCs are also finding technical applications [5,6]. As technical acceptance is achieved, additional challenges and insertion issues are discovered resulting in additional efforts and insight. One area that is coming to light as testing proceeds is the material interaction with the supporting structure and contacting hardware.

As part of the initial effort to understand such interactions, a melt infiltrated silicon carbide/silicon carbide (MI SiC/SiC) CMC was used in a series of wear and reactivity testing. The wear testing was done at temperature against a series of materials that could be envisaged as possible materials that would be present in actual applications. These materials were Waspaloy, Yttria Stabilized Zirconia (YSZ) and Silicon Nitride. In addition, material couples were made to note material interactions. The effort focused on characterization after select times to see if micro-structural changes were present (mainly surface related).

### PROCEDURE

#### Material

As noted, the material for this study was the MI SiC/SiC CMC system. This material has been studied by other investigators [7-9]. The MI SiC/SiC composite is comprised of a stoichiometric SiC Sylramic™ with a boron nitride (BN) interface coating. The SiC matrix is infiltrated by vapor deposition followed by slurry casting of SiC particulates and a final melt infiltration of Si. The fiber preform was a cross-ply balanced 5 harness satin weave with 18 ends

per inch and 36% fiber volume fraction. The material cross section (micro-structure) has been shown by other investigators [8,9].

### Wear Testing

Wear testing was done using a wear rig developed by United Technologies Research Center (UTRC). A schematic of the wear rig is shown in Figure 1. This unit consists of a fixture that holds two materials in contact inside a furnace capable of 1200°C. The fixture permits loads up to 36 N to be applied to the specimens while simultaneously imposing displacements at frequencies ranging from 0.005m at 100 Hz to 0.0127m at 20 Hz. The tests were performed under a flowing oxygen atmosphere at various temperatures. The experimental series is shown in Table I. For this testing series, the surface of the Mi SiC/SiC was left in the as-processed state with the surface finish not machined to remove any surface asperities. All samples were 25 mm x 25 mm for the faces in contact.

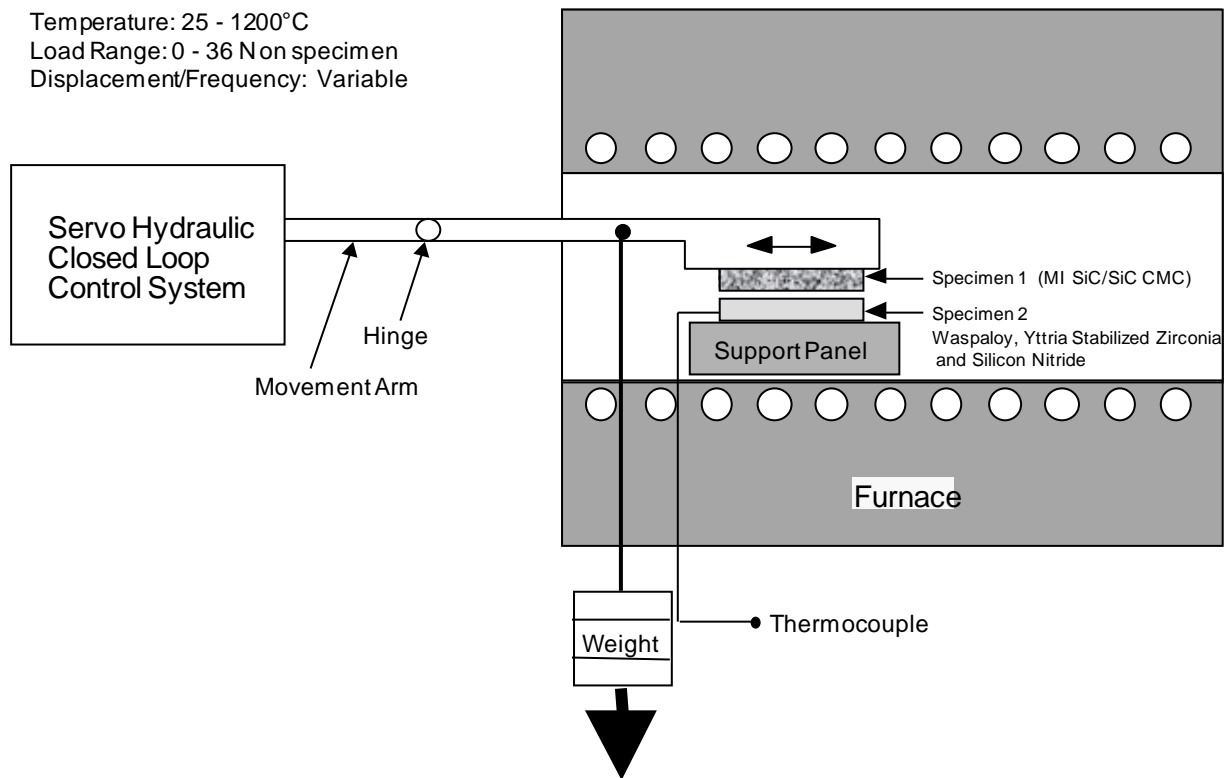


Figure 1. Schematic of UTRC Wear Rig

Table I. Wear Testing Experiment Series

Specimen 1	Specimen 2	Temp (°C)	Time (hr)	Displacement (mm)	Hz	Pressure (MPa)
Mi SiC/SiC	Si3N4	1038	100	0.1	90	0.055
Mi SiC/SiC	YSZ	1010	100	0.1	90	0.055
Mi SiC/SiC	Waspaloy	649	300	0.1	103	0.055

## Reactivity Testing

Reactivity tests were run in an alumina tube furnace with flowing oxygen atmosphere ( $2.0 \cdot 10^{-5} \text{ m}^3/\text{s}$  or 1.2 liter/min). The experimental test plan is shown in Table II. Close contact between the samples for reactivity testing was done by having a weight on top of the sample stack. This weight was achieved using an alumina plate and resulted in an estimated average pressure of 1.8 MPa. Samples were removed from the furnace after the furnace had cooled to room temperature. After furnace exposure, select samples were sectioned for micro-structural characterization. All samples were 25 mm x 25 mm for the faces in contact.

**Table II. Reactivity Testing Experiment Series**

<b>Specimen 1</b>	<b>Specimen 2</b>	<b>Temp</b>	<b>Time</b>
		(°C)	(hr)
Mi SiC/SiC	Si <sub>3</sub> N <sub>4</sub>	1038	500
Mi SiC/SiC	Si <sub>3</sub> N <sub>4</sub>	1038	1000
Mi SiC/SiC	Si <sub>3</sub> N <sub>4</sub>	1038	4000
Mi SiC/SiC	YSZ	1038	1000
Mi SiC/SiC	YSZ	1038	2000
Mi SiC/SiC	YSZ	1038	4000
Mi SiC/SiC	Waspaloy	649	1500
Mi SiC/SiC	Waspaloy	649	4000

## Characterization Post Exposure:

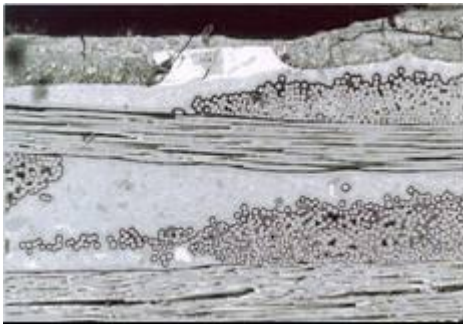
Selected samples from both the wear and reactivity studies were laser cut and a cross section was mounted and polished for optical microscopy, SEM and microprobe analysis. In some cases, the contacted surface of the sample was also examined.

## RESULTS

### Wear

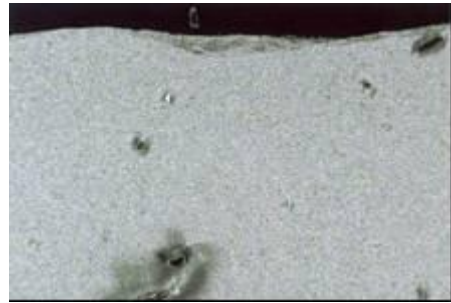
No visible amount of wear was seen on the surface of the MI SiC/SiC against Si<sub>3</sub>N<sub>4</sub> both materials. There was a white coating observed on the surface of the samples. SEM and microprobe analysis is shown in Figure 2. The surface coating was most likely silica, SiO<sub>2</sub>.

The wear couple between the MI SiC/SiC and YSZ showed an area of wear. The wear was present in an area where the asperity peaks of the MI SiC/SiC rubbed on the YSZ. This is shown in Figure 3. There was little material transfer of the YSZ to the MI SiC/SiC. Detailed microprobe analysis showed that a small portion of the YSZ was scrapped off and that is consistent with the images shown in Figure 3. In addition, oxidation of the surface of the MI SiC/SiC was observed.



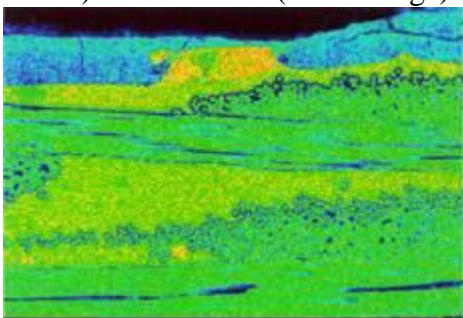
200µm

a) MI SiC/SiC (SEM Image)

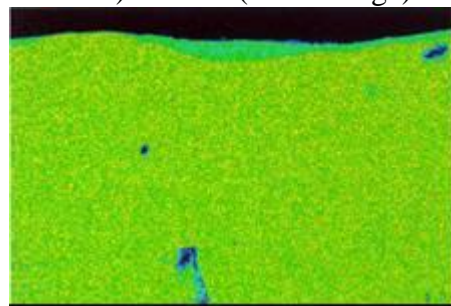


200µm

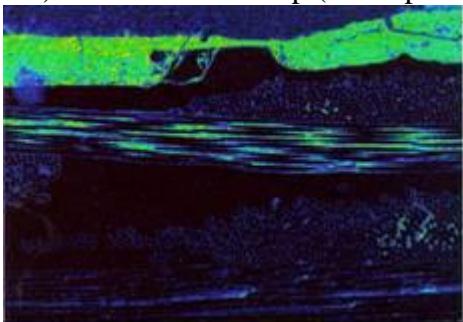
b) Si3N4 (SEM Image)



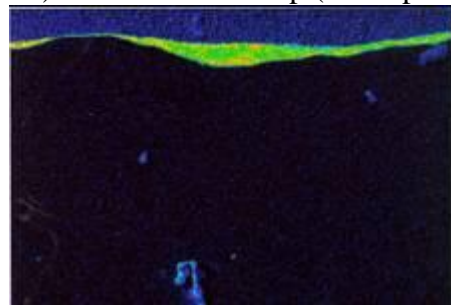
c) Si Elemental Map (Microprobe)



d) Si Elemental Map (Microprobe)

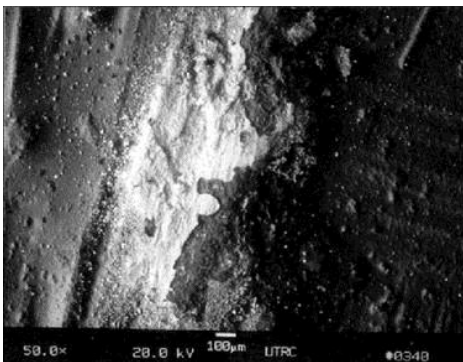


e) O Elemental Map (Microprobe)

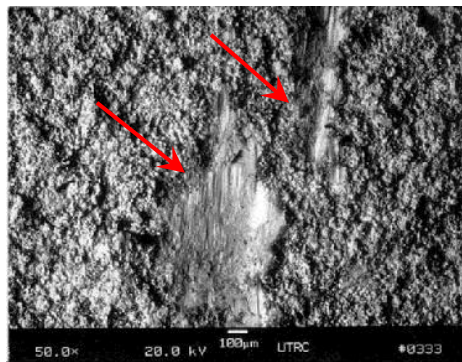


f) O Elemental Map (Microprobe)

**Figure 2. Analysis of MI SiC/SiC – Si3N4 Wear Test**



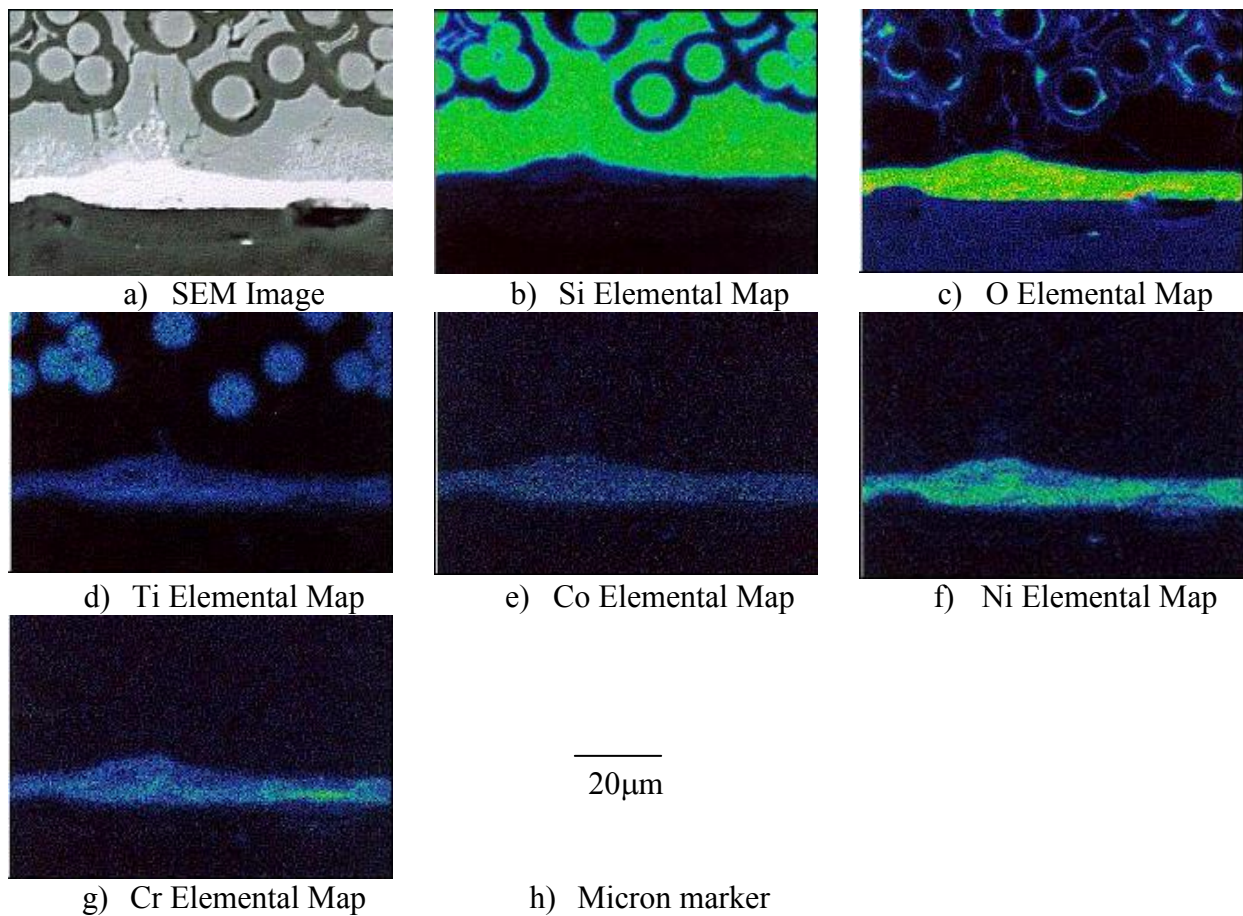
a) MI SiC/SiC Surface



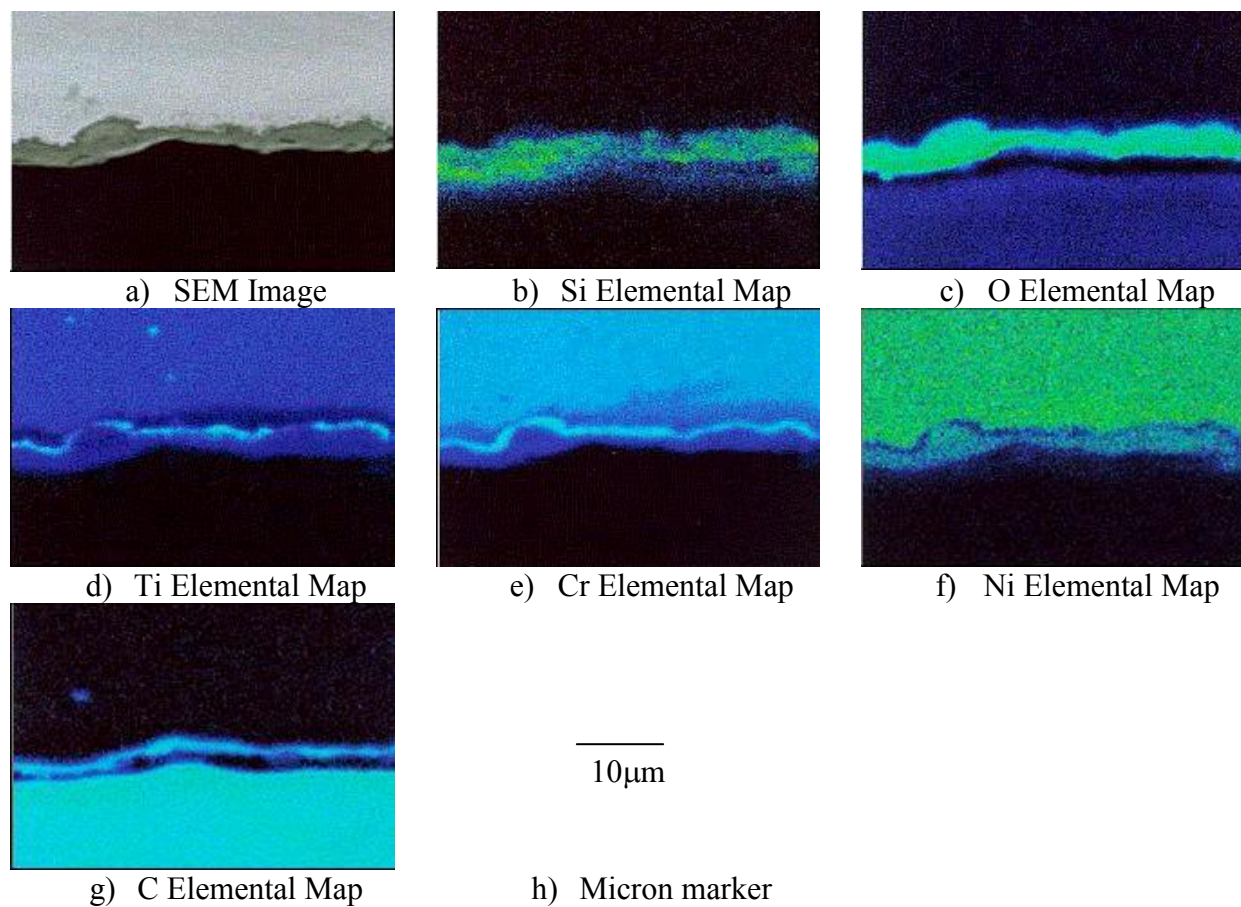
b) YSZ Surface

**Figure 3. SEM Images of Surfaces from MI SiC/SiC – YSZ Wear Test**

The wear between the MI SiC/SiC and Waspaloy showed more areas of wear (8 areas on the 25 mm x 25 mm surface) where the MI SiC/SiC peak surface roughness came into contact of the surface of the Waspaloy. The localized pressure was determined to be on the order of 27 to 69 MPa based on the applied load and size of the surface asperities. The areas of wear were found to be 12.7  $\mu\text{m}$  deep (via surface profilometry). The corresponding areas on the MI SiC/SiC showed some discoloration but no wear. It was clear that material was transferred between the wear couples. Figure 4 shows the microprobe analysis of the surface of the MI SiC/SiC. There was a metal oxide layer present with the atomic species consistent with the Waspaloy material sample. In addition, there were suspected silicide layers present on the Waspaloy, as shown in Figure 5.



**Figure 4. Analysis of MI SiC/SiC Surface After Wear Test with Waspaloy**



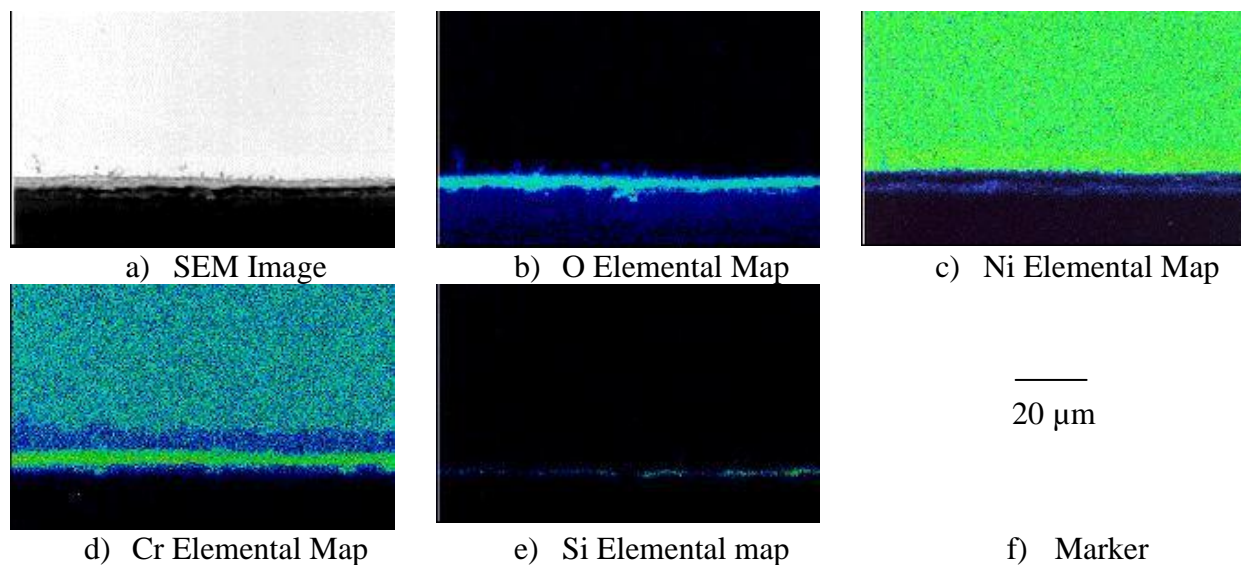
**Figure 5. Analysis of Waspaloy from Wear Test with MI SiC/SiC**

### Reactivity

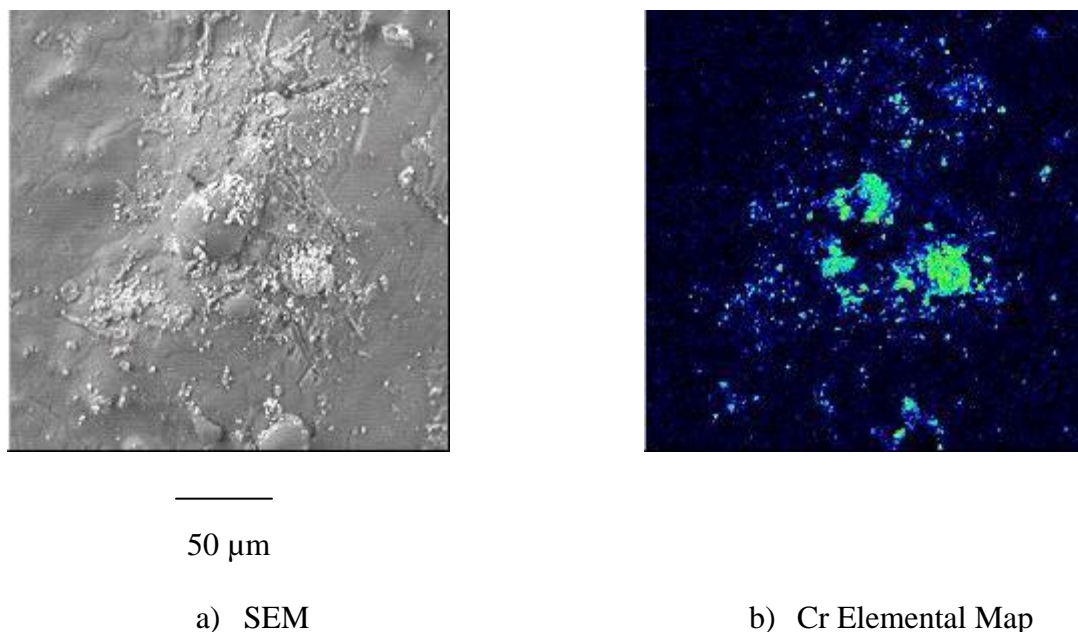
There was no evidence of any reactivity of the MI SiC/SiC against  $\text{Si}_3\text{N}_4$  and YSZ and the only thing to note was the presence of the silica scale on the MI SiC/SiC as was seen in the wear efforts. This is consistent with the relatively low temperature present and the preponderance of ceramic species in the effort.

More complex oxidation product was seen on the reactivity couple of the MI SiC/SiC against the Waspaloy. The Waspaloy generated a Cr oxide scale which is consistent with expectations for such a metal system in an oxidizing environment. [10]. In addition, there were areas in the oxide scale that were rich in silicon as shown in Figure 6. The silicon was measured to be about 2  $\mu\text{m}$  in thickness. This may be due to areas where the surface roughness of the MI SiC/SiC allowed point contact. As a result, silicon was diffused from the SiC/SiC into the Waspaloy. Only a thin film of silica was formed on the surface of MI SiC/SiC, even after 4000hrs of exposure which is consistent with long term exposure (see Table II).

The nature of the point contact can be seen by looking at the surface of the MI SiC/SiC after 4000 hrs as shown in Figure 7, looking at just one of the species found in the Waspaloy. Figure 7 shows the element Cr and the surface map shows the contact nature of the test and the effect of the surface roughness as well as the mobility within the Si phase.



**Figure 6. Analysis of Waspalloy after 1500 hr Reactivity Test**



**Figure 7. Analysis of MI SiC/SiC Surface after 4000 hr Reactivity Test**

## DISCUSSION

The wear and reactivity testing of ceramic species against ceramic species only showed limited concern due to the surface condition of the as-processed MI SiC/SiC surface (Figure 3). There was some formation of oxide (silica) scale but that was not unexpected (and predicted based on thermodynamic calculations) [11] (Figure 2). When considering the presence of a metal present in the couple, other researchers of metal/ceramic couples have seen that Ni is the

major species that diffuses into the Si based carbide ceramic [12]. They have also shown that Si and C move into the metal. The presence of Si diffusing in was seen in this effort (Figure 6).

The difference in this study is the presence of Si as part of the melt infiltrated process that is a metal in the structure. It is not carbide. This provides a path for species to move within the composite as shown in previous work of one of the authors [9]. This is seen in the Cr elemental map of Figure 7, where Cr is present on the surface and has diffused. It was surmised that the Si provides a path for diffusion.

## CONCLUSION

No significant reaction occurred at the interface for the various material couples under the current reactivity tests except for some oxidation and minor reaction (SiC/SiC-Waspaloy). The wear tests have shown that small amount of wear and reaction occurred under the current experimental conditions. In most cases, thermodynamic calculation as well as previous experimental studies could account for the present experimental observations. There is concern about the Si present in the material from the melt infiltrated process acting as a transport media to facilitate the migration of chemical species within the MI SiC/SiC.. This later point warrants additional investigation with polished faces present to assure greater contact between the samples to clearly note if Si provides a preferred path for diffusion.

## ACKNOWLEDGMENTS

The authors are indebted to Dr. Hong Du, formerly of United Technologies Research Center, for her assistance in performing and organizing the work presented in this paper. Her help is greatly appreciated. This work was performed under the Enabling Propulsion Materials Program, Contract NAS3-26385, Task A, David Brewer program manager.

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