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# **SYNTHESIS AND DESIGN OF SILICIDE INTERMETALLIC MATERIALS**

## **DOE/AM QUARTERLY PROGRESS REPORT October-December 1998**

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### **INTRODUCTION:**

The overall objective of this program is to develop structural silicide-based materials with optimum combinations of elevated temperature strength/creep resistance, low temperature fracture toughness, and high temperature oxidation and corrosion resistance for applications of importance to the U.S. processing industry. A further objective is to develop silicide-based prototype industrial components. The ultimate aim of the program is to work with industry to transfer the structural silicide materials technology to the private sector in order to promote international competitiveness in the area of advanced high temperature materials and important applications in major energy-intensive U.S. processing industries.

The program presently has a number of industrial connections, including a CRADA with Johns Manville Corporation targeted at the area of MoSi<sub>2</sub>-based high temperature materials for fiberglass melting and processing applications. We are also developing an interaction with the Institute of Gas Technology (IGT) to develop silicides for high temperature radiant gas burner applications, for the glass and other industries. With the Exotherm Corporation, we are developing advanced silicide powders for the fabrication of silicide materials with tailored and improved properties for industrial applications.

In October 1998, we initiated a new activity funded by DOE/OIT on "Molybdenum Disilicide Composites for Glass Processing Sensors". With Accutru International Corporation, we are developing silicide-based protective sheaths for self-verifying temperature sensors which may be used in glass furnaces and other industrial applications. With Combustion Technology Inc., we are developing silicide-based periscope sight tubes for the direct observation of glass melts.

### **Accutru International Interaction:**

R.G. Castro and J.J. Petrovic visited Accutru International Corporation in Houston, Texas on 5 November 1998, to discuss and plan interactions associated with the new activity on molybdenum disilicide composites for glass processing sensors. It was decided to initiate activities by

performing baseline molten glass corrosion and thermal shock tests on standard thermocouples in alumina sheaths which were then plasma spray overcoated with  $\text{MoSi}_2$ .

### Glass Corrosion Resistance of $\text{MoSi}_2$ Coated Thermocouples:

A molten glass corrosion resistance test was performed at  $1050^\circ\text{C}$  for 380 hours (approximately 16 days) in an alkali borosilicate fiberglass composition. Figure 1 shows the test configuration. After the thermocouple was exposed for 380 hours in molten glass at  $1050^\circ\text{C}$ , it was removed from the molten glass. A temperature recorder showed no problems in the thermocouple temperature reading during the 380-hour test. Figure 2 shows optical photos of the tested thermocouple. As we have reported previously, the maximum corrosion rate of  $\text{MoSi}_2$  occurs at the glass-air line. However, a degradation of the  $\text{MoSi}_2$  coating was observed at the top of the heating furnace where the temperature was  $500^\circ\text{C}$ . This degradation was due to oxidation pesting of the  $\text{MoSi}_2$ . We will analyze the microstructures of the  $\text{MoSi}_2$  coated thermocouple to characterize the above observations in more detail.

We plan to put a protective coating of  $\text{ZrO}_2$  or  $\text{ZrSiO}_4$  over the  $\text{MoSi}_2$  to reduce the corrosion rate at the glass-air line. A protective coating of  $\text{SiO}_2$  will be employed to reduce oxidation pesting of the  $\text{MoSi}_2$  at the  $500^\circ\text{C}$  temperature location.

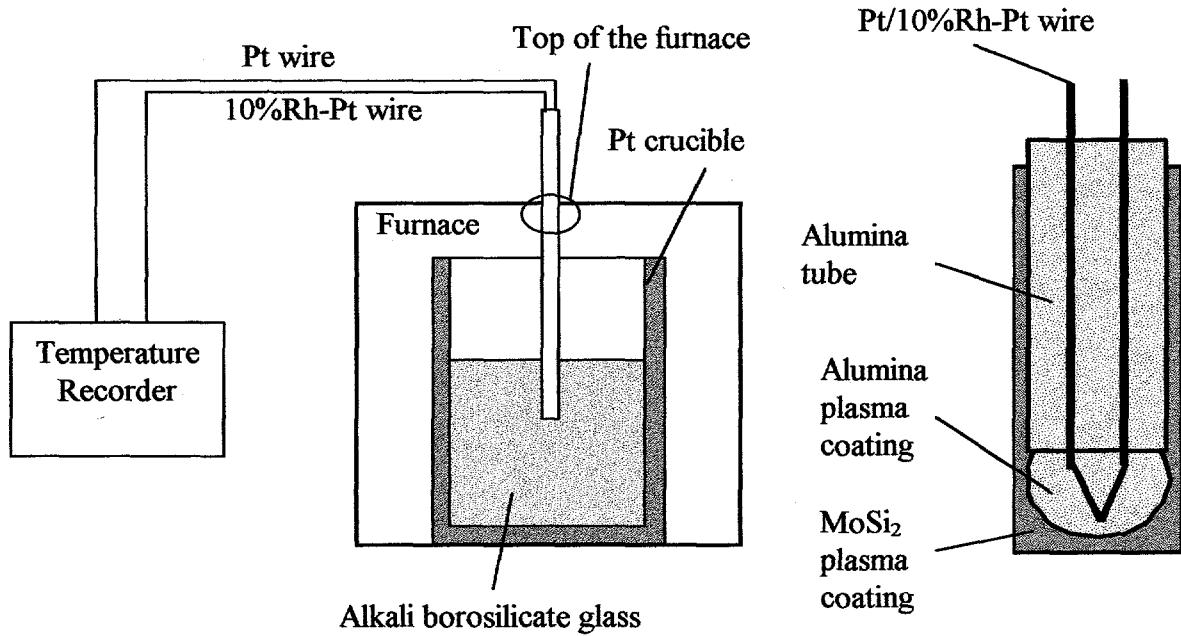


Figure 1: Configuration for the molten glass corrosion test of a Type S alumina-sheathed thermocouple with a  $\text{MoSi}_2$  plasma sprayed overcoating.

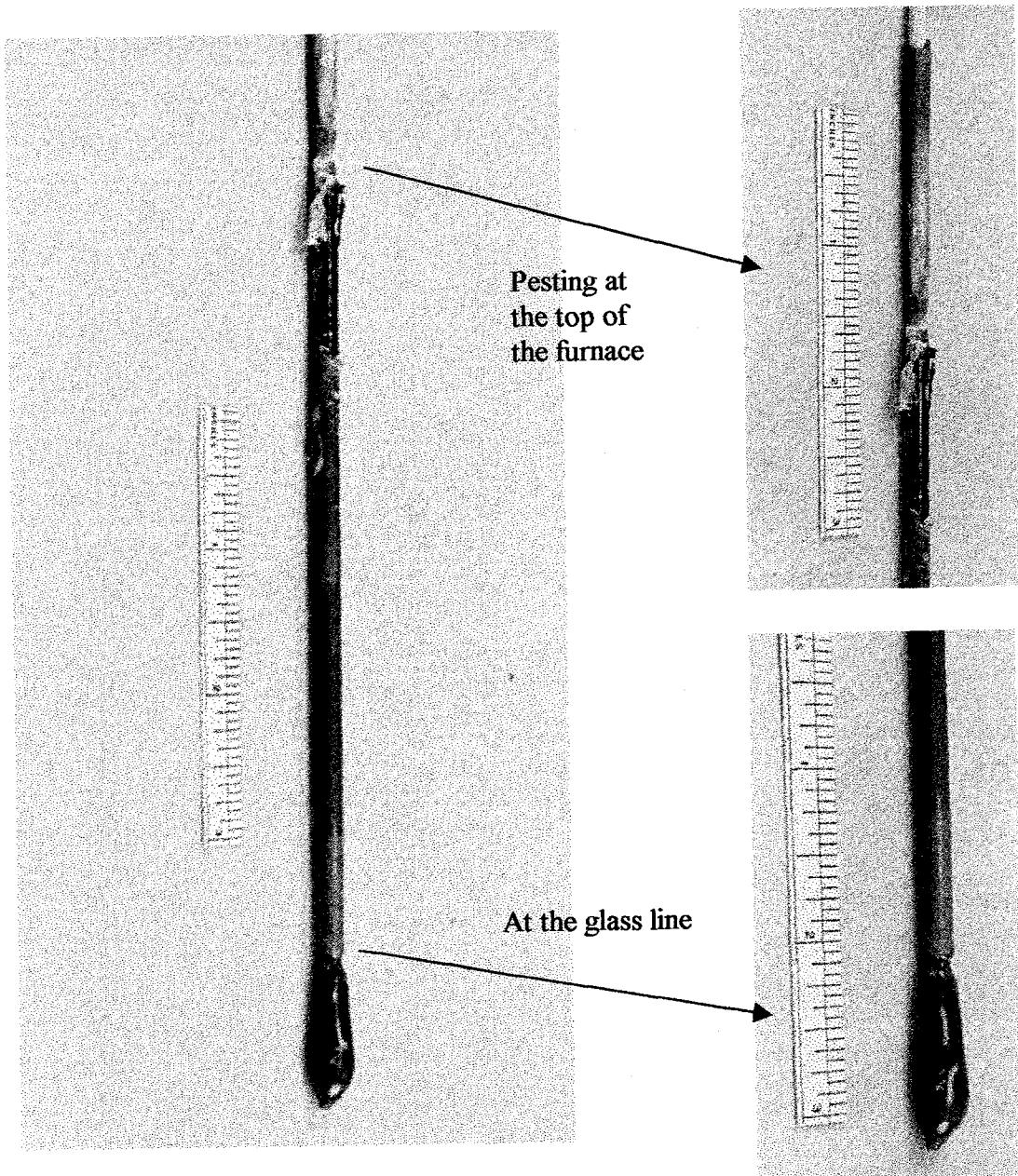


Figure 2: Optical photos of MoSi<sub>2</sub> coated S-type thermocouple after the molten glass corrosion test for 380 hours at 1050°C.

### **Thermal Shock Resistance of MoSi<sub>2</sub> Coated Thermocouples:**

An experimental set-up has been established for thermal shock testing of thermocouple sheath materials that have been coated by plasma spraying (Figure 3). The test procedure will rapidly expose thermocouple sheath materials to temperatures up to 1600 °C by immersing coated thermocouples inside of an air furnace followed by rapid cooling to room temperature. A number of heating and cooling cycles will be performed to evaluate the performance of the plasma sprayed coating on the thermocouple. Initial investigation will concentrate on plasma sprayed MoSi<sub>2</sub>-based materials.

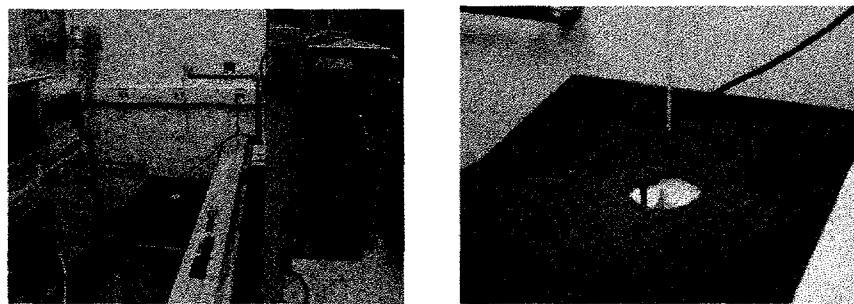


Figure 3: Thermal shock equipment for immersing thermocouples into an air furnace for investigating the thermal shock behavior of plasma sprayed coatings on Al<sub>2</sub>O<sub>3</sub> thermocouples.

### **Silicide Joining:**

A schematic of a shear stress-linear displacement plot of a MoSi<sub>2</sub>-stainless steel brazed joint is shown in Figure 4. The interfacial shear stress was calculated by balancing the normal force exerted on the MoSi<sub>2</sub> over the sheared area at the joint interface. The peak stress in the plot corresponds to the peak load at which shear failure occurred in the joint. A corresponding load drop followed by a moderate increase in the load represents the friction in the sliding process. Once the two rings (MoSi<sub>2</sub> and stainless steel) have slid apart by 0.15 mm, the load drops monotonically. A total of 5 samples were tested. All of these samples were brazed according to the procedure outlined earlier, and employed a brazing foil thickness of 25 micrometers. With the exception of one sample, all of the shear failure occurred in the braze material. The average shear stress was calculated to be 72.3 MPa. The interfacial shear stress values ranged from 60.3 to 83.1 MPa.

The sample which was excluded from the statistics exhibited compressive cracking in the MoSi<sub>2</sub>. Calculations revealed that the normal stress on the MoSi<sub>2</sub> was on the order of 50 MPa, which is low for MoSi<sub>2</sub>. We believe that this sample might have either had inherent defects and/or large residual stresses to cause it to fail at this low stress.

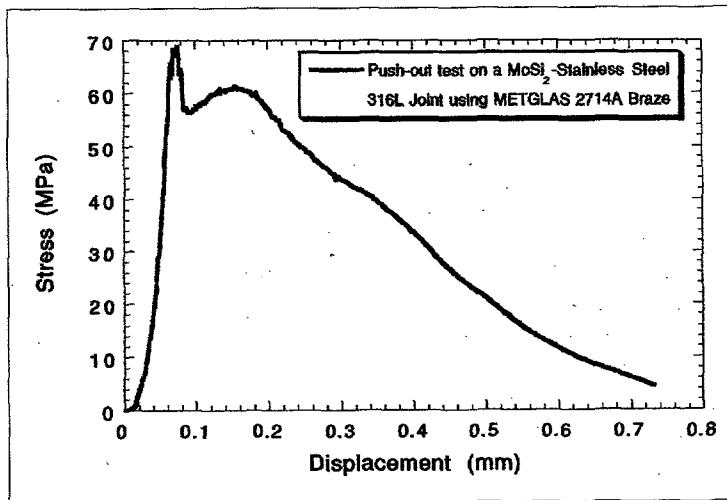


Figure 4: Shear strength test on a  $\text{MoSi}_2$ -stainless steel brazed joint.

#### Robotic Plasma Spray Facility:

Training on the Fanuc S10 robot has been completed. The robot has been programmed to coat  $\text{Al}_2\text{O}_3$  thermocouples and tube materials with  $\text{MoSi}_2$ -based material,  $\text{Y}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  (Figure 5). This operation will be used to support the development of sheath materials for glass processing sensors.

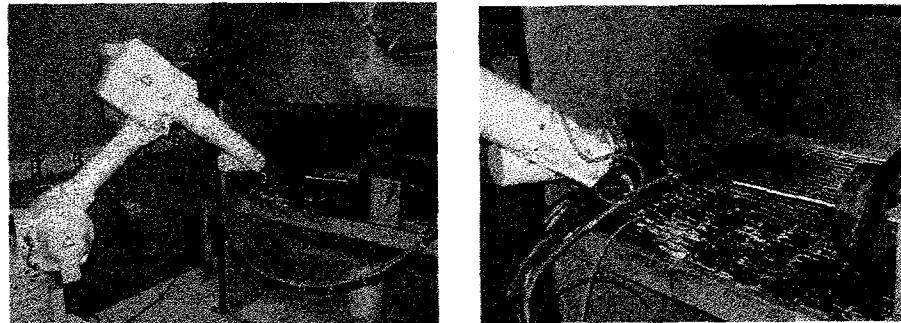


Figure 5: Robotic manipulation for plasma spraying thermocouple sheaths for glass processing sensors.