**PRB-CERAMIC MONOCOMPOSITE AND CERAMIC COATINGS BY
SOL-GEL-PYROLYSIS AND SLURRY-PYROLYSIS PROCESSING**

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**RECEIVED
SEP 07 1999
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JANUARY 29, 1997

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OUTLINE OF PRESENTATION

- 1. COATING SYSTEMS**
- 2. STARTING MATERIALS**
- 3. PROPERTIES OF COATINGS**

COATING SYSTEMS

BINARY PHASES:

- 1) $\text{TiO}_2\text{-SiO}_2$
- 2) $\text{ZrO}_2\text{-SiO}_2$
- 3) $\text{ZrO}_2\text{-TiO}_2$
- 4) $\text{Y}_2\text{O}_3\text{-ZrO}_2$

TERNARY PHASES:

- 5) $\text{P}_2\text{O}_5\text{-TiO}_2\text{-K}_2\text{O}$
- 6) $\text{LiO}_2\text{-Al}_2\text{O}_3\text{-SiO}_2$

COATING SYSTEMS

SOL-GEL PRECURSORS

SINGLE, BINARY AND TERNARY PYROGENIC PHASES

- POLYSILAZANE —→ SILICON NITRIDE
- POLYBORAZINLYAMINE —→ BORON NITRIDE
- TI AND SI ALKOXIDE BLEND —→ TiO_2 - SiO_2 SYSTEMS
- ZR AND SI ALKOXIDE BLEND —→ ZrO_2 - SiO_2 SYSTEMS
- ZR AND TI ALKOXIDE BLEND —→ ZrO_2 - TiO_2 SYSTEMS
- AL AND SI ALKOXIDE BLEND —→ SiO_2 + MULLITE SYSTEMS
- Y AND ZR ALKOXIDE BLEND —→ YITTRIA-STABILIZED ZIRCONIA
- P, K, AND TI ALKOXIDE BLEND —→ P_2O_5 - TiO_2 - K_2O SYSTEMS

STARTING MATERIALS

- ZIRCONIUM-N-PROPOXIDE (ZNP) 70% IN PROPANOL

- YTTRIUM ACETATE (YA) 99.9%

- ALUMINUM SEC-BUTOXIDE (ASB) 95%

- DIETHANOLAMINE (DEA)

- ISOPROPYL ALCOHOL (IPA)

- HYDROCHLORIC ACID

- WATER

BASIC FORMULATION

ZrO₂

<u>ZNP</u>	<u>DEA</u>	<u>HCL</u>	<u>WATER</u>	<u>IPA</u>
29.4%	44.1%	2.9%	8.8%	14.7%
PH 9.45				

Al₂O₃

<u>ASB</u>	<u>DEA</u>	<u>HCL</u>	<u>WATER</u>	<u>IPA</u>
32.2%	48.4%	3.2%	-	16.1%
PH 9.09				

Y₂O₃

<u>YA</u>	<u>DEA</u>	<u>HCL</u>	<u>WATER</u>	<u>IPA</u>
12.6%	68.4%	3.2%	-	15.8%
PH 9.30				

Table 1

<u>Panel No.</u>	<u>Coating</u>	<u>Weight of uncoated panel, g</u>	<u>Total weight of coated panel, g</u>	<u>Weight of coating, g</u>
Zr-1	100% ZrO_2	27.7967	27.8036	0.0069
Zr-2	100% ZrO_2	26.5529	26.5600	0.0071
Al-1	100% Al_2O_3	28.1437	28.1492	0.0055
Al-2	100% Al_2O_3	26.7626	26.7676	0.0050
YZr-1-1	5% Y_2O_3 -95% ZrO_2	26.5121	26.5305	0.0184
YZr-1-2	5% Y_2O_3 -95% ZrO_2	26.5243	26.5427	0.0184
YZr-2-1	10% Y_2O_3 -90% ZrO_2	34.7744	34.7860	0.0116
YZr-2-2	10% Y_2O_3 -90% ZrO_2	26.8056	26.8169	0.0113
Y_2O_3 -0-1*	100% Y_2O_3	28.0463	28.0587	0.0124
Y_2O_3 -0-2	100% Y_2O_3	26.9981	27.0099	0.0118
Y_2O_3 -1-1**	100% Y_2O_3	27.5683	27.5773	0.0090
Y_2O_3 -1-2	100% Y_2O_3	26.5180	26.5275	0.0095
Y_2O_3 -2-1***	100% Y_2O_3	26.2032	26.2072	0.0040
Y_2O_3 -2-2	100% Y_2O_3	26.8739	26.8778	0.0039

* derived from 60 wt% Y_2O_3 solution

** derived from 40 wt% Y_2O_3 solution

*** derived from 20 wt% Y_2O_3 solution

SLURRY COATINGS

<u>LIQUID REACTANT</u>	<u>SOLID REACTANT</u>	<u>PYROGENIC MAJOR PHASE</u>
$-[-\text{MPO}_3-]_n$	$\text{CaO} \cdot \text{Al}_2\text{O}_3$	$\rightarrow \text{CaO} \cdot \text{P}_2\text{O}_5$
$-[-\text{MPO}_3-]_n$	$\text{CaO} \cdot \text{SiO}_2$	$\rightarrow$
$-[-\text{MPO}_3-]_n$	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	$\rightarrow \text{M}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$
$-[-\text{MPO}_3-]_n$	$\text{MgO} \cdot \text{Al}_2\text{O}_3$	$\rightarrow$
$-[-\text{M}_2\text{O} \cdot \text{SiO}_2-]_n$	$\text{CaO} \cdot \text{Al}_2\text{O}_3$	$\rightarrow$
$-[-\text{M}_2\text{O} \cdot \text{SiO}_2-]_n$	$\text{CaO} \cdot \text{SiO}_2$	$\rightarrow$
$-[-\text{M}_2\text{O} \cdot \text{SiO}_2-]_n$	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	$\rightarrow$
$-[-\text{M}_2\text{O} \cdot \text{SiO}_2-]_n$	$\text{MgO} \cdot \text{Al}_2\text{O}_3$	$\rightarrow$

M: NH_4^+ , Na^+ , K^+ , OR Li^+

	Type III Cement	Class C Fly Ash	Meta- Kaolin	GGBF Slag	Class F Fly Ash	Silica Fume
SiO ₂	20.20	39.50	59.91	35.60	53.00	95.86
Al ₂ O ₃	2.32	16.90	39.68	10.40	18.60	0.36
TiO ₂	0.11		0.54			0.05
Fe ₂ O ₃	4.47	6.40	0.93	1.16	15.60	0.05
MgO	3.54	6.30		12.84	1.26	0.25
MnO	0.05					0.03
CaO	63.90	24.80	0.32	37.59	6.37	0.31
Na ₂ O	0.12	1.44		0.07	0.94	0.16
K ₂ O	0.49	0.53	1.20	0.33	2.08	0.68
P ₂ O ₅	0.21					0.09
SO ₃	3.00	1.99			2.02	0.10
S				0.67		
LOI	0.85	0.22	0.23	1.00*	1.68	0.80

GGBF - Ground granulated blast furnace

* - note - gain on ignition indicates the presence of sulfur as S rather than SO₂

**GLASS CERAMIC COATINGS FROM FLY ASH
AS INDUSTRIAL WASTE**

**FORMULATION: 60 WT% FLY ASH F - 16 WT% $[-\text{NaPO}_3-]_n$ -
24 WT% WATER**

DIPPING PANELS INTO SLURRY AT 25°C

WITHDRAWING

PREHEAT IN AIR OVEN FOR 5 HRS AT 200°C

ANNEALING FOR 12 HRS AT 900°C

Sample: FA0200
Size: 31.6010 mg
Method: TGA-900-15

TGA

File: C:NC.042
Operator: NC
Run Date: 8-Jan-97 09:15

XPS Atomic Fraction of Fly Ash-Derived Coating (FA)/Ti Interfaces

<u>Specimen</u>	<u>Failure side</u>	Atomic composition, %							
		<u>Al</u>	<u>Si</u>	<u>P</u>	<u>C</u>	<u>Ti</u>	<u>O</u>	<u>Na</u>	<u>Fe</u>
25°C Ti	-	9.88	12.78	-	23.85	6.67	46.82	-	-
900°C Ti	-	25.51	4.72	-	7.82	6.92	55.04	-	-
900°C FA/Ti	-	4.72	12.20	1.20	20.24	3.30	48.20	4.13	6.01
900°C FA/Ti	FA	16.56	6.12	12.49	5.71	6.15	50.79	2.18	-
900°C FA/Ti	Ti	10.49	2.72	9.24	13.51	12.17	48.37	3.50	-

TASKS

- 1) SYNTHESIS AND PROPERTIES OF COLLOIDAL METAL-ORGANIC PRECURSOR SOLUTIONS (BNL)**
- 2) SURFACE PREPARATION OF SUBSTRATES (P&W AND BNL)**
- 3) WETTING AND SPREADING BEHAVIORS OF PRECURSORS OVER PRE-TREATED SUBSTRATE SURFACES (BNL)**
- 4) SCREENING OF PRECURSORS (BNL)**
- 5) FILM-FORMING PERFORMANCE OF PRECURSORS ON SUBSTRATE SURFACES AT TEMPERATURES IN THE RANGE 400 TO 1200°C (BNL)**
- 6) CORROSION, OXIDATION, AND WEAR RESISTANCE TESTS (P&W)**
- 7) SCREENING OF COATING SYSTEMS, AND PATENT APPLICATIONS (BNL AND P&W)**

POLARIZATION CURVES FOR Y_2O_3 -COATED STAINLESS STEELS

COATING	TREATMENT TEMPERATURE OF Y_2O_3 PRECURSOR, °C	CORROSION RATE MILLI-INCHES/YEAR
STAINLESS STEEL (SS)		5.17×10^{-1}
Y_2O_3 /SS	300	1.80×10^{-1}
Y_2O_3 /SS	500	2.17×10^{-2}
Y_2O_3 /SS	900	1.08×10^{-2}