T.D. Project Review

by Randy Lee

"Alternate SiC Bonding Methods"

Project Status & Overview

- Two pre-ceramic resins, 'Ceraset' and 'AHPCS' are being evaluated under various process conditions.

- Alternate approaches to reaction bonding are also being considered.

Bonding of VTR Assemblies

- Standard method gives strong, acid-resistant bonds but slows overall process.
- Objective is to develop a method to eliminate the second conversion run.
- Previous work investigated 'reaction bonding'. Furnace differences were noted.

General Methodology

- Vendor guidelines followed for sample processing but variations have been tested.
- Resins are mixed with SiC powder, cured to 125°-250° and fired to 1200°-1680° in SPD under Argon or Nitrogen.
- Heavy Duty furnace produces good bonds but SPD is the desired system for firing.

Properties of Ceraset & AHPCS

- Both handle like common resins but are converted to ceramic when fired.
- Ceraset contains Si, C and N and can yield Si_3N_4 and/or SiC relative to atmosphere.
- AHPCS contains Si and C but no N and should yield only SiC in any atmosphere.

Powder Types & Loading Levels

- Fillers provide thermal and mechanical stability as well as purity control.
- Several SiC powders have been evaluated as possible fillers.
- Powder loading trial indicated no differences relative to loading level.

Powder Loading Trial

Resin Type	Powder Type (SiC)	Loading Levels (%)	Mechanical Quality (4" drop)	EDX Results (% atomic)			
				Si	O	C	Fe
Ceraset	Norton	55 - 65	pass	55	3	42	0.2
Ceraset	T. Krull	55 - 65	fail	55	2	43	0
AHPCS	Norton	55 - 65	pass	54	2	44	0.2
AHPCS	T. Krull	55 - 65	fail	N/A			

Firing Conditions: In SPD to 1680°C @ 4°/min in 30 cfh N_2

Effects of Firing Temperature

- Dupont & Starfire recommend 1600°-1700° but SPD runs ≥1600° give poor bonds.
- 1200° runs always produce 'good' bonds but have high Oxygen levels.
- 'Good' bonds fired to 1200°-1400° degrade rapidly in HF acid.

Temperature Effects

Bonding Material	Firing Temperature (°C)	Mechanical Quality (6" drop)	Mechanical Quality (5' drop)	EDX Results (% atomic) Si	O	C	Fe
Ceraset / SiC	1200°	pass	pass	49	23	28	0.2
Ceraset / SiC	1300°	pass	pass	49	12	39	0
Ceraset / SiC	1400°	pass	pass	66	9	25	0
Ceraset / SiC	1500°	pass	pass	68	5	26	0
Ceraset / SiC	1680°	pass	fail	54	2	43	0
AHPCS / SiC	1200°	pass	pass	29	71	0	0
AHPCS / SiC	1300°	pass	pass	40	60	0	0.1
AHPCS / SiC	1400°	pass	pass	51	37	12	0
AHPCS / SiC	1500°	pass	pass	47	34	19	0.1
AHPCS / SiC	1680°	pass	fail	48	2	50	0

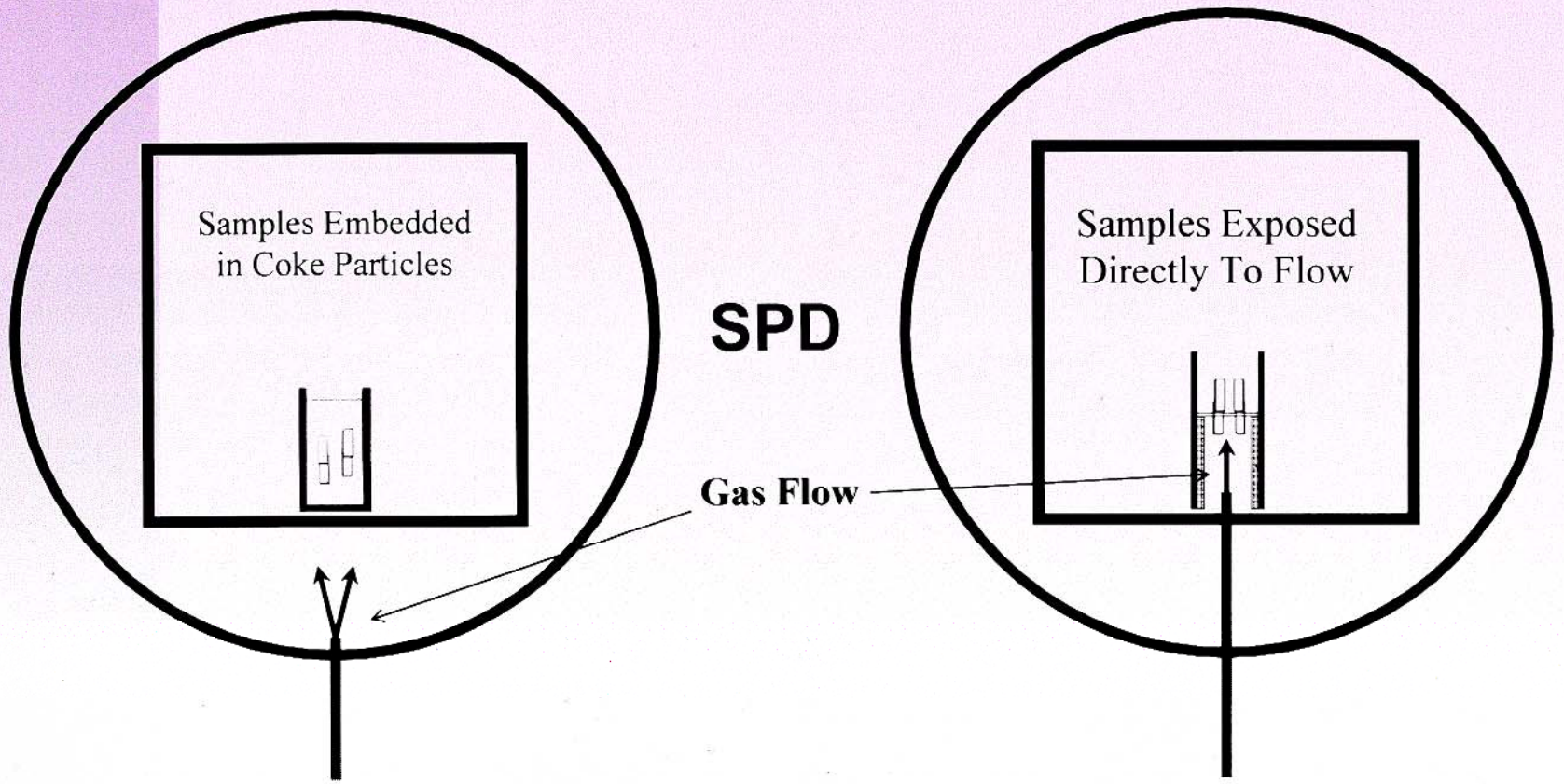
Firing Conditions: In SPD @ 4°/min to peak firing temperature in 30 cfh N_2

Effects of Gas Flow

- ★ Dupont & Starfire discount relevance of high flow during firing.
- ★ High flow in Heavy Duty may be crucial to strong bond formation.
- ★ 'No flow' vs. 'higher flow' runs indicate little difference in apparent bond strength.

No Flow Condition

Higher Flow Condition



Gas Flow Effects

Bonding Material	Flow Condition	Mechanical Quality (6" drop)	Mechanical Quality (1' drop)	EDX Results (% atomic) Si	O	C	Fe
Ceraset / SiC	**No Flow** Packed Coke	pass	pass	**58**	2	39	0
Ceraset only	**No Flow** Packed Coke	pass	fail	**58**	3	39	0
AHPCS / SiC	**No Flow** Packed Coke	pass	pass	**52**	2	46	0
AHPCS only	**No Flow** Packed Coke	pass	pass	53	3	45	0
Ceraset / SiC	**Higher Flow** 70 cfh	**pass**	**fail**	63	3	34	0
AHPCS / SiC	**Higher Flow** 70 cfh	**pass**	pass	69	2	28	0
Ceraset / SiC	**Typical Run** 30 cfh	pass	pass	52	2	46	0
AHPCS / SiC	**Typical Run** 30 cfh	pass	pass	47	2	51	0

Firing Conditions: In SPD @ 4°/min to 1680° in 30 cfh Ar

Future Goals & Plans

- ✓ Identify source or cause for high Oxygen content in SPD fired samples (during cure or during firing?).
- ✓ Extend data base to include weight loss, mechanical tests and other characterization methods (XRD).
- ✓ Continue working with Dupont and Starfire regarding processing problems in SPD.
- ✓ Determine possible correlations between post-fire bond composition and peak firing temperature.
- ✓ Identify unique attributes or conditions in Heavy Duty and adapt to SPD if possible.
- ✓ Investigate other joining concepts including reaction bonding techniques.