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INTEGRATED, INC.

VIA U.S. MAIL

November 1, 1995
96RD0248

Dr. Steven Fishman
Program Officer/Code 332
Office of Naval Research
800 North Quincy Street
Arlington, Virginia 22217-5660

Subject: Transmittal of Progress Report for "High Strength, High Toughness In Situ Ceramic Composites," Under Contract No. N00014-95-C-0242, Data Item A001

Dear Dr. Fishman:

Enclosed, please find the subject document. It is a summary of our activities on the subject contract for the first quarter, covering July 31, 1995 through October 31, 1995.

Should you have any questions, concerns, or suggestions regarding the program, please contact me at the phone number and address listed below.

Sincerely,

Ender Savrun, Ph.D.
Director
Advanced Materials

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Enclosure

cc: G. Rogers, ONR
Director, NRL
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HIGH STRENGTH, HIGH TOUGHNESS IN SITU CERAMIC COMPOSITES

Progress Report for the Quarter:
July 31, 1995 to October 31, 1995

Ender Savrun
Cetin Toy

November 1995

Prepared for

OFFICE OF NAVAL RESEARCH

Under Contract No. N00014-95-C-0242, Data Item A001

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Task 1. Composite Synthesis

Raw materials were purchased and two batches were prepared by wet mixing of the powders for the following two compositions; 3TiC+2Si (composition 32) and 3TiC+3Si (composition 33). Thermal analysis (DTA/TGA) of these two batches were performed up to 1500°C at a scan rate of 10°C/min to determine the critical temperature range where the reactions start. Simultaneous TGA/DTA of the 3TiC+2Si and 3TiC+3Si powder mixtures showed presence of exothermic peaks at 700°C for both mixtures. 3TiC+3Si system showed a strong endothermic peak at 1480°C. This peak is attributed to the melting of excess silicon. However, further investigation is underway to clarify the nature of the peaks observed. Hot pressing of dry mixed 3TiC+2Si powder composition was performed by using a hot pressing schedule developed by Battelle as the starting point.

Materials and Power Preparation

Two of the three different compositions proposed in our study were prepared using the following powders; (i) 99.999% high purity silicon metal with average particle size 4.5 microns (CERAC Inc.), (ii) 99% purity titanium carbide powder with average particle size of 2 microns (Alfa-Johnson-Matthey Inc.).

Sedimentation experiments were conducted for 3TiC+2Si composition to determine the dispersant to be used in the wet mixing process. Three suspensions with 10% solids by volume were prepared in acetone with KD-1 and DM55 dispersants. 0.5% (of the powder weight) KD-1 and DM55 dispersants were completely dissolved in acetone. First TiC, later Si powders were added while mechanically stirring the suspension. Suspensions were sonicated for 15 seconds before pouring it in a graduated cylinder. Sedimentation height as a function of time was recorded for next 18 hours, and is presented in Table 1.

Table 1 - Sedimentation behavior of 3TiC+2Si powder mixture in acetone

Acetone Only		Acetone with KD1		Acetone with DM55	
Time (min)	Height(mm)	Time (min)	Height (mm)	Time (min)	Height (mm)*
30	41	30	35	15	7
60	35	60	31	45	13
120	31	120	26	18 hrs.	17
18 hrs.	31	18 hrs.	21		

* Did not show a clear supernatant portion due to the particle separation

As shown in Table 1, KD1 is a better dispersant DM55, since it has the lowest packing height. Packing density of KD1 containing sediment is at least 35% better than the sediment with only acetone solution (0.14 g/cm^3 versus 0.09 g/cm^3). The particles in DM55 containing suspension separated into two distinct portions after 10 minutes. While the TiC particles were sedimented and formed a rising packing layer at the bottom of the graduated cylinder, the most of the Si particles remained in the suspension, thus not giving

a clear supernant solution. Therefore, KD1-acetone solution was selected as the dispersion media for the rest of the experiments.

280 grams of 3TiC+2Si and 3TiC+3Si batches were prepared by using 0.5% KD1 dispersant in acetone. Although 30 volume % solid containing suspensions were targeted, only 20 volume % solid containing suspensions were prepared due to increasing solution viscosity. After the complete dissolution of the dispersant, first TiC powder then Si powder was slowly added to acetone solution while mixing it mechanically. The suspension was transferred into a large glass tray for drying. After drying, the remaining mixture was hand ground with a spatula and was sieved through a 60 mesh sieve. A free flowing granule mixture was obtained and stored in tightly sealed plastic containers for the hot pressing experiments.

Hot Pressing

The first batch of 3TiC+2Si powder mixture used in this study, was prepared by dry mixing method at Battelle Pacific Northwest laboratory. It was provided to us for the first hot pressing trial in our laboratory. The purpose of this hot pressing experiment was not only to determine the densification temperature range of the powder mixture, but also to form some comparative samples for our future study. Vacuum hot pressing of the powder was done under 4.5 ksi pressure first at 1350°C for 2 hours, followed by heating and holding at 1500°C for 10 minutes. Heating rate was 10°C/min during the hot pressing. The ram displacement was recorded during the hot pressing process, as an indicator of the material's densification. The densification of the material was slowed down after 90 minutes at 1350°C, as shown in Figure 1. There was not any significant change in ram position after the first 10 minutes of holding time at 1500°C, therefore, longer waiting period was not applied in this process, as it was suggested by Battelle program. After the hot pressing process, a tile with (3 x 3 x 0.39) inches dimension was obtained. The bulk density of the tile was determined by water immersion method as 4.07 g/cm^3, after grinding the surfaces. This composition was reported to yield 15% SiC + 85% Ti_3SiC_2 by our collaborators at Battelle. The calculated theoretical density of such a composition is 4.34 g/cm^3. If the hot pressed sample has a similar composition to that was reported, then, only 93-94% of the theoretical density has been achieved in this experiment. Based on this result, hot pressing of the two batches mentioned above, is on the way.

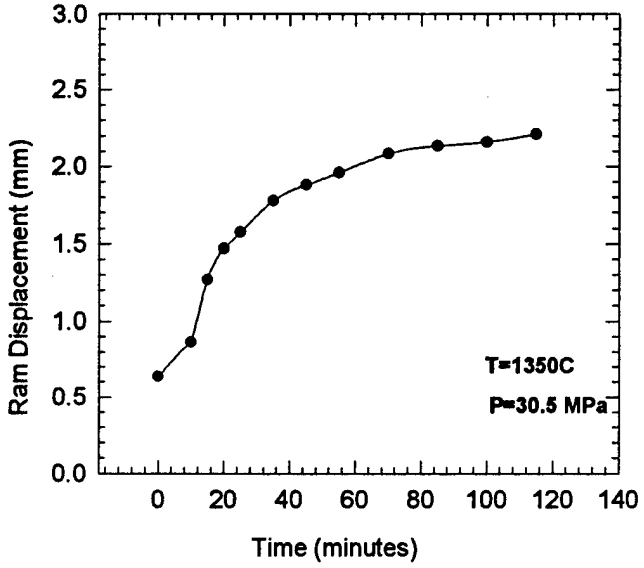

Figure 1: Densification of 3TiC+2Si powder mixture at constant temperature and pressure.

Task 2: Composite Characterization

The hot pressed sample was ground and cut with a diamond saw to make flexural strength test samples according to the MIL STD 1942 B. Flexural strength measurements are being carried out at Battelle as of this writing. Optical microscopy examination of the polished surface indicates the presence of extremely fine microstructure in hot pressed samples, as shown in Figure 3. Four distinct features were determined within the resolution limits of optical microscope; (i) the presence pores larger than 50 microns, (ii) acicular shaped disperse phase throughout the microstructure, SiC particles, (iii) continuous matrix phase, Ti_3SiC_2 phase and (iv) round shape shiny particles, most likely unreacted phase. X-ray diffraction analysis of the samples are currently being conducted to determine the phases present after the hot pressing. The more characterization of these samples will be carried out during the next phase of the study.

Schedule

The program is on schedule without any delays. As of November 1, 1995, 23% of the project budget has been spent.

Future Work

For the next reporting period the following activities are planned:

1. Complete flexural strength testing, phase analysis, and microstructural characterization of first (3TiC+2Si) composition.

2. Hot press billets with wet mixed 3TiC+2Si and 3TiC+3Si compositions.

3. Prepare specimens for flexural strength and wear tests.

4. Characterize microstructures and phases present in the samples mentioned above.

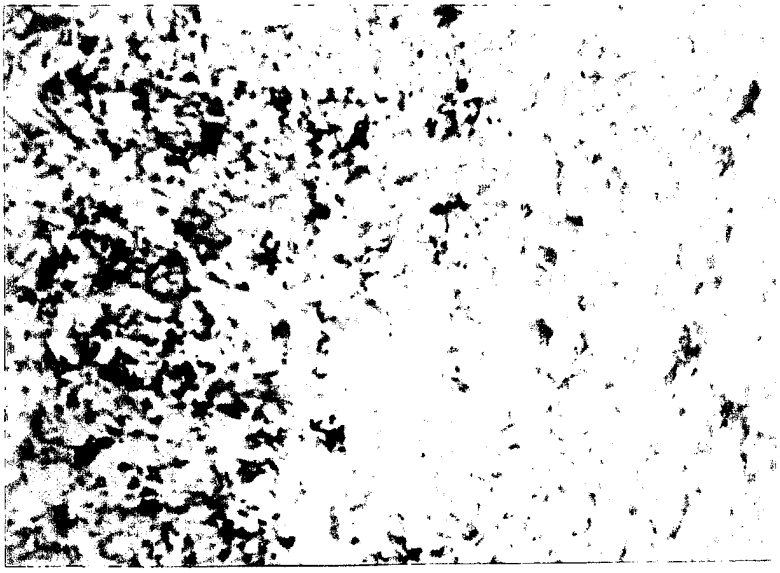

Figure 2: Microstructure of the first hot pressed sample with 3TiC+2Si starting composition (magnification 1000X).