Date: 8/8/78

Project Title: Evaluation of Shell Molds for Forming Precision Slip-Cast Sinterable Silicon Carbide Shapes

Project No: A-2191

Project Director: Mr. J. N. Harris

Sponsor: The Carborundum Company, Research and Development Division

Agreement Period: From 7/31/78 Until 11/30/78

Type Agreement: Standard Industrial Research Agreement dated 6/16/78

Amount: $16,513 (includes $1,000 for Patent and Data Rights)

Reports Required: Monthly Progress Reports; Final Report

Sponsor Contact Person(s):

Technical Matters

Contractual Matters (thru OCA)

Mr. Martin R. Kasprzyk
The Carborundum Company
P. O. Box 1054
Niagara Falls, NY 14302

Defense Priority Rating: N/A

Assigned to: Applied Sciences Laboratory/SEMTD (School/Laboratory)

COPIES TO:

Library, Technical Reports Section
EES Information Office
EES Reports & Procedures
Project File (OCA)
Project Code (GTRI)
Other

☑ Security Coordinator (OCA)
☑ Reports Coordinator (OCA)
GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: January 7, 1980

Project Title: Evaluation of Shell Molds for Forming Precision Slip-Cast Sinterable Silicon Carbide Shapes

Project No: A-2191

Project Director: Mr. J.N. Harris

Sponsor: The Carborundum Company

Effective Termination Date: 6/30/79

Clearance of Accounting Charges: 6/30/79

Grant/Contract Closeout Actions Remaining: None

- Final Invoice and Closing Documents
- Final Fiscal Report
- Final Report of Inventions
- Govt. Property Inventory & Related Certificate
- Classified Material Certificate
- Other

Assigned to: CMSL/MSD (EES/XXX/Laboratory)

COPIES TO:

Project Director
Division Chief (EES)
School/Laboratory Director
Dean/Director—EES
Accounting Office
Procurement Office
Security Coordinator (OCA)
Reports Coordinator (OCA)

Library, Technical Reports Section
EES Information Office
Project File (OCA)
Project Code (GTRI)
Other

CA-4 (1/79)
The Carborundum Company
P. O. Box 1054
Niagara Falls, New York 14302

Attention: Mr. Martin R. Kasprzyk


Gentlemen:

The purpose of this project is: (a) to develop a method for the slip casting of complex shapes of sinterable silicon carbide using a shell mold similar to those used for the investment casting of metals, and (b) to assist Carborundum in improving the casting characteristics of their current sinterable silicon carbide slips.

Work on this program was not initiated until 14 August 1978, when Mr. J. N. Harris visited the Carborundum Research Facilities to discuss the program with Carborundum personnel. The current status of slip-cast sinterable silicon carbide materials, slip preparation and casting procedures were reviewed and materials were provided to be hand carried to Georgia Tech to begin studies on improving slip casting characteristics. The automotive turbocharger rotor prototype shape was examined and discussions were held with Carborundum personnel on the techniques currently being examined to fabricate this part.

Work was initiated at Georgia Tech on preparation of silicon carbide shapes using the materials provided by Carborundum. Initial efforts were directed towards duplicating the Carborundum slip by following exactly the directions provided. All of the components necessary were provided by Carborundum or were available at Georgia Tech except for the ARCO Chemical Company SMA 1440H. This material was not available locally and required a three-week delivery. A small quantity was requested from Carborundum and received in approximately one week. The first batch was prepared with all ingredients except the SMA 1440H.

Apparent viscosity was measured with a Brookfield viscometer using a number 3 spindle and compared with the sample of Carborundum 89-13. Apparent viscosity measurements are shown in Table I.
TABLE I

APPARENT VISCOSITY AND pH OF SINTERABLE SILICON CARBIDE SLIP

<table>
<thead>
<tr>
<th>Brookfield</th>
<th>RPM</th>
<th>GT-01</th>
<th>Carborundum 89-13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent</td>
<td>6</td>
<td>1900</td>
<td>5460</td>
</tr>
<tr>
<td>Viscosity</td>
<td>12</td>
<td>1170</td>
<td>3100</td>
</tr>
<tr>
<td>(#3 Spindle)</td>
<td>30</td>
<td>540</td>
<td>1504</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>290</td>
<td>900</td>
</tr>
<tr>
<td>pH</td>
<td></td>
<td>7.1</td>
<td>6.9</td>
</tr>
</tbody>
</table>

Plaster molds were prepared from a two inch diameter truncated conical model with a hemispherical nose for comparison of casting characteristics. A second batch of slip was prepared using the SMA-1440H. The beginning of fall quarter classes occurred before the student working on this program could make castings or finish the second batch of slip. He has not met the hours promised prior to the beginning of the fall quarter, therefore, work had to be transferred to a full time technician. Unfortunately, the full time technician will not be fully available until October 11, 1978. At that time he will begin to apply essentially full time effort until the program is back on schedule.

No effort was accomplished on preparation of shell molds during the reporting period except to obtain the necessary materials to be prepared to fabricate molds when the patterns were provided by Carborundum. The first batch of wax rotor patterns arrived during the reporting period, however, all but four of these were badly broken. The four had small chips missing on one or more blades. The polystyrene patterns did not arrive until the first week of October. Work on shell molds will be initiated beginning 11 October 1978.

The next progress report will cover the period 22 September to 21 October 1978.

Respectfully submitted,

Joe N. Harris
Project Director

jw
The Carborundum Company  
P.O. Box 1054  
Niagara Falls, New York 14302  
Attention: Mr. Martin R. Kasprzyk  


Gentlemen:

A. Preparation of Shell Molds

Shell Molds were prepared using both the wax and polystyrene patterns of the automotive turbocharger rotor prototype provided by Carborundum. The shell mold compositions were made with various grain size suspensions of graphite particles bonded with polyvinyl alcohol.

Both the wax and polystyrene patterns were cleaned by immersing them briefly into trichloroethylene and then into acetone. After drying the patterns were dipped into a 25 weight percent polyvinyl alcohol solution. Upon removal they were quickly sprinkled with -50 + 140 mesh graphite grain. After drying at ambient temperature for six hours the above process was repeated and the rotors left to dry over night. The rotors were then dipped into the graphite slurry and sprinkled with -25 + 50 mesh graphite grain to provide the final stucco coat. After thorough drying at ambient temperature the patterns were removed.

The shell molds containing the wax patterns were placed briefly in a furnace at 870 degrees C until the wax vapors began to "flash". They were then transferred to a 150 degrees C oven to melt the remainder of the wax. The first shell mold formed around a wax pattern was broken accidentally in removing it from the oven after melting out all the wax. This occurred due to softening and loss of strength of the PVA binder at 150 degrees C. The 150 degrees C temperature is not sufficiently high to cause plastic flow of the PVA and deformation of the mold. This experience showed that the molds will require very careful handling at this temperature. No further problems are anticipated in handling the molds after removing the wax cores.

Initial attempts to remove the polystyrene chemically were made by soaking the mold in acetone. The acetone dissolved the polystyrene, but rate of
removal was considered too slow. By soaking the mold in trichlorethylene the polystyrene pattern was removed in approximately 48 hours. The initial PVA bonded shell mold will be filled with Carborundum slip batch 89-13.

B. Sinterable Silicon Carbide Slips

The apparent viscosity of the Carborundum slip batch 89-13 decreased slightly from 31 August to 20 September 1978. Measurements for these two dates were as follows:

<table>
<thead>
<tr>
<th>RPM</th>
<th>31 August</th>
<th>30 September</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>5460</td>
<td>5120</td>
</tr>
<tr>
<td>12</td>
<td>3100</td>
<td>2920</td>
</tr>
<tr>
<td>30</td>
<td>1504</td>
<td>1440</td>
</tr>
<tr>
<td>60</td>
<td>900</td>
<td>864</td>
</tr>
</tbody>
</table>

The slip batch prepared at Georgia Tech according to Carborundum instructions except without the SMA 1440 H, also decreased in apparent viscosity as shown:

<table>
<thead>
<tr>
<th>RPM</th>
<th>31 August</th>
<th>30 September</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>1900</td>
<td>1020</td>
</tr>
<tr>
<td>12</td>
<td>1170</td>
<td>580</td>
</tr>
<tr>
<td>30</td>
<td>540</td>
<td>242</td>
</tr>
<tr>
<td>60</td>
<td>290</td>
<td>160</td>
</tr>
</tbody>
</table>

A check of the solids content indicates that Carborundum batch 89-13 has an actual solids content of 62.2 percent rather than the reported 60 percent. The Georgia Tech prepared slip has a solids content of 60.9 percent.

No further work was conducted on slips due to the loss of the student assistant working this problem. The full time technician now assigned will begin immediately to prepare a fresh batch of slip following Carborundum's directions, except solids content will be increased to 62.2 percent to duplicate batch 89-13.

C. Further Work

Personnel turnover has prevented reaching the first two milestones on "initial slip studies" and "part casting". The addition of a full time
technician to the program is expected to bring it back on schedule by 21 November.

Respectfully submitted.

Joe N. Harris
Project Director
November 21, 1978

The Carborundum Company
P.O. Box 1054
Niagara Falls, New York 14302

Attention: Mr. Martin R. Kasprzyk


Gentlemen:

A. Preparation and Filling of Shell Molds

Shell molds were prepared using polystyrene patterns. Some were made with graphite powders and a polyvinyl alcohol binder. Others were prepared with powders bonded with colloidal silica (Ludox AS).

The first five molds were prepared by dipping the rotors into a slurry made from 25 weight percent -325 mesh graphite powder in a 10 percent polyvinyl alcohol solution. Upon removal they were coated with -50 +140 mesh graphite grain. After drying this process was repeated. Again after drying a final coat of slurry was applied, followed by coating with a coarser graphite grain (-25 +50 mesh). These patterns were removed by soaking the molds in trichloroethylene.

The first attempt to fill a shell mold was made using a mold made in the previous month with a wax pattern. The pattern was removed by heating. The heat softened the PVA binder and handling of the warm mold caused some damage to the blade roots and tips. This caused leakage through the shell when an attempt was made to fill it with slip.

A mold in which the polystyrene pattern was leached out with trichloroethylene was soaked in water prior to filling in an attempt to slow the casting rate. This softened the PVA binder and again resulted in leakage while attempting to fill it with slip. The next mold was filled without incident. This mold was rotated at an approximate 80 rpm and was filled under vacuum. After filling, rotation was continued with the mold under vacuum for an additional four hours. Upon removal the mold was soft and pliable, apparently due to the water in the slip softening the binder. However, the mold retained its shape without damage to the cast part. This mold was hand carried to Carborundum by Mr. Kasprzyk.
An attempt was made to fill another mold during Mr. Kasprzyk's visit on 9 November. Slip spilled on the rotating coupling causing it to bind, creating a delay in the casting process. When this was corrected the mold had developed a leak, apparently due to water in the slip softening the mold and causing leaks at the thinnest parts of the mold.

During Mr. Kasprzyk's visit a decision was made to examine the effect of a colloidal silica binder to replace the PVA. The colloidal silica could be rendered insoluble by heating the mold to about 400 degrees F., eliminating the solubility of the mold by the water in the slip. Several molds were prepared but, all of them cracked while dissolving the polystyrene with trichloroethylene, acetone or toluene. Apparently the organic solvent causes the polystyrene pattern to swell before it softens, thus creating a stress on the mold. The PVA binder is more pliable and does not fail catastrophically. However, swelling of the patterns during removal may have caused unseen cracks in the PVA bonded shell molds.

The remaining PVA molds were recoated twice with the PVA slurry and graphite grain after removal of the polystyrene pattern, in an effort to seal any cracks and to strengthen the molds. Two of these molds were filled and one shipped to Carborundum by Federal Express on 20 November.

### B. Silicon Carbide Slip Preparation

Several silicon carbide slips have been prepared following the procedures of Carborundum. These slips were prepared for comparison with Carborundum Slip 89-13 and for use in filling shell molds.

The apparent viscosity of the Carborundum slip has varied considerably over a period of time as measured with Brookfield Viscometer and a number three spindle. These measurements along with pH and solids content are shown in Table I. Table II shows data for five slips prepared at Georgia Tech with solids content varying from 59.3 to 68.9 percent.

In discussions with Mr. Kasprzyk during his visit at Georgia Tech on 9 November it was decided that emphasis should be placed on developing the shell molds and not on improving the silicon carbide slips. Therefore, no effort has been placed on producing slips of greater than 70 percent solids, on lowering the viscosity of existing or new slips, or on improving casting characteristics in conventional plaster molds.
C. Future Work

Work will continue on developing and filling shell molds to be sintered and evaluated at Carborundum.

Respectfully submitted,

Joe N. Harris
Project Director
### TABLE I

APPARENT VISCOSITY MEASUREMENTS ON CARBORUNDUM SILICON CARBIDE SLIP 89-3

<table>
<thead>
<tr>
<th>Brookfield Viscometer No. 3 Spindle @</th>
<th>8/31</th>
<th>9/5</th>
<th>10/20</th>
<th>11/7</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 rpm</td>
<td>5460</td>
<td>5820</td>
<td>5120</td>
<td>7700</td>
</tr>
<tr>
<td>12 rpm</td>
<td>3100</td>
<td>3230</td>
<td>2920</td>
<td>4410</td>
</tr>
<tr>
<td>30 rpm</td>
<td>1505</td>
<td>1580</td>
<td>1440</td>
<td>2125</td>
</tr>
<tr>
<td>60 rpm</td>
<td>900</td>
<td>950</td>
<td>865</td>
<td>1280</td>
</tr>
<tr>
<td>Percent Solids</td>
<td>-----</td>
<td>-----</td>
<td>62.23</td>
<td>62.31</td>
</tr>
<tr>
<td>pH</td>
<td>6.9</td>
<td>6.8</td>
<td>6.8</td>
<td>6.7</td>
</tr>
<tr>
<td>Property</td>
<td>Slip Number</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>--------------------------</td>
<td>-------------</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>GT-1</td>
<td>GT-2</td>
<td>GT-3</td>
<td>GT-4</td>
</tr>
<tr>
<td>Percent Solids</td>
<td>60.9</td>
<td>59.3</td>
<td>63.4</td>
<td>66.2</td>
</tr>
<tr>
<td>pH</td>
<td>6.7</td>
<td>6.8</td>
<td>6.7</td>
<td>7.0</td>
</tr>
<tr>
<td>Apparent Viscosity @</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6 rpm</td>
<td>1020</td>
<td>1385</td>
<td>1800</td>
<td>7200</td>
</tr>
<tr>
<td>12 rpm</td>
<td>580</td>
<td>690</td>
<td>1060</td>
<td>3710</td>
</tr>
<tr>
<td>30 rpm</td>
<td>290</td>
<td>315</td>
<td>505</td>
<td>1700</td>
</tr>
<tr>
<td>60 rpm</td>
<td>160</td>
<td>170</td>
<td>260</td>
<td>905</td>
</tr>
</tbody>
</table>
The Carborundum Company  
P. O. Box 1054  
Niagara Falls, New York 14302  
Attention: Mr. Martin R. Kasprzyk  

Subject: Monthly Progress Report No. 4, "Evaluation of Shell Molds for

Gentlemen:

A. Preparation and Filling of Carbide Shell Molds

The final three plastic rotor patterns from the original group provided by Carborundum were used to prepare shell molds which were subsequently filled with sinterable silicon carbide slip.

The shell mold for casting number 8 consisted of five coats applied by dipping the pattern in a 10 percent solution of polyvinyl alcohol (PVA) containing 25 percent by weight of -325 mesh graphite grain. After each dipping a stucco coating of coarser graphite was applied by sprinkling the grain uniformly over the wet coating. The first three "stuccos" were -50 +140 mesh graphite. The final two were -25 +50 mesh graphite. After drying the mold the plastic pattern was leached out by soaking the entire mold in trichloroethylene at 25° C for 48 hours. The mold was filled while rotating under vacuum with a 68.9 percent solids silicon carbide slip designated GT-5. Apparent viscosity measurements were made on the slip just prior to filling the mold. Measurements made with a Brookfield Viscometer were: 1500, 1000, 480 and 280 centipoise at 6, 12, 30 and 60 rpm, respectively.

Casting number 9 used a shell mold similar to casting 8 except the final two coatings of stucco grain were 46 grit silicon carbide rather than -25 +50 mesh graphite. The plastic pattern was leached out using the same procedure as with the previous mold. The mold was filled with the 68.9 percent solids silicon carbide slip designated GT-5. Apparent viscosity measurements just prior to filling were: 1865, 1050, 535 and 305 centipoise at 6, 12, 30 and 60 rpm, respectively.
The shell mold for casting number 10 was identical to the one used for casting number 8. This mold was filled with a 70.2 percent solids sinterable silicon carbide slip designated GT-6. Apparent viscosity measurements just prior to filling the mold were: 4840, 2900, 1325 and 760 centipoise at 6, 12, 30 and 60 rpm, respectively.

Twenty-four new plastic patterns were received from Carborundum on 5 December. A number of shell molds were prepared with either five or six coatings of the PVA-graphite slurry and with graphite and/or silicon carbide stucco grain. An unexpected development was the difficulty of leaching the new plastic rotors from the shell molds. The shell molds consisting of six coats cracked after soaking in the trichloroethylene solvent over a weekend. The molds with five coats did not crack but a residue was left in the mold after a total of 120 hours of soaking in trichloroethylene at 25°C. Efforts will be continued to completely remove the plastic patterns and to fill additional molds.

The three filled molds described above and possibly a fourth filled mold from the new plastic rotor patterns will be shipped to Carborundum during December.

B. Future Work

This program was initially scheduled to terminate on 21 December 1978, however, it is estimated that approximately $1,500 to $2,000 dollars in unexpended funds will be available for a small effort in the month of January. In addition to preparing a final report it is estimated that these remaining funds can best be used to fill already prepared shell molds (provided all of the plastic patterns can be removed) with different viscosity and solids content sinterable silicon carbide slips. The choice of slips will be based on the results of Carborundum’s sintering of molds filled with their slip and the Georgia Tech prepared silicon carbide slips designated 5 and 6. Any further efforts will require a new contract or an extension of the current contract and additional funding.

Respectively submitted,

Joe N. Harris
Project Director

jw