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C/ORNL96-0440

Metals and Ceramics Division

CRADA Final Report
for CRADA Number ORNL96-0440

HIGH TEMPERATURE PARTICLE FILTRATION TECHNOLOGY

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Dow Corning Corporation

Date Published – August 2001

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ABSTRACT

High temperature filtration can serve to improve the economic, environmental, and energy performance of chemical processes. This project was designed to evaluate the stability of filtration materials in the environments of the production of dimethyldichlorosilane (DDS). In cooperation with Dow Coming, chemical environments for the fluidized bed reactor where silicon is converted to DDS and the incinerator where vents are cornbusted were characterized. At Oak Ridge National Laboratory (ORNL) an exposure system was developed that could simulate these two environments. Filter samples obtained from third parties were exposed to the environments for periods up to 1000 hours. Mechanical properties before and after exposure were determined by burst-testing rings of filter material. The results indicated that several types of filter materials would likely perform well in the fluid bed environment, and two materials would be good candidates for the incinerator environment.

CRADA OBJECTIVE

The objective of this Cooperative Research and Development Agreement (CRADA) project involving the Dow Corning Corporation and ORNL was to demonstrate the feasibility of high temperature particle filtration in two applications in the process stream for producing DDS. Dow Coming Corporation is a market leader in the production of DDS which is utilized as an intermediate in a wide variety of silicone products. As such, in Carrollton, Kentucky they currently operate the largest plant of its type for the production of DDS, housing several generations of production streams. Production efficiency for their energy-intensive process is adversely affected by the entrainment of unreacted silicon particulates in the product stream. This causes reduced throughput and run times for this semi-batch process. Previously tested filter systems resulted in either unacceptable material interactions or pin-holing in which the efficacy of the filter was lost. In addition, substantial energy and capital loss is engendered by the need for exhaust gas cleanup from an incinerator which oxidizes waste chlorosilanes and entrained silicon. The ability to remove particulates from the hot incinerator exhaust gas would allow heat recovery. The project was therefore designed to assess the applicability of high temperature filter materials to solve these problems.

ASSESSMENT OF CRADA

The results of the exposure testing and subsequent mechanical property evaluation of filter specimens successfully demonstrated that existing materials may be used in both filtration applications in manufacturing DDS as identified by Dow Corning. Eight different materials from six different suppliers were found to survive prolonged exposure in the lower temperature, fluidized bed application. Two materials from two different suppliers were also found to survive long-term exposure to the higher temperature, more challenging environment of the thermal oxidizer (incinerator).

CRADA BENEFIT TO DOE

This CRADA has enhanced capabilities at ORNL for the exposure of materials to extreme environments and the assessment of their behavior. The work has also identified a set of filter materials that would be applicable in a wide range of high temperature applications. These will likely be important in many industrial processes leading to significant economic, environmental, and energy benefits.

TECHNICAL DISCUSSION

This project was a joint effort between ORNL and Dow Coming to assess the use of high temperature filtration materials for use in the process stream for the production of DDS. Fiber-reinforced composites in particular have excellent thermal shock capability due to the toughening effect of the fiber reinforcement. The increased toughness also prevents filter failure due to the mechanical stress of handling or system upset. Samples of filters were obtained from third party suppliers.

Two applications were studied. The first requires filtration of silicon particles from the process stream which exits the fluidized bed reactor. This is a lower temperature environment in which un-reacted silicon, product DDS, and various byproducts are present. The second application involves filtration of high temperature, much smaller particle-size fumed silica which exits the incinerator at the exhaust end of the process line.

ORNL developed basic compatibility information on filter materials in simulated process environments. Dow Coming was to have provided test units in slip-streams to offer comparative compatibility and operational testing in the industrial setting. Follow-on efforts were to focus on small-scale filtration demonstrations utilizing sub-commercial scale filters and eventual full-scale testing.

Exposure System

A bench-scale furnace and gas flow system was designed and built to simulate two particular processing environments (Fig. 1). The first environment, called the fluidized bed reactor (FBR) environment, was simulated at 290°C with typical flows of methyl chloride and DDS. The second environment, called the thermal oxidizer (THROX) environment, was simulated at 1040°C with gas flow rates of 9.6 cm^3/min of hydrogen chloride and 887 cm^3/min of mixed gas consisting of nitrogen, oxygen, carbon dioxide, and water vapor. Filter specimens were collected from various manufacturers and exposed to simulated process environments for different times. The filter media were prepared from various materials, included carbon, silicon carbide, oxide ceramics, and metal alloys. All of the samples were in the form of rings 2.5 cm in height obtained from candle filter shapes.

Mechanical Properties Testing

After exposure, mechanical properties testing was carried out to determine the feasibility of using the various types of filter materials. Three unexposed samples of each material were also tested. The strength test consisted of filling the volume of the ring with'elastomer and bursting the sample by applying pressure to the elastomer with a plunger in a test frame. Displacement was measured via sensors on the circumference of the ring and the burst pressure was recorded.

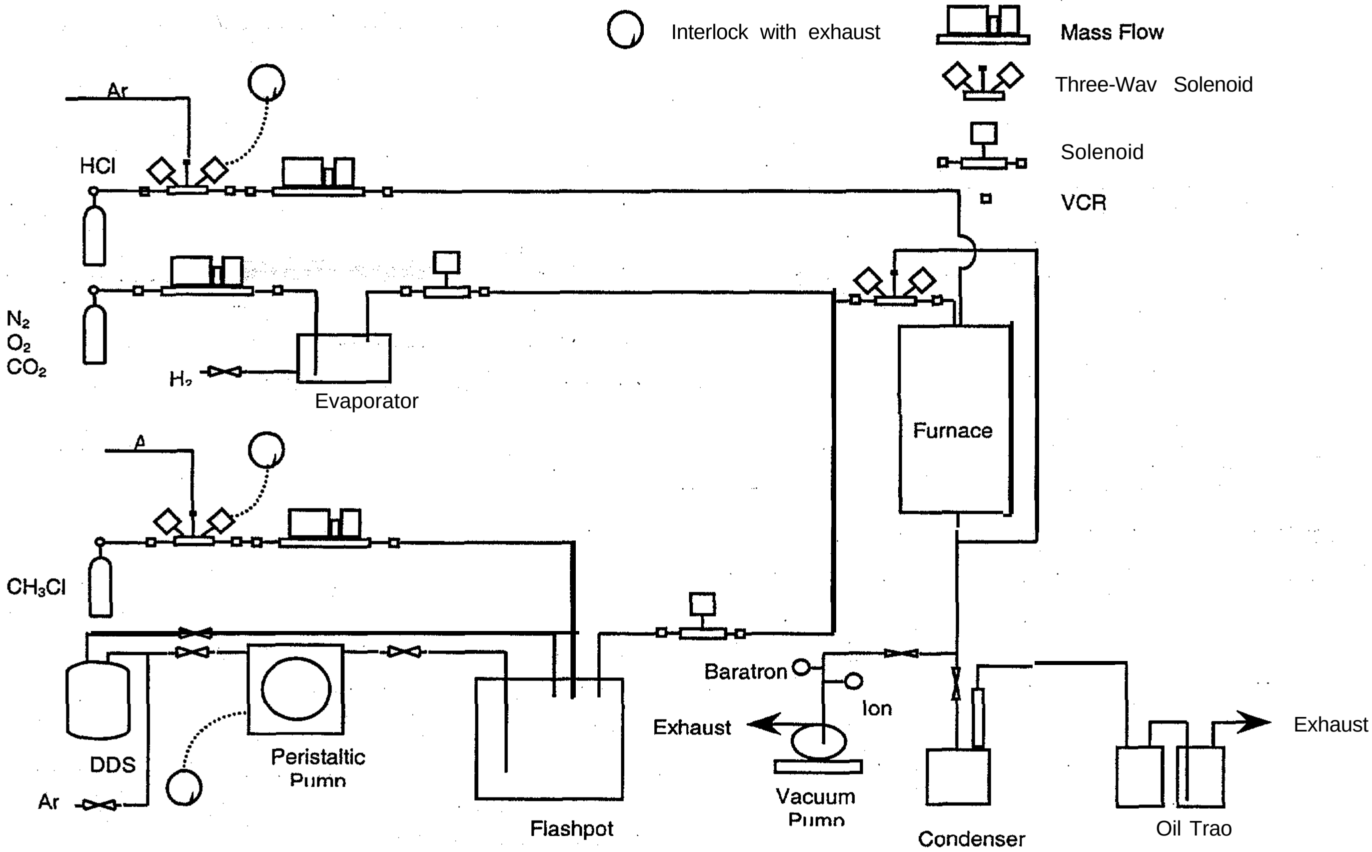

Fig. 1. Flow diagram of experimental exposure system.

RESULTS

All rings selected for FBR testing were exposed for 24 hours and rupture tested to assess their mechanical strength (Table I). Several material types were eliminated for various reasons including failure before testing. It was collectively decided to pursue seven material types for the 1000 hour FBR exposure. Before the 1000 hour test, however, FBR simulations were run for 72 hours with the filters in direct contact with captured silicon powder from the Dow Corning-Carrollton manufacturing plant. Burst testing of these particular samples demonstrated that no loss in mechanical strength occurred due to the exposure to the furnace conditions and contact with the powder. No significant mechanical strength loss occurred after the 1000-hour furnace exposure of the same materials. Apparently, all materials which we initially down-selected were promising candidates for the FBR application (Table I).

The initial 24-hour THROX simulations resulted in positive results for all the tested samples (Table I). Rupture testing of these materials demonstrated no significant loss of mechanical strength due to exposure. The 1000-hour exposures, however, resulted in significant/catastrophic strength loss for all samples except the Honeywell SiC/SiC and the Pall Corporation Vitripore 326 (Table I).

Table I. Results of the Mechanical Property Testing of Filter Specimens

Manufacturer	Type	Control	FBR	FBR	FBR	Therm. Ox.	Therm. Ox.
			24 h	72 h	1000 h	24 h	1000 h
		(Mpa)	(Mpa)	(Mpa)	(Mpa)	(Mpa)	(Mpa)
Honeywell	SiC/SiC	37.36	41.39	45.85	31.55	30.64	24.4
Honeywell	PRD-66C	6.75	6.6	7.86	6.12	6.37	0.647
Honeywell	PRD-66M	4.72	5.41	6.42	6.67	5.7	0.537
3M Company	SiC/Nextel	11.34	9.64	13.14,	10.65	Incomplete	2.92
Techniweave	Carbon/SiC	11.4.	11.34	11.41	12.23	FBR Only	
Techniweave	Mullite/Nextel 610	22.19	Thermal Oxidizer Only			22.37	1.8
Techniweaver	Mullite/Nextel 720	14.69	Thermal Oxidizer Only			13.57	1.89
Smart Ceramics	Oxide	28.59	32.69	26.37	24.84	29.23	1.2
McDermott Technologies	Oxide	8.94	9.11		8.87	8.32	0.813
Amercom	SiC	1.15	0.56””		**		
Amercom	Oxide	0.75	0.64””		**		
Blasch	Oxide	4.62	4.65	” ”	**		
Pall Corporation	Hastelloy X	205.71	256.86	260.85	208.4	FBR only	
Pall Corporation	Hastelloy C276	65.6	60.37*			FBR only	
Pall Corporation	Inconnel 600	144.35	148.21”			FBR only	
Pall Corporation	Vitripore 326	15.57	Thermal Oxidizer only			14.48	15.3

*Dropped since not commercially available

**Eliminated by properties

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