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DEVELOPMENT OF CHEMICAL
VAPOR COMPOSITE, CVC MATERIALS

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SIGNIFICANT ACCOMPLISHMENTS:

┌ A powder/fiber sensor and controller for CVC reactors was successfully designed, fabricated, tested, and modified. A carbon fiber feeder and controller for a CVC SiC reactor was designed and fabricated but has not yet been tested. SiC material was fabricated using SiC powder ranging in size from 30 to 40 microns. SiC was also fabricated using multiple SiC powders of 10, 30, or 90 microns. Still other SiC materials were fabricated using platelets of 30 or 40 microns. The strength of this SiC material was low due to impurities in the material: ways to eliminate material impurities are currently being thoroughly investigated. Data was assembled on the operation and performance of a typical furnace using a metal fan.

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TASK--Equipment modification:

The objective of this task is to design and implement particulate feeders and manifolds which can accurately monitor and control the particulate feed rate (grams/hour) into the CVC reactor.

A powder/fiber sensor and controller for CVC reactors was successfully designed, fabricated, tested, and modified. The sensor functions with the user of a laser. Laser light is directed across the flow of gas and particulates in the plumbing to the reactor. The sensor, set at right angles to the laser, detects the amount of light reflected off the particulates. Figure A illustrates the design principle, and Figure B shows the sensor in the CVC control system. The controller compares the sensor readings to a predetermined setpoint and accordingly regulates the particulate feeder to produce a uniform flow of particulates. Figure C is a photo of the sensor and the controller in operation. This design has been successfully used to control accurately a wide range of both powder and carbon fiber sizes, at flow rates varying from 0.01 grams/hour to 100 grams/hour. Currently, three particulate sensors and controllers have been placed in routine CVC operation.

A carbon fiber feeder and controller for a CVC SiC reactor has been designed and fabricated but has not yet been tested. It is based on a design similar to an old-fashioned flour sifter: the goal is to produce a stream of individual fibers from an agglomerated mass. The agglomerated fibers are agitated; this agitation forces the individual fibers through a screen into the reactor. The amount of fibers allowed into the reactor can be controlled by controlling the degree of agitation and the size of the screen, and finally by using the particulate sensor.

The inlet plumbing for the CVC gas and solid reactants was modified. This was done because EDAX analysis of fracture origins indicated a small percentage of iron and vanadium present in the deposited CVC material (see the EDAX trace in Figure D). These impurities may be introduced into the CVC deposit because of abrasion of the SiC powder on the stainless steel tubing, and on the powder auger feeder drive, or because of

a chemical reaction between the reactant vapors and the hot inlet line. The first attempted solution was to purify the powder by chemical etching, but this did not reduce the amount of impurities in the CVC deposited material. Plumbing modification was the next solution to be considered.

As an alternative to the original half inch diameter, non-cooled stainless steel inlet manifold, a half inch diameter, water cooled inlet manifold has been constructed from molybdenum. The plumbing material between the reactor and the feeder itself has been changed from stainless steel (304SS) to copper (OFHC). Copper was chosen because either it will evaporate in the high heat of the reactor, or it will leave easily detectable traces.

The inlet plumbing has been further modified by the addition of several thumpers. These are solenoids (2 or 3 in number) connected to a timer, and attached at T's or elbows in the inlet plumbing. The timer causes the solenoids to "thump" every 1 or 2 seconds, loosening any powder clinging to the inside diameter of the plumbing, thus ensuring a more uniform powder flow into the reactor.

TASK--CVC Material:

The objective of this task is to fabricate SiC CVC material with powders or fibers that meets the program goals.

CVC materials were fabricated using various sizes and combinations of powder. The sensor described in the previous task was used in this process. Powder and gas reactants were mixed in a single half inch diameter manifold inlet. Both the stainless steel and the water cooled molybdenum inlets were tried. Material was deposited in a 20KW horizontal graphite tube reactor 4.5 inches in diameter by 24 inches long. The tube was heated by a 20KW inductive power supply. The reactor's hot zone produced CVC SiC material, which is characterized in the following task.

SiC material was fabricated using SiC powder ranging in size from 30 to 40 microns. SiC was also fabricated using multiple SiC powders of 10, 30, or 90 microns. Still other SiC materials were fabricated using platelets of 30 or 40 microns. Finally, CVC material was fabricated using diamond powder. Scanning electron microscope analysis of this material, however, showed a high incidence of voids, suggesting that the diamond powder may have evaporated during the process.

These results are summarized in Summary Table A.

TASK--Material Characterization:

The objective of this task is to analyze the microstructure of CVC materials to determine porosity, impurities, powder and fiber distribution, and fiber matrix interaction.

The micro structure of CVC SiC fabricated using single or multiple powder loading was characterized. CVC SiC was fabricated using 30 and 40 micron powder. MOR bend bars were machined and fractured.

The strength of this material was approximately 40-50 KPSI. This low strength was due to impurities in the material. Furthermore, bands of porosity were detected in the material, even though the sensor indicated that the powder stream was uniform. These defects led to the modification of the plumbing material, and the addition of the thumpers, which have been previously described.

The fabrication process has been repeated, using the same size powder, but also using the copper tubing, the water cooled molybdenum inlet manifold, and the thumpers. The material that has been produced in this batch will be analyzed in the next quarter.

TASK--Technology Transfer:

The objective of this task is to develop, fabricate, and test CVC SiC ceramic components for industrial use. In order to evaluate the effects of a high temperature fan in heat treating furnaces, data was assembled on the operation and performance of a typical

furnace using a metal fan. Then, certain parameters were varied in order to produce a temperature profile of heat-up times, thus determining a fan performance scaling. As a result, fan effect on furnace performance could be assessed.

It was established that the trays to be heat treated have low flow resistance. The flow volume is proportional to the product of the speed of the fan, the square of its diameter, and the height of the blade. Furthermore, increasing the volume of the flow appeared to be more effective than increasing the flow pressure (or velocity) in reducing heat treating times, making the process more energy efficient.

A graphite substrate for prototype fan fabrication has been designed and ordered. The material will be SiC CVC reinforced with 30 micron powder, to be deposited on a 1 inch shaft of SiC. The fans will be approximately 8 inches in diameter.

APPENDIX--GRI SiC Radiant Tube Development:

The objective of this task is to fabricate SiC tubes for vacuum heat treating. The tubes are to be 7.5 feet long, 6 inches in diameter, and 0.1 inch thick. They will have flanges on the open end; the other end will be domed.

The 100KW copper induction coil was modified to flatten the temperature profile along a 4 foot length for CVC deposits. The length of the 2 center coil sections was increased by 9 inches to reduce the midpoint temperature. The end coils were tightly bundled with Nextel insulation to increase the temperature at each end. The coil spacing in the middle section was uniform, and the coil helium leak was checked.

The 12 foot horizontal CVC chamber was repaired and modified to make the CVC SiC tube 7.5 feet long and 7 inches in diameter. The stainless steel water jacket was checked to locate any problems. Various welds were repaired on the water jacket, which was then successfully pressurized to 80 pounds with nitrogen overnight with no loss of pressure.

To date, the graphite tube substrate has been delivered, and the SiC powder feeder and sensor developed in Task 1 are ready for CVC deposit.

Figure a. Particulate Sensor Schematic

Figure b. Particulate Feeder Control System

FIGURE C: PHOTO OF SENSOR AND CONTROLLER IN OPERATION

FIGURE D. EDAX TRACE OF CVC SiC

TABLE A DOE RUN SUMMARY CVC SIC

run #	date	unit	powder	powder size	powder g/ltr	fiber	max dep. rate ml/hr	Purpose	density	uniformity
3455	10/26/94	H	Norton 7621 SIC	30 micron	4.5	Zolltek Panex		Fiber Composite	semi-porous	fibers in clumps
3459	11/1/94	H	N/A			Carbon		Fiber Composite	100%	some fiber clumps
3462	11/4/94	H	N/A			Zolltek Panex		Fiber composite	100%	Some fiber clumps
3465	11/11/94	H	N/A			Carbon		Baseline data for	100%	A few dendrites
3471	11/21/95	H	N/A					CVD run		
3473	12/8/94	H	Norton #7621 green SIC	30 micron	9.0	N/A		Baseline data for	100%	smooth and solid
3477	12/20/94	H	Norton #7621 green SIC	30 micron	9.0	Zolltek Panex		Fiber composite	100%	Fibers in clumps
3480	12/27/94	H	Norton #7621 green SIC	30 micron	9.0	Carbon		Fiber composite	100%	
3483	1/5/95	H	SIC platelet (Millenium Tech.)	40 micron	9.0	N/A		Bend bars	100%	Smooth and solid
3486	1/11/95	H	SIC platelet (Millenium Tech.)	30 micron	8.0	N/A		Bend Bars	100%	Smooth and solid
3488	1/13/95	H	SIC platelet (Millenium Tech.)	40 micron	8.0	N/A		Bend bars	100%	Smooth and solid
3507	2/22/95	H	Norton #7621 green SIC	30 micron	8.4	N/A		Bend bars	3.034 g/cc	Smooth and solid
3508	2/24/95	H	Norton #7621 green SIC	30 micron	9.0	N/A		Bend bars	2.979 g/cc	Smooth and solid
3511	3/3/95	H	SIC platelet (Millenium Tech.)	40 micron	5.0	N/A		Bend bars	3.117 g/cc	smooth and solid
3512	3/8/95	H	SIC platelet (Millenium Tech.)	40 micron	5.0	N/A		Bend bars	2.882 g/cc	Smooth and solid
3514	3/14/95	H	SIC platelet (Millenium Tech.)	40 micron	9.0	N/A		Bend bars	3.035 g/cc	Smooth and solid
3517	3/17/95	H	50% diamond + 50% Norton SIC	20 micron diamond + 30	9.0	N/A		Diamond Composite	100%	Smooth and solid
3519	3/22/95	H	Norton #7621 green SIC	30 micron	8.5	N/A		Equip test		
3521	3/24/95	H	Norton #7621 green SIC	1/3-17, 1/3-30 1/3-72 micron	8.3	N/A		Bend bars		Smooth and solid
3527	3/27/95	V	Norton #7621 green SIC	30 micron	8.9	N/A		Test BN, AL2O3, release agents		1 BN released, tube released from alumina core
3530	3/31/95	V	Norton #7621 green SIC	30 micron	18.5	N/A		Test BN release agent		All pcs stuck