Low Conductive Thermal Barrier Coatings Produced by Ion beam Assisted EB-PVD with Controlled Porosity, Microstructure Refinement and Alloying Additions for High Temperature Applications

by

Douglas E. Wolfe and Jogender Singh
Applied Research Laboratory, Penn State University
University Park, PA 16801

Final Report
NASA-GRC Cooperative Agreement NCC3-767

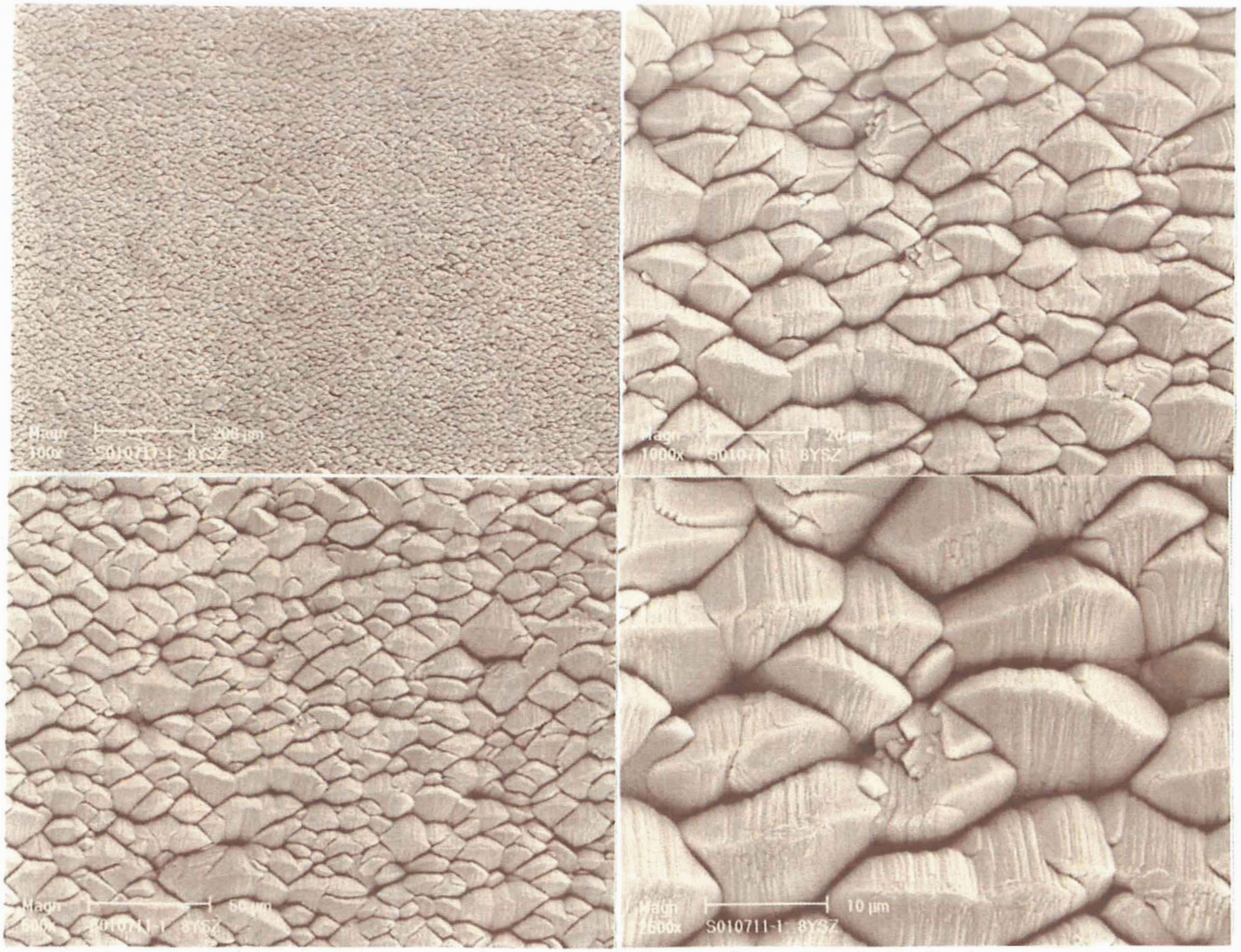
January 20th, 2005

Sample description and coating thickness for various Hafnia-coated samples (NASA)

Sample Number	TBC	Number of layers	TBC
S010615-1	HfO_2-$27Y_2O_3$	1	138.7 +/- 1.829
S010618-1	HfO_2-$27Y_2O_3$	2	181.9 +/- 1.101
S010619-1	HfO_2-$27Y_2O_3$	5	201.3 +/- 1.889
S010622-1	HfO_2-$27Y_2O_3$	10	173.9 +/- 1.595
S010625-1	HfO_2-$27Y_2O_3$	20	222.4 +/- 1.713
S010626-1	HfO_2-$27Y_2O_3$	1	208.2 +/- 2.201
S010627-1	HfO_2-$27Y_2O_3$	40	219.5 +/- 2.224
S010802-1I	HfO_2-$27Y_2O_3$	1	191.1 +/-1.370
S010803-1E	HfO_2-$27Y_2O_3$	40	194.4 +/-1.838

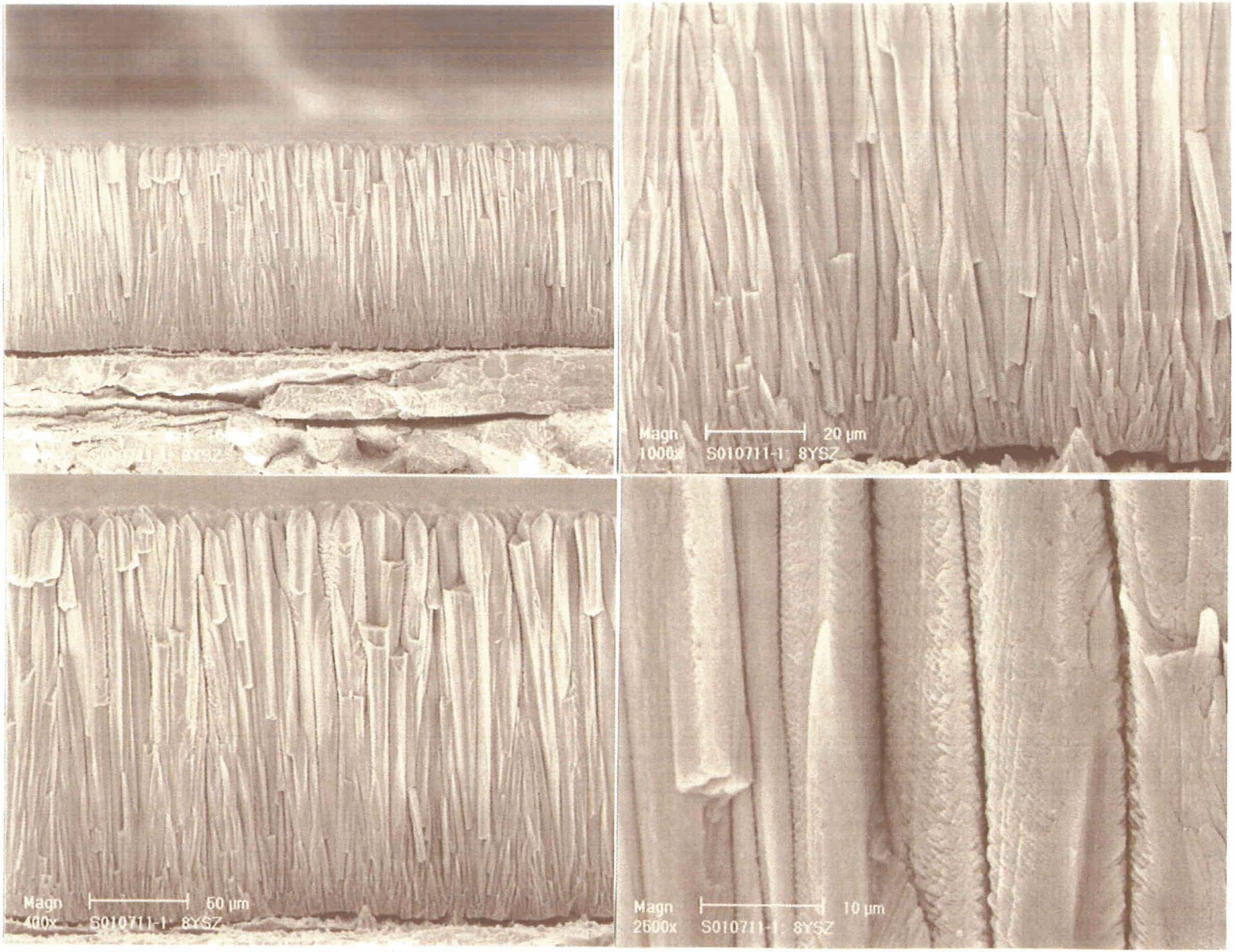
Scanning Electron Microscopy

Deposited at 1000 ^{0}C, 1- layer



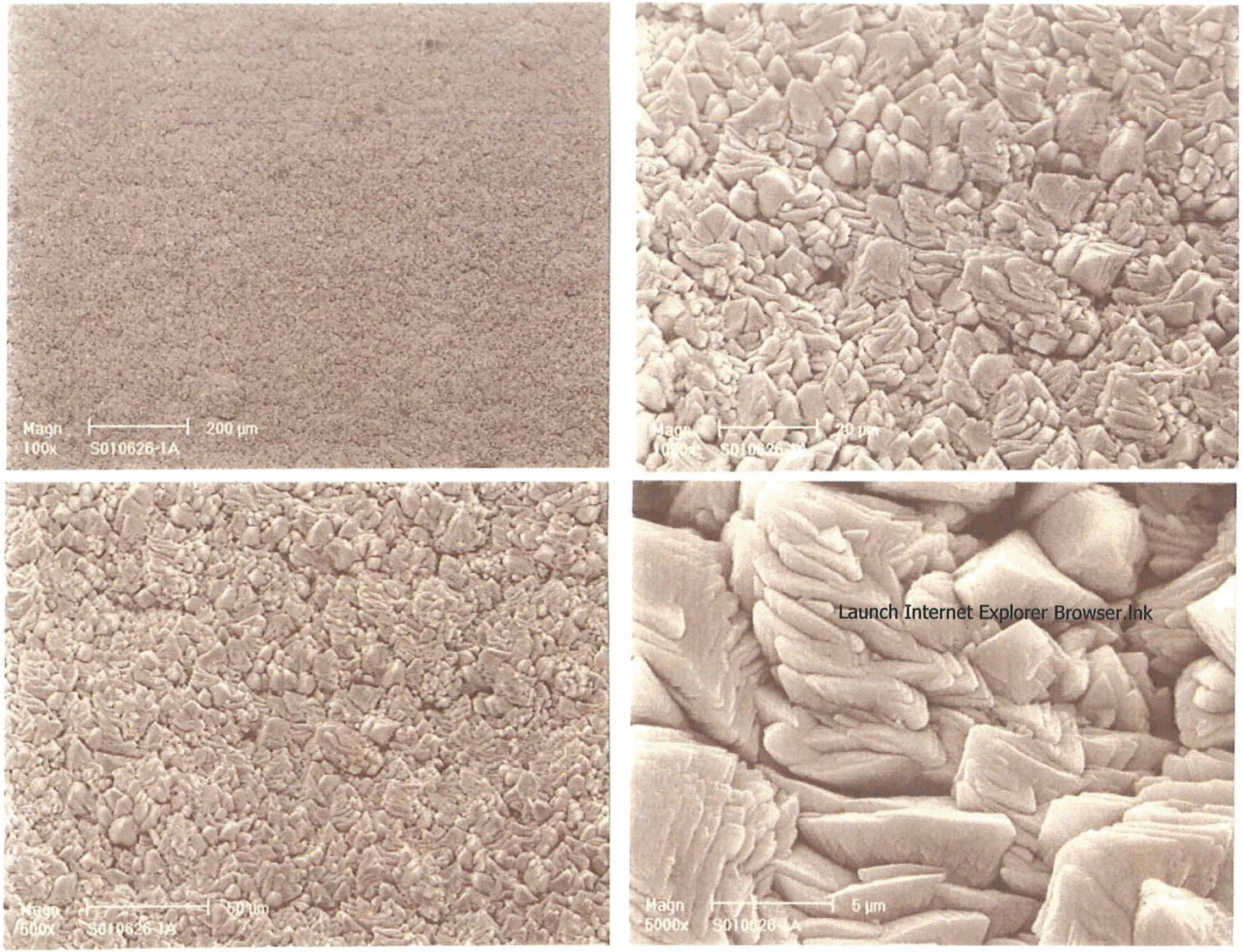
SEM micrographs showing the surface morphology of standard 8YSZ deposited on PtAl-coated MAR-M-247. Sample #S010711-1.

Deposited at 1000 ^{0}C, 1- layer

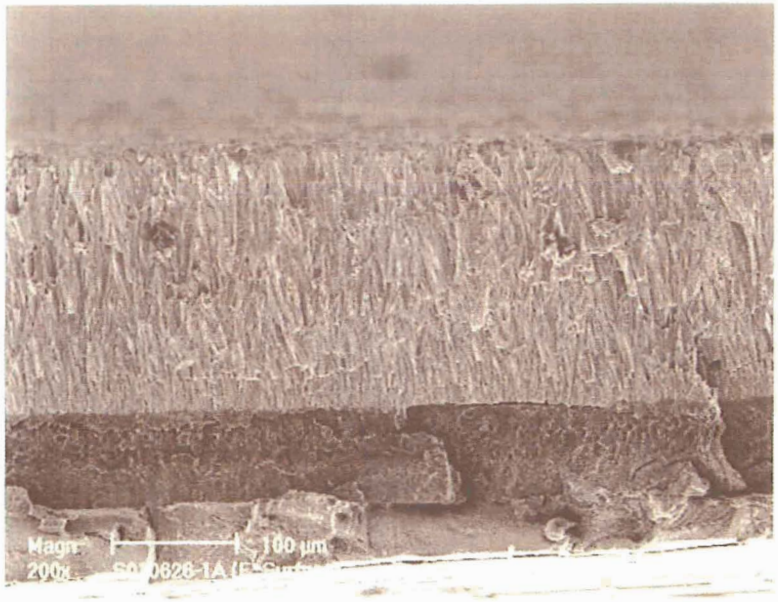


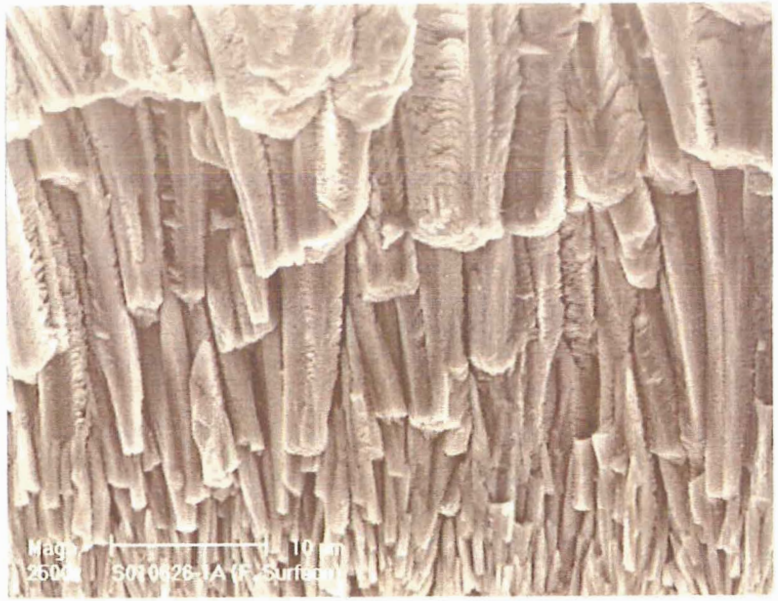
SEM micrographs showing the fracture surface of standard 8YSZ deposited on PtAl-coated MAR-M 247 Sample #S0 07

Deposited at 1000 ^{0}C, 1- layer



SEM micrographs showing the surface morphology of HfO_2 – 27wt% Y_20_3 (PCR-1 layer) deposited on platinum aluminide-coated MAR-M-247. Sample # S010626-1A

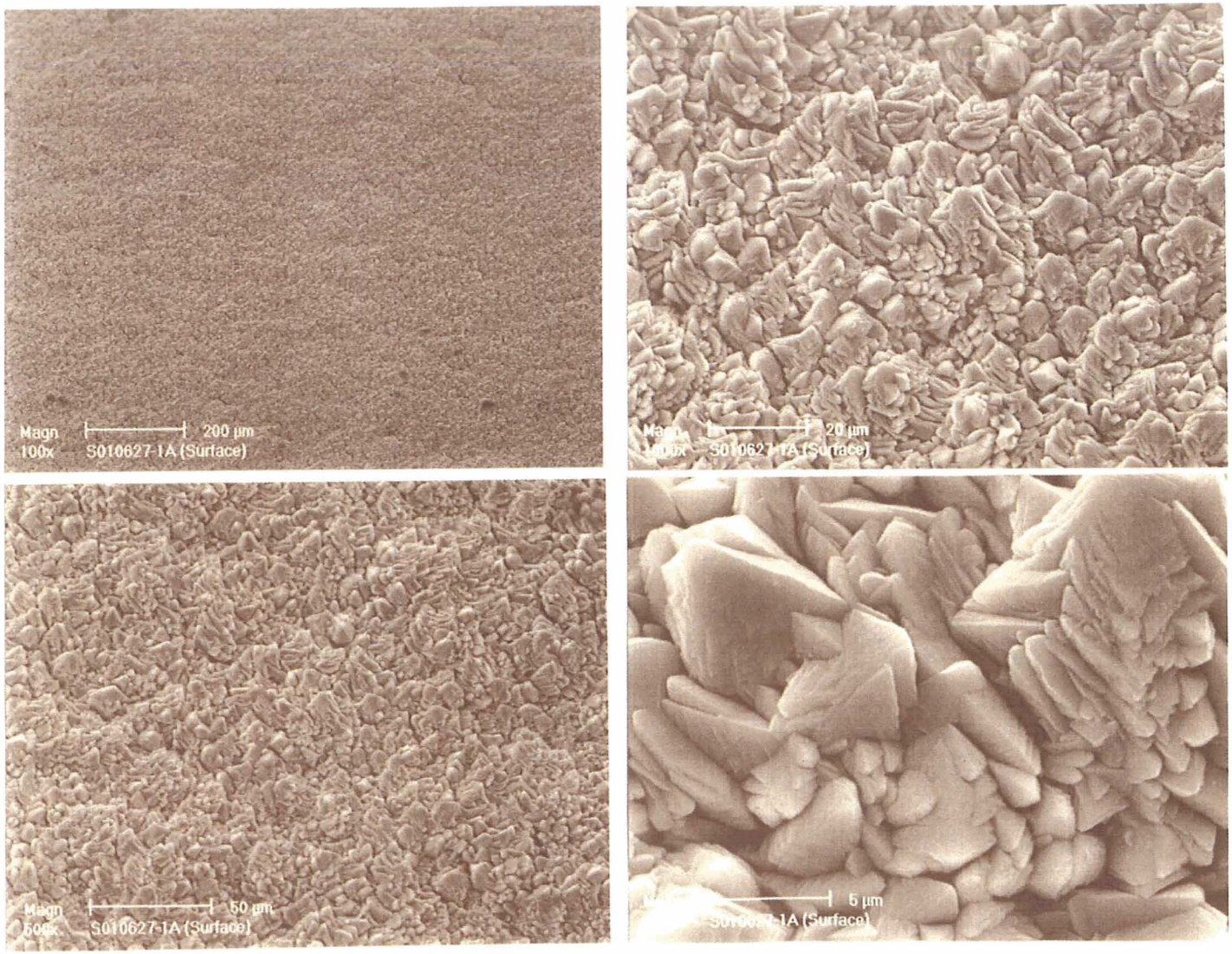




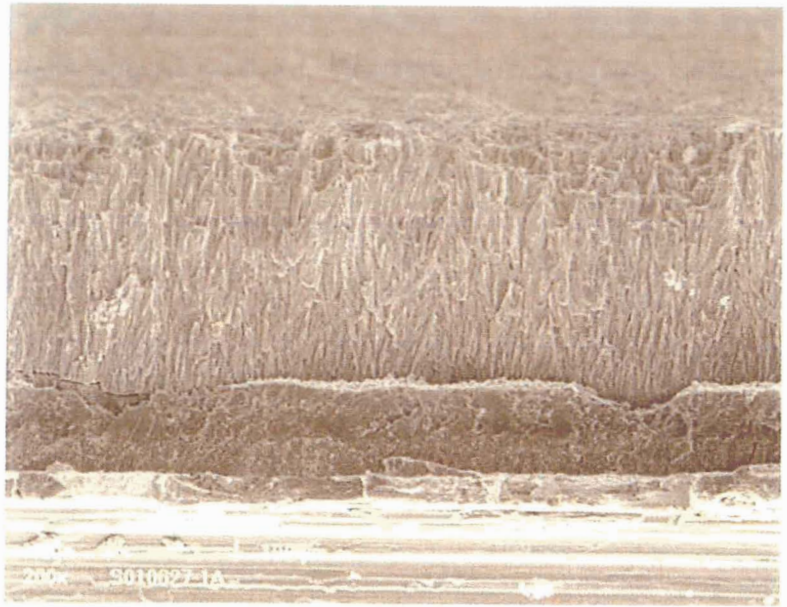
Deposited at 1000 ^{0}C
1- layer

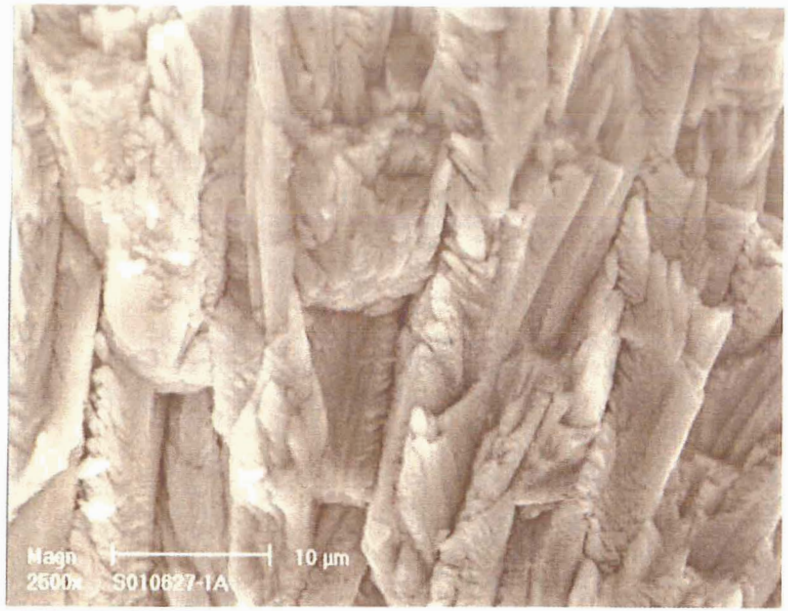
SEM micrographs showing the cross section of HfO_2 – 27wt%Y_20_3 (PCR-1 layer) deposited on platinum aluminide-coated MAR-M-247. Sample # S010626-1A

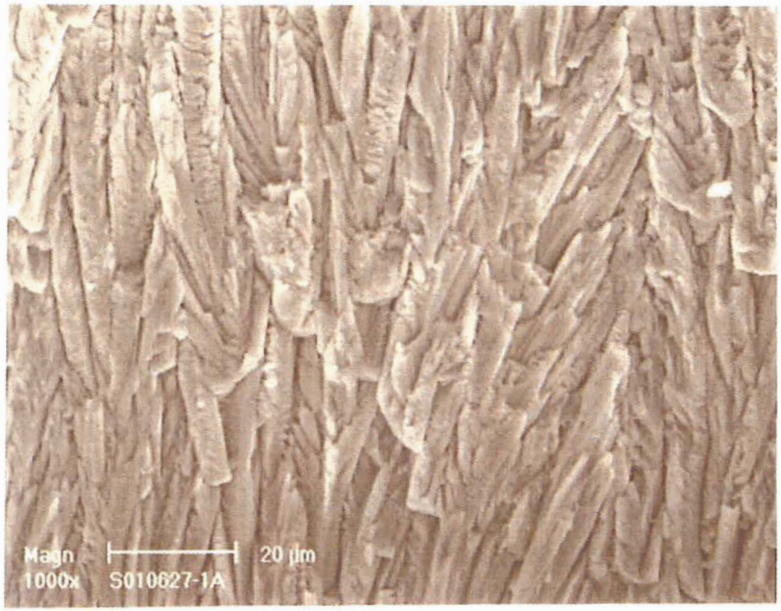
Deposited at 1000 ^{0}C, 40- layers



SEM micrographs showing the surface morphology of HfO_2 – 27wt% Y_2O_3 (PCR 40 layer) deposited on platinum aluminide-coated MAR-M-247. Sample # S010627-1A



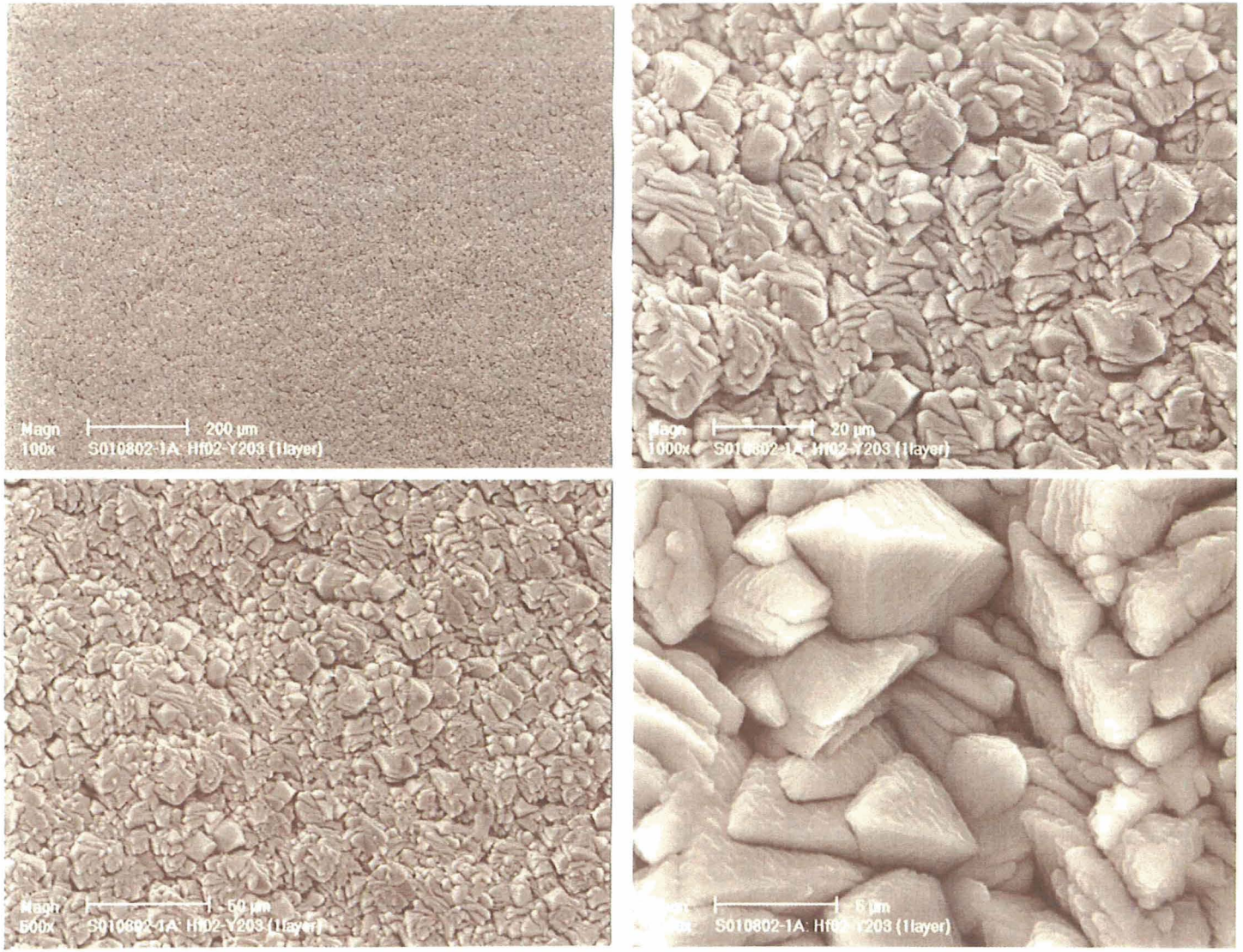




Deposited at 1000 ^{0}C
40 layers

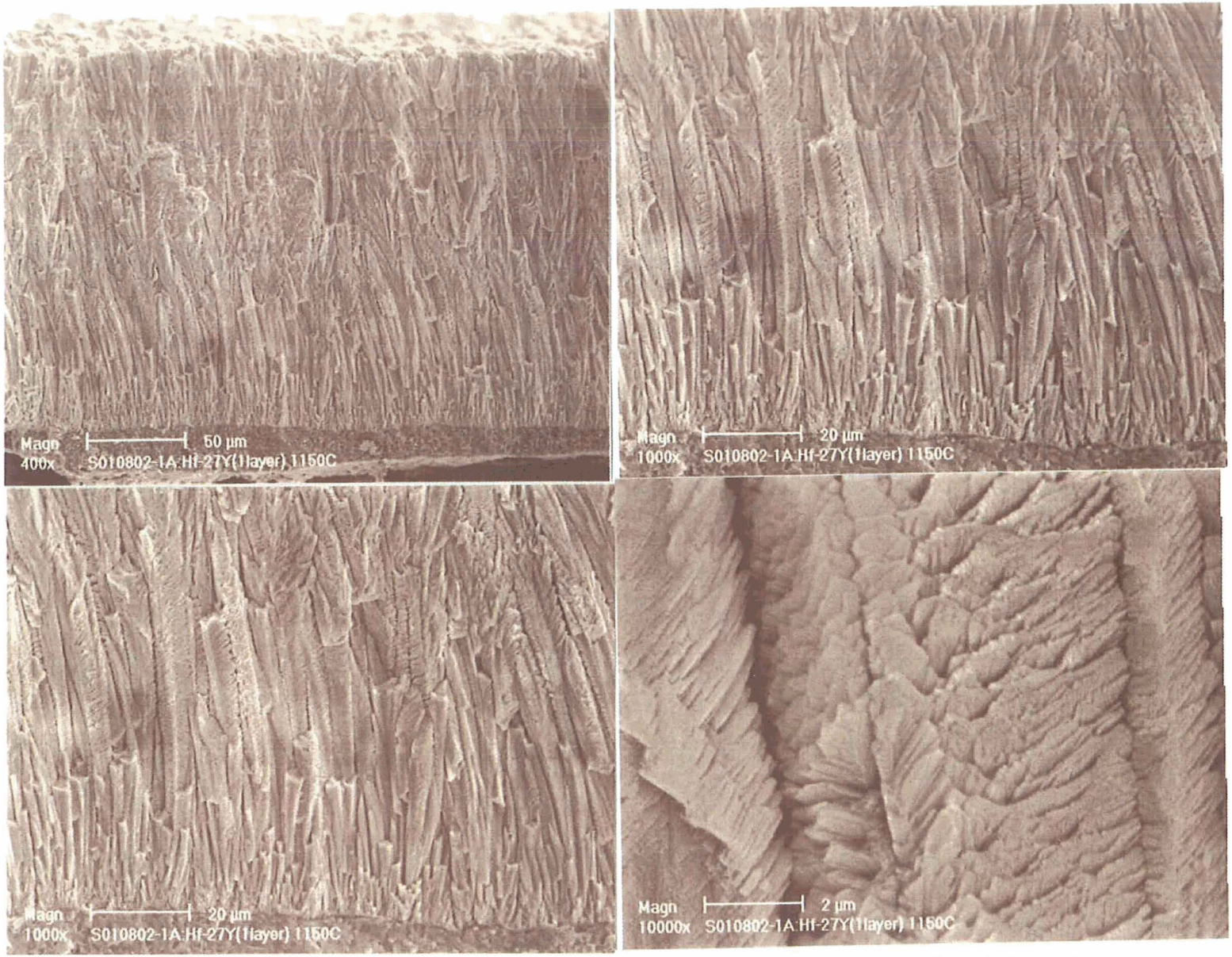
SEM micrographs showing the cross section of HfO_2 – 27 wt% Y_20_3 (PCR – 40 layer) deposited on platinum aluminide-coated MAR-M-247 Sample # S010627-1A

Deposited at 1150 ^{0}C, 1- layer



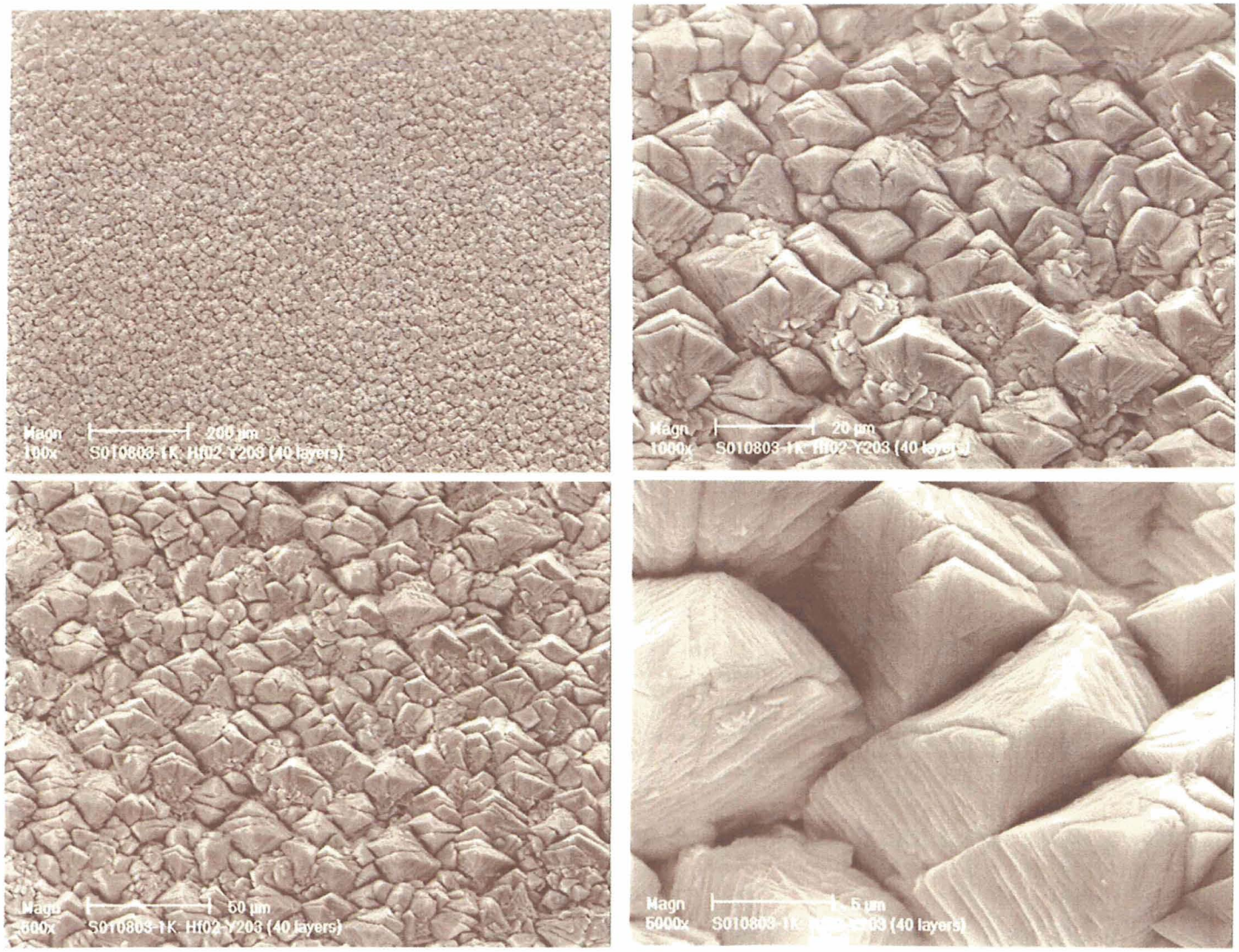
SEM micrographs showing the surface morphology of HfO_2 – 27 wt% Y_2O_3 (PCR - 1 layer) deposited on platinum aluminide - coated MAR-M-247. Sample # S010802-1A

Deposited at 1150 ^{0}C, 1- layer



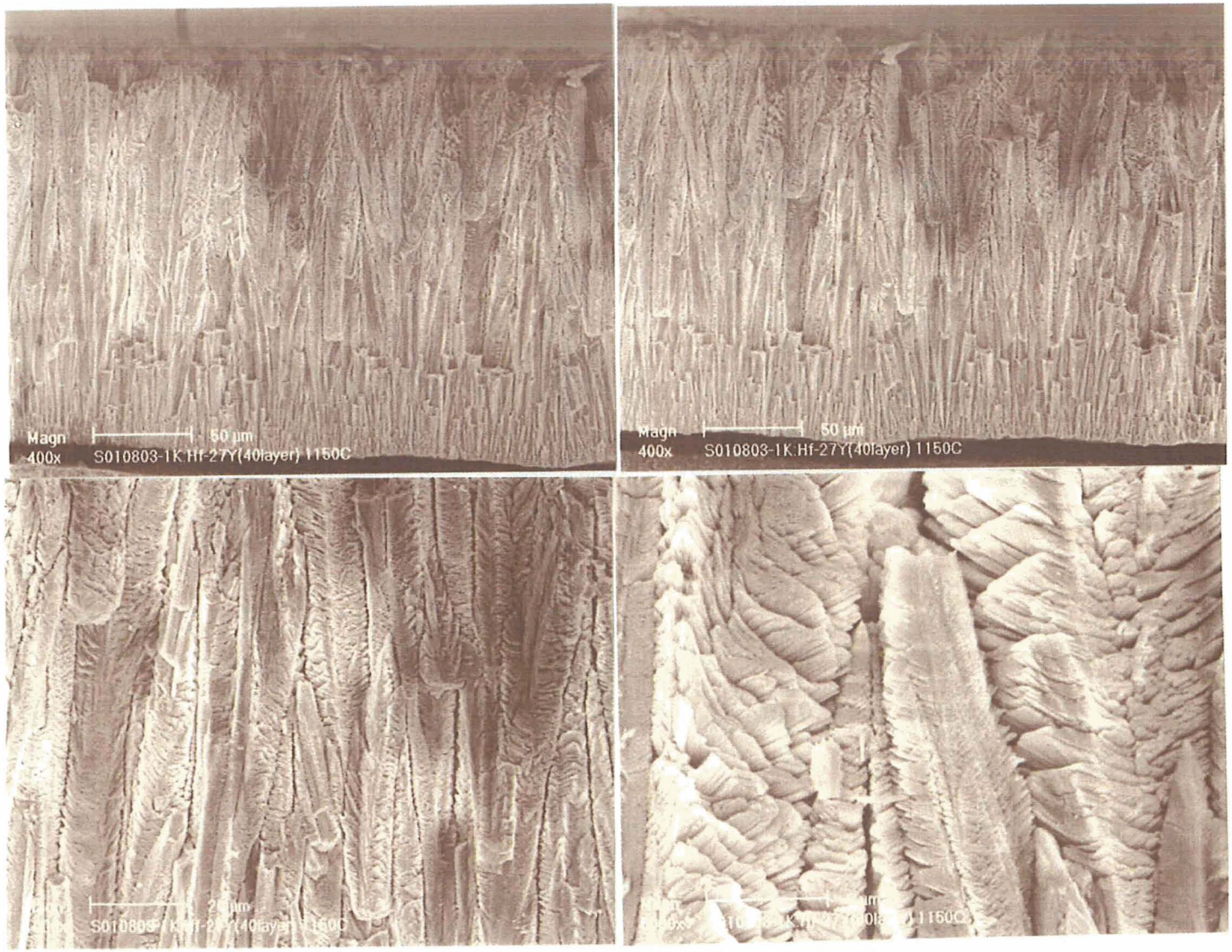
SEM micrographs showing the fracture surface of HfO_2-27wt.%Y_2O_3 (1-layer) deposited on PtAl-coated MAR-M-247 by EB-PVD (1150°C). Sample #S010802-1A

Deposited at 1150 ^{0}C, 40- layers



SEM micrographs showing the surface morphology of HfO_2 – 27 wt% Y_2O_3 (PCR - 40 layers) deposited on platinum aluminide - coated MAR-M-247. Sample # S010803-1K

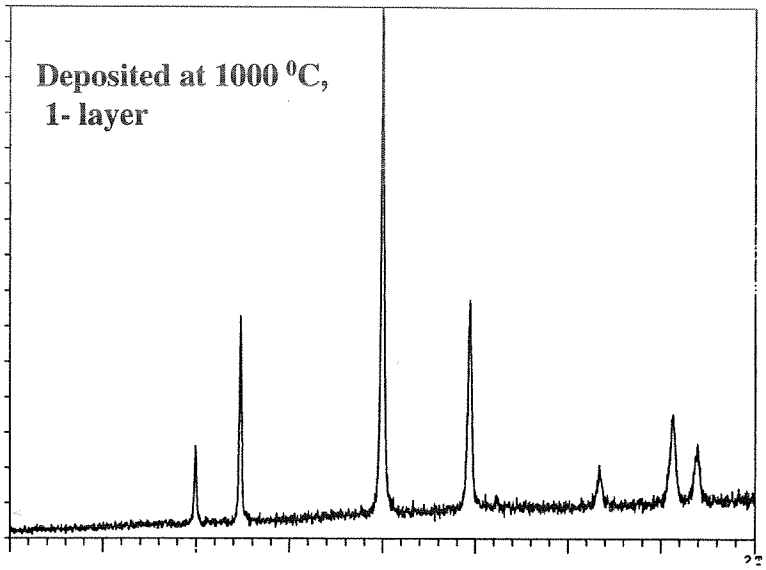
Deposited at 1150 ^{0}C, 40- layers



SEM micrographs showing the fracture surface of HfO_2-27wt.%Y_2O_3 (40-layer) deposited on PtAl-coated MAR-M-247 by EB-PVD (1150^{o}C). Sample #S010803-1K.

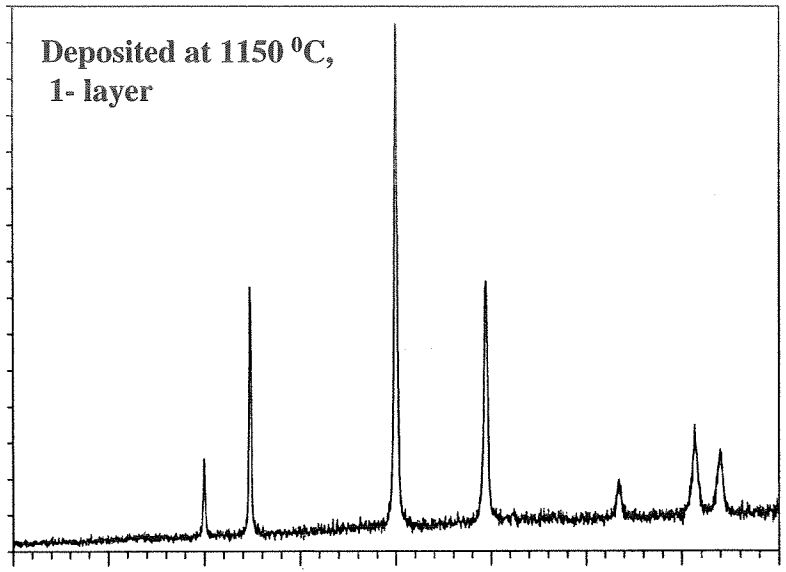
X-ray Diffraction

S010626-1E: HfO2-27wt.%Y2O3(1-layer)/PtAl/RENE-N5 3-jul-1999



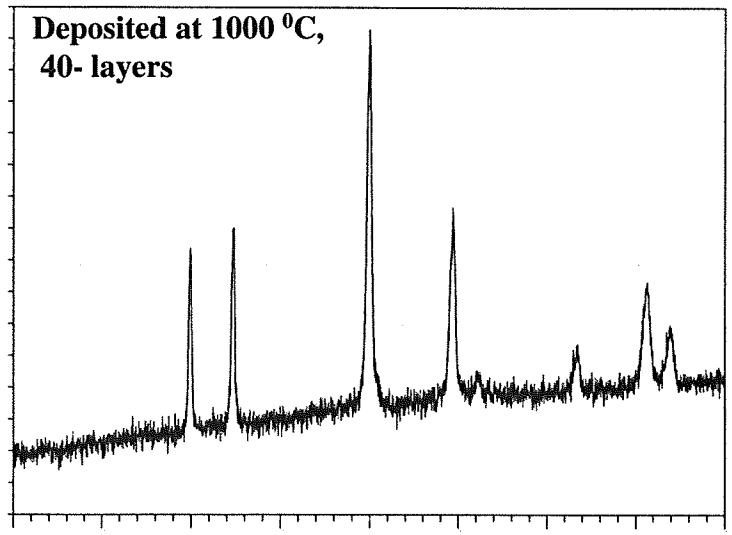
0010626H.SD

S010802-1L: HfO2-2 wt %Y2O3 1layer /PtAl/Rene-N5 6-aug-1999 14 06



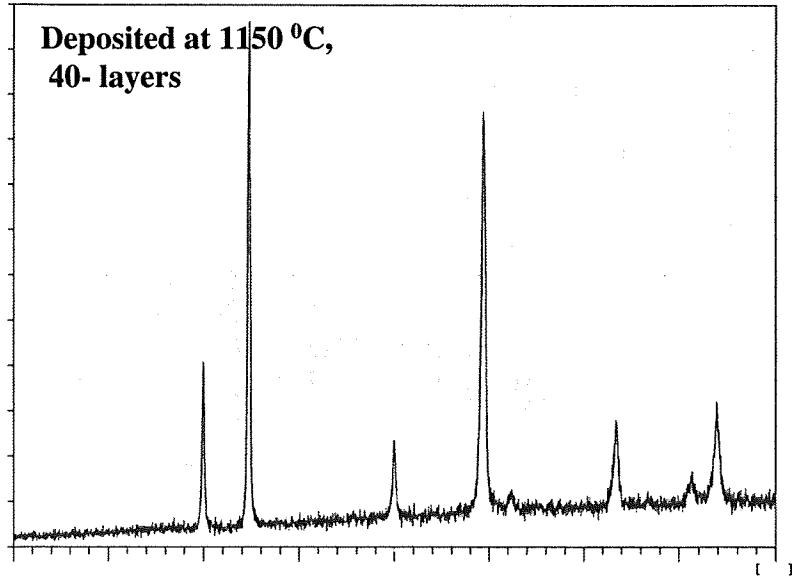
0010802L.SD

S01062 1H:HfO2-27wt.%Y2O3 40layer)/PtAl/RENE-N5 3 jul-1999 10:29



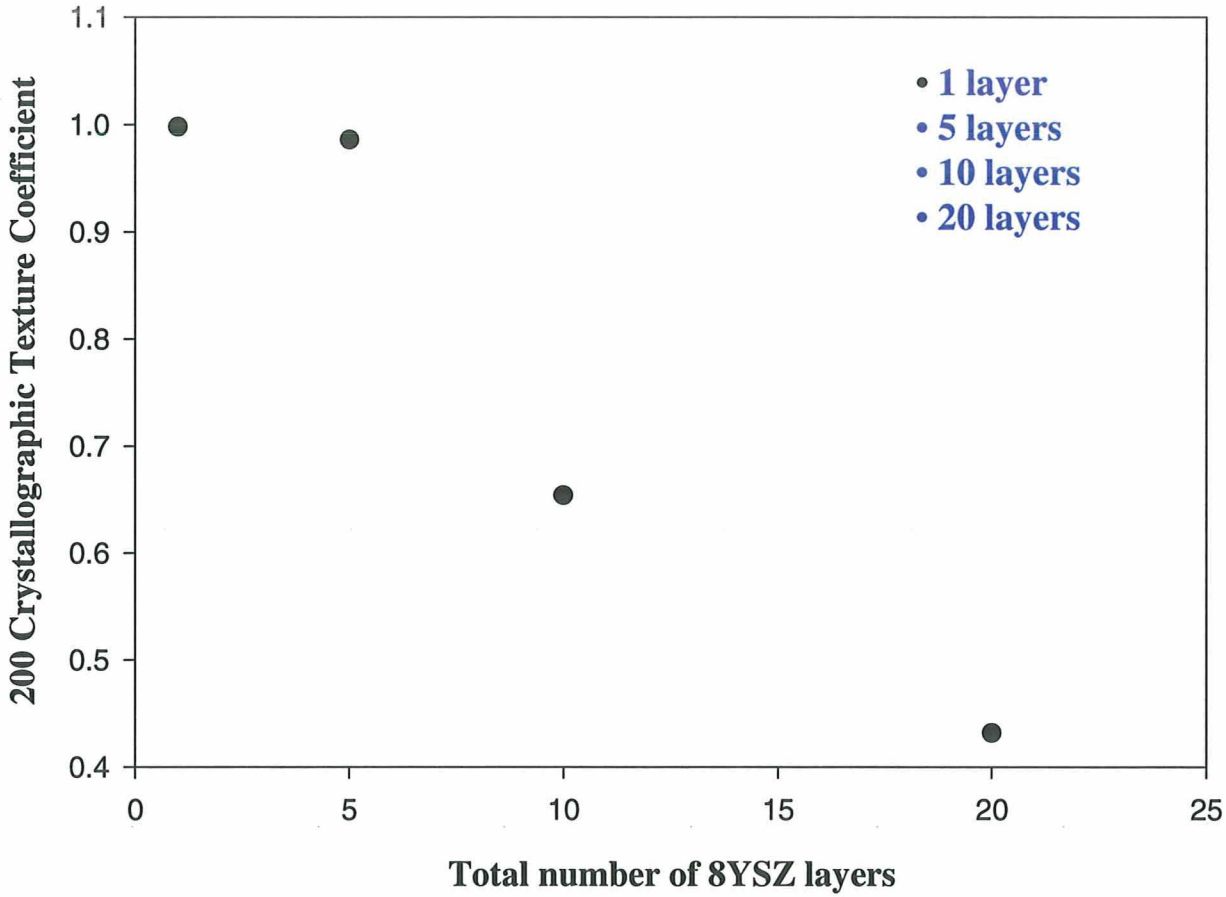
SD10627H.SD

S010803-1D: HfO2-27wt.%Y2O3(40-layer)/PtAl/ReneN5 6-aug-1999



0010803D.SD

X-ray texture coefficient for 8YSZ layered structure



200 Texture Coefficient as a function of total number of layers for 8YSZ deposited on CoNiCrAlY-coated MAR-M-247

Preliminary Strain Evaluation

Strain tolerance experiments on layered structure

- **5 layers**
- **10 layers**
- **20 layers**

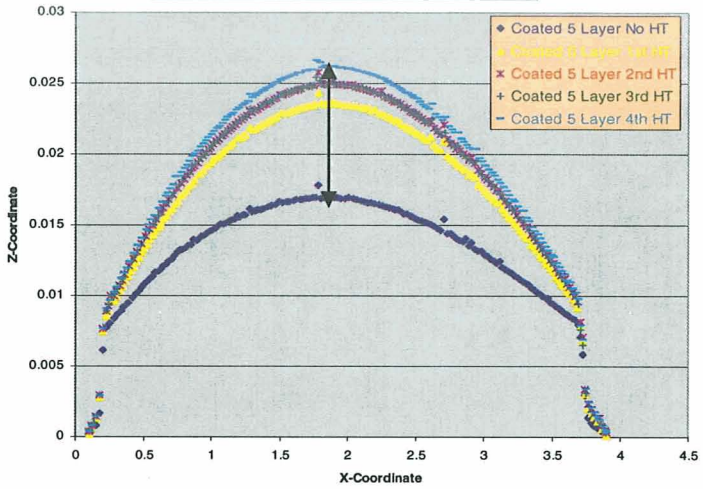
- **Experimental condition: Coated strip annealed at 900 ^{0}C every 10 hours in argon atmosphere and measured bending**

Conclusion:

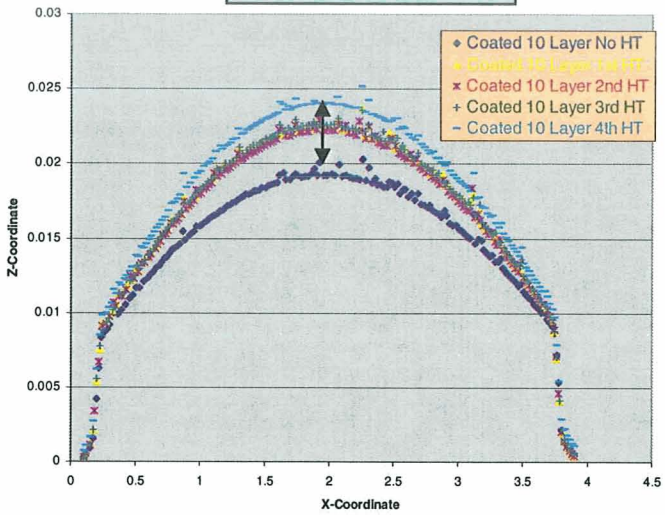
- **Less bending as a function of increasing layers**

Deflection in Strip (4"X 0.5"X 0.04") as Function of 8YSZ Layers After 900^{0}C Exposure

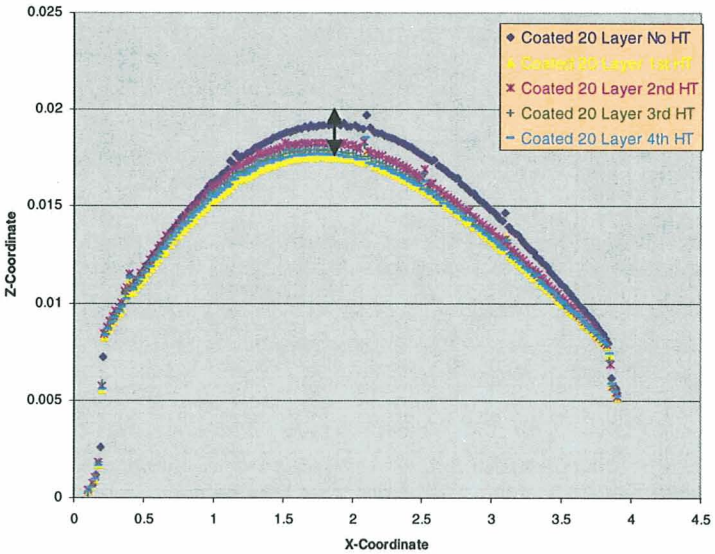
Five layers

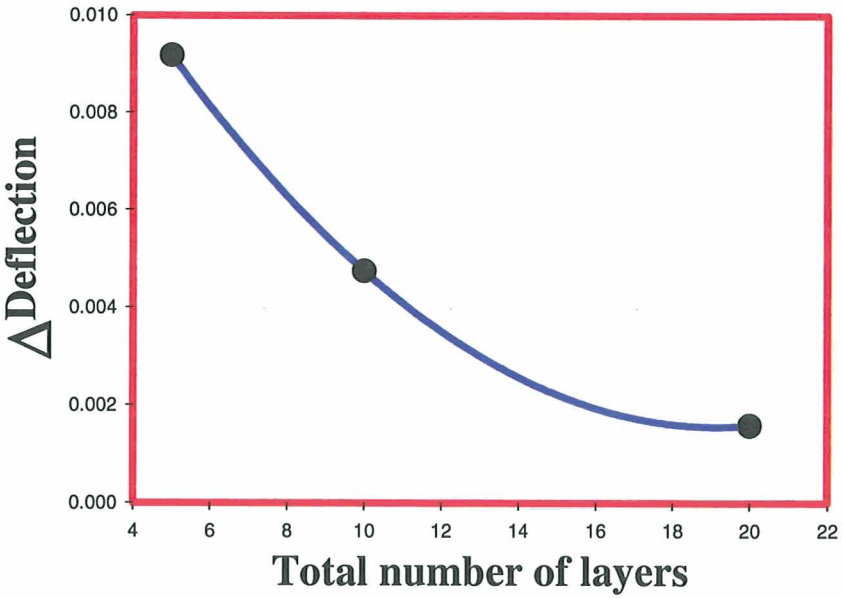


Ten layers

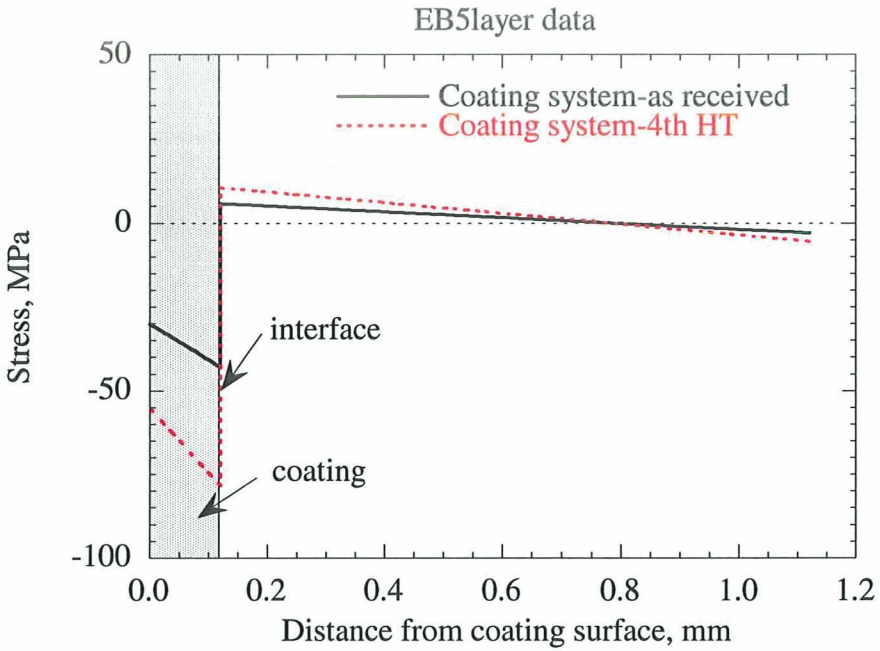


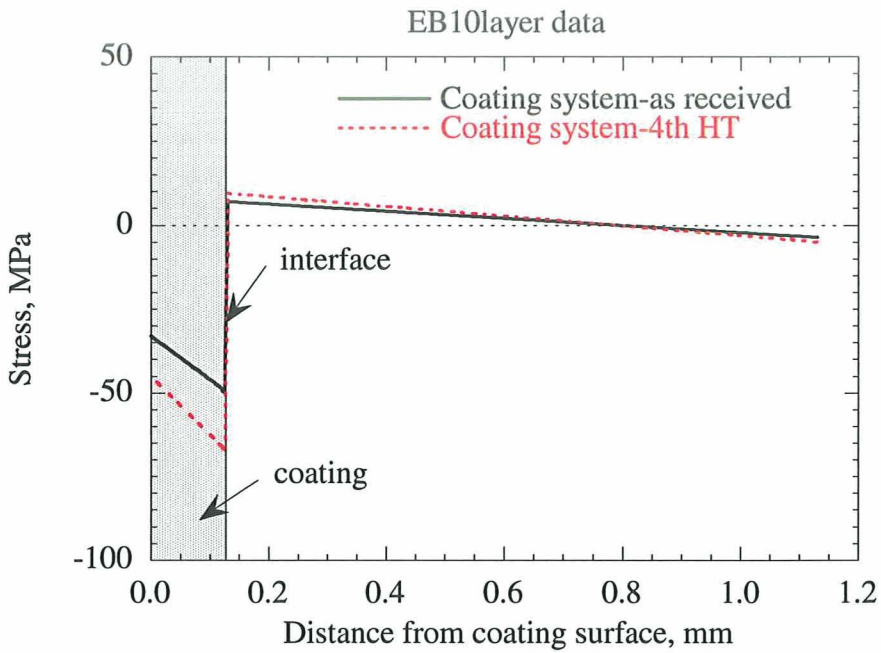
Twenty layers

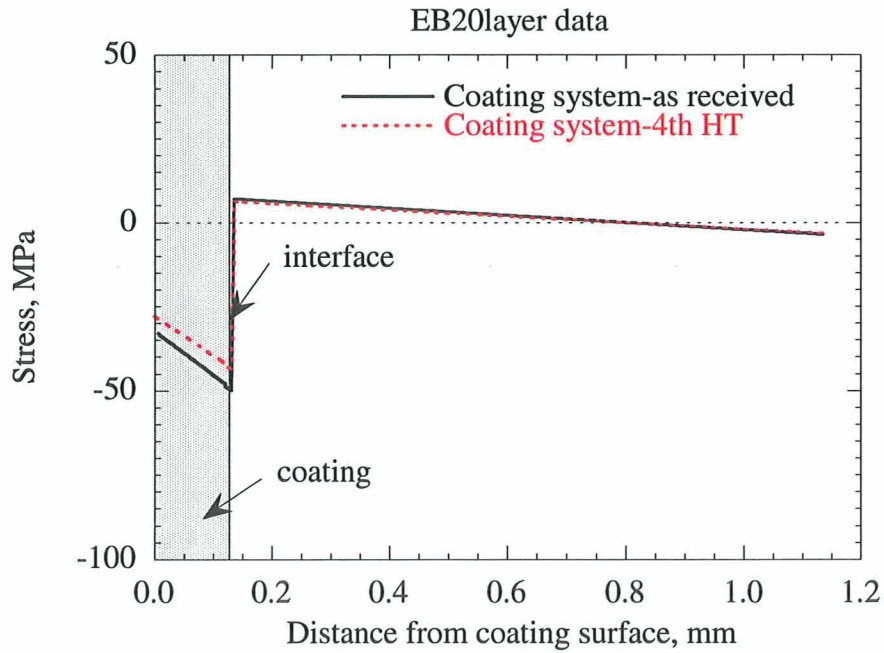


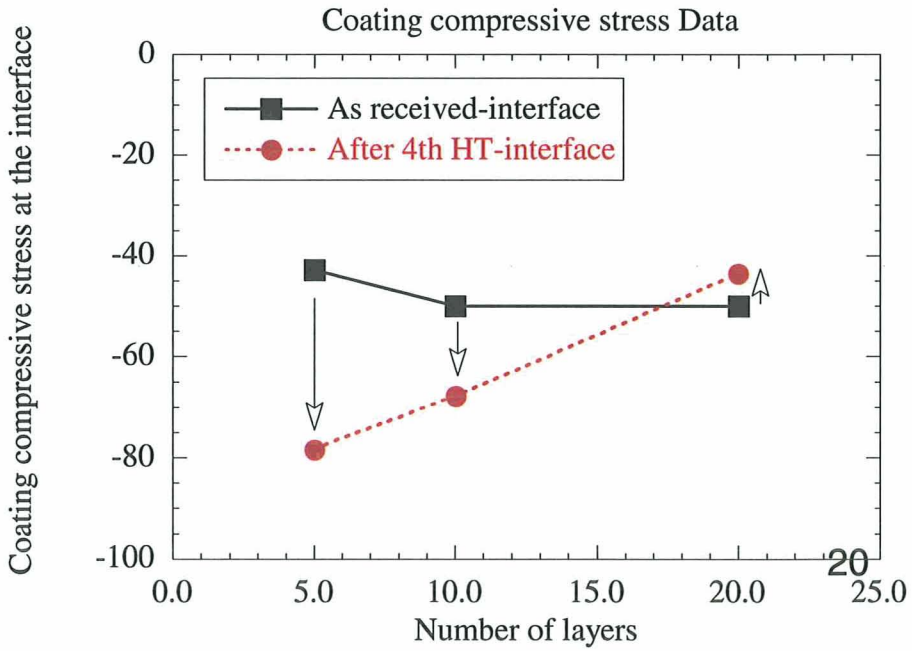


Stress Distributions in Bending Specimens









Thermal Cyclic Results

Thermal Cycling Results for Various TBC on CoNiCrAlY Bond Coated MAR-M-247 Buttons

CoNiCrAlY	TGO	TBC 8YSZ	TBC (μ m)	# of TBC layers	# of Cycles	Improvement
OEM	std	OEM	130	1	157, 143 > 131, 151	X
PSU	std	PSU	130	1	185,	23 %
PSU	std	PSU	129	40	216, 247 271+, 252+	45-54 %
PSU	std	PSU	136	60	216,	45 %

CoNiCrAlY	Al_2O_3 (0.5 μ m)	TBC 8YSZ	TBC (μ m)	# of TBC layers	# of Cycles	Improvement
PSU	10 layers	PSU	130	1	345, 334	125 %

OEM: Baseline

TGO: Heated upto 1000 ^{0}C and injected O_2 for 20 min @ 150 sccm flow before TBC evaporation (1X10^{-3} torr)

> Precipitation hardened HT for bond coat/base material.

Thermal Cyclic Results of Advanced HfO_2-based TBC on MAR-M-247 Substrate.

Sample #	Bond coatings	8YSZ 25µm	TransTech -TBC ingot composition	Thickness µm	Cyclic to failure
S000918-1H, 2H S001002-1F	Pt-Al	No	HfO_2 -27 Y_2O_3	138	4,9 7
S000929-1E, 1F	Pt-Al	YES	HfO_2 -27 Y_2O_3	138	11,12
S000921-1A, C	CoNiCrAlY	NO	HfO_2 -27 Y_2O_3	163	80, 109
S000929-1I	CoNiCrAlY	YES	HfO_2 -27 Y_2O_3	163	80
S000925-10	CoNiCrAlY	No	40 HfO_2 –40 ZrO_2 - 20 Y_2O_3	245	30
S000927-1I	CoNiCrAlY	YES	40 HfO_2 –40 ZrO_2 - 20 Y_2O_3	170	173

Thermal cycling results for HfO_2-27wt.%Y_2O_3 deposited on CoNiCrAlY/MAR-M-247

Sample Number	Substrate	Bond coating	TBC	Number of layers	# of cycles
S010615-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1	5
S010615-1J	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1 (HT)	5
S010618-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	2	4
S010619-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	5	4
S010625-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	20	1
S010626-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1	5
S010627-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40	1
S010803-1N	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40	5
S010802-1F	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1	6
S010802-1G	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1 (HT)	24

Preliminary thermal cycling results for HfO_2-27wt.%Y_2O_3 deposited on PtAl/MAR-M-247

Sample Number	Substrate	Bond coating	TBC	Number of layers	# of cycles
S010615-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	1	3
S010618-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	2	4
S010619-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	5	4
S010622-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	10	6
S010625-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	20	6
S010626-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	1	6
S010627-1B	MAR-M-247	PtAl	HfO_2-27Y_2O_3	40	6
S010627-1D	MAR-M-247	PtAl	HfO_2-27Y_2O_3	40 (HT)	6
S010802-1C	Rene-N5 **(1150C)**	PtAl	HfO_2-27Y_2O_3	1	5
S010802-1I	MAR-M-247	PtAl	HfO_2-27Y_2O_3	1	5
S010803-1E	Rene-N5 **(1150C)**	PtAl	HfO_2-27Y_2O_3	40	5

Thermal cycling results for HfO_2-27wt.%Y_2O_3 deposited on CoNiCrAlY/MAR-M-247

Sample Number	Substrate	Bond coating	TBC	Number of layers	# of cycles
S010615-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1	5
S010615-1J	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1 (HT)	5
S010618-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	2	4
S010619-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	5	4
S010625-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	20	1
S010626-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	1	5
S010627-1I	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40	1
S010803-1N	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40	5
S010802-1F	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40 (HT)	6
S010802-1G	MAR-M-247	CoNiCrAlY	HfO_2-27Y_2O_3	40	6

Thermal Conductivity

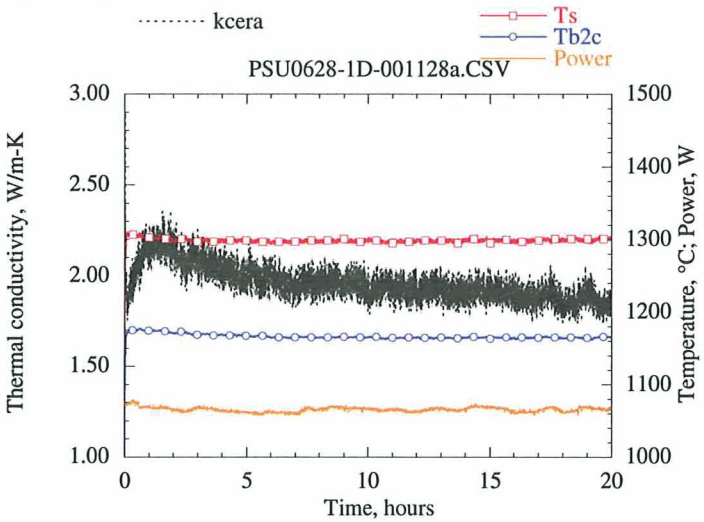
Thermal Conductivity Values of EB-PVD Coatings at Various Times

k0: initial conductivity
k20: conductivity after 20 hr testing
kmax: peak values and corresponding time

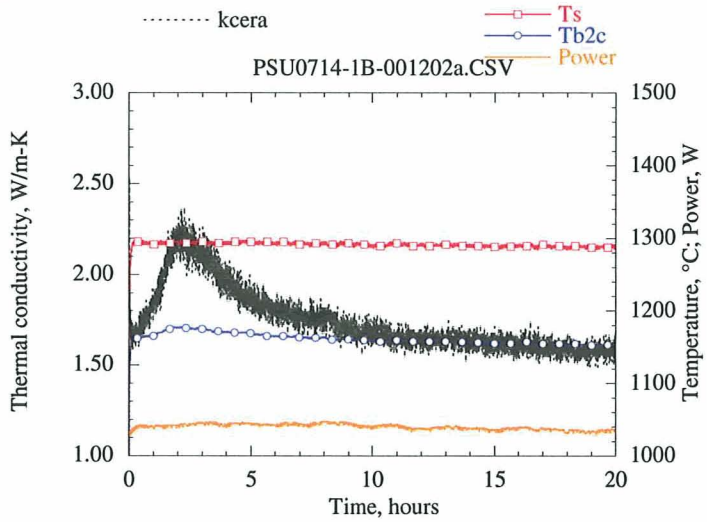
PSU EB-PVD Coatings	k_0 (W/m-K)	k_{max} (W/m-K) @Time, hour		K_{20} (W/m-K)
S000628-1D	1.88	2.18	1.1 hr	1.83
S000714-1B	1.67	2.17	2.4 hr	1.56
S000718-1B	1.74	2.23	1.8 hr	1.93
S000725-1B	1.77	2.19	2.0 hr	-
S000727-1B	1.59	1.89	1.0 hr	1.56

Thermal Conductivity of PSU-S000628 EB-PVD Thermal Barrier Coatings

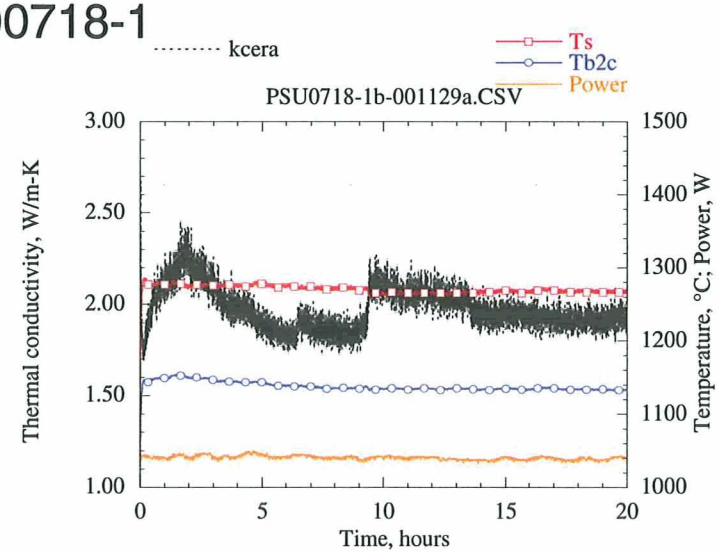
S000628-1



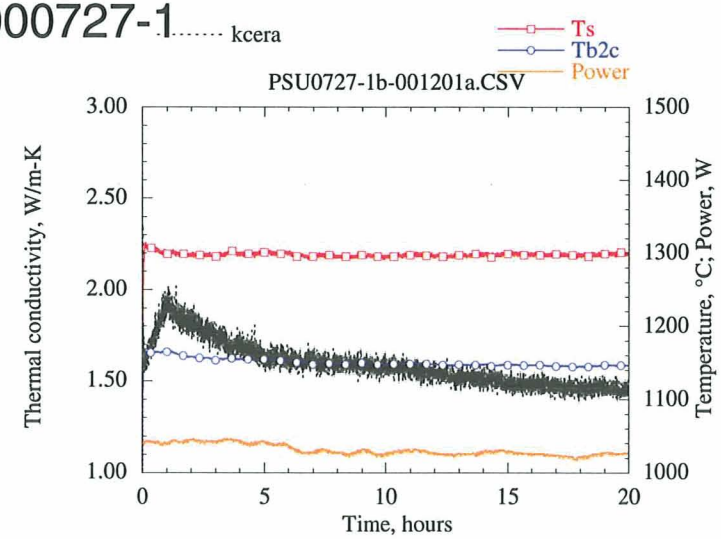
S000714-1



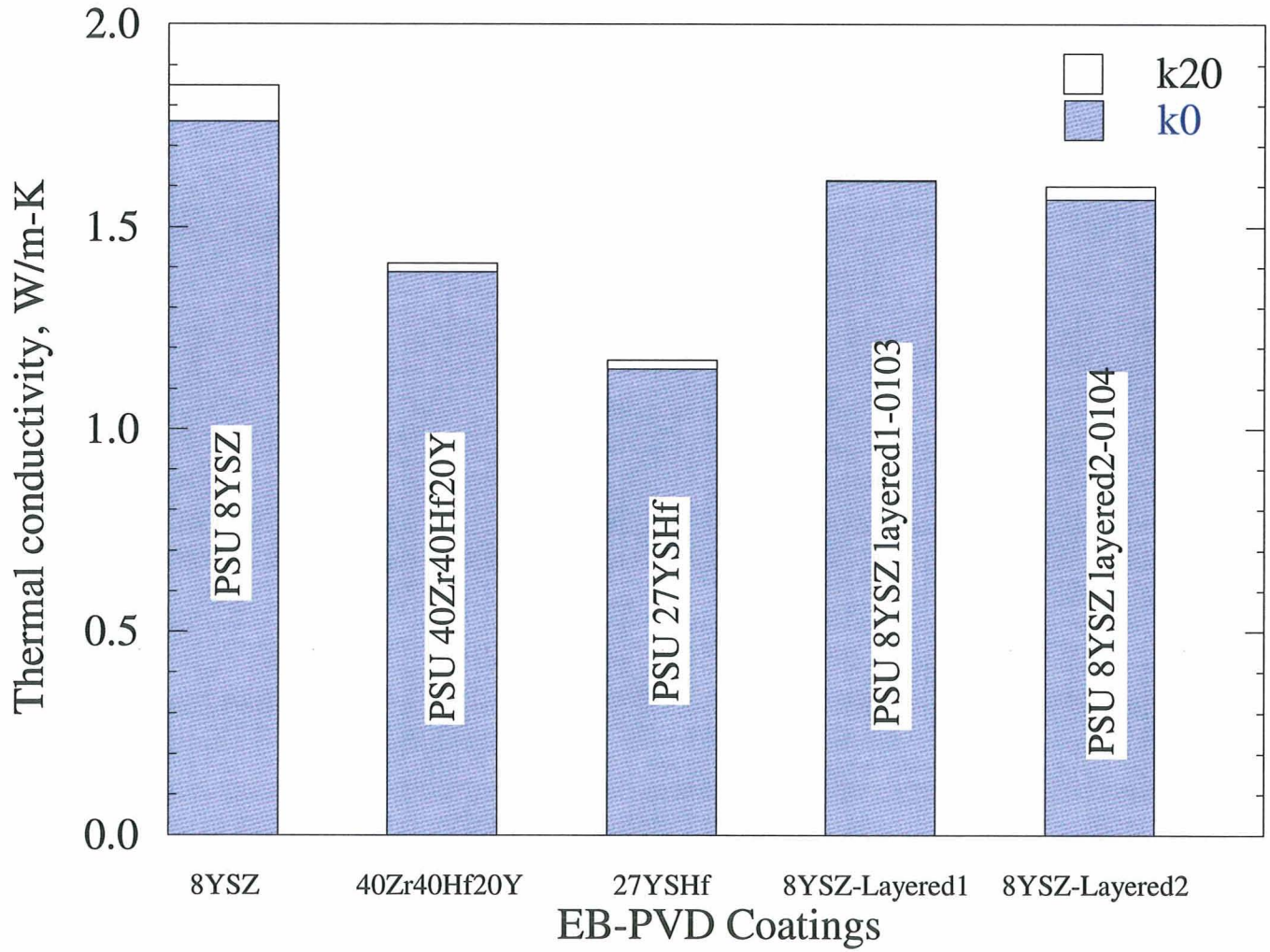
S000718-1



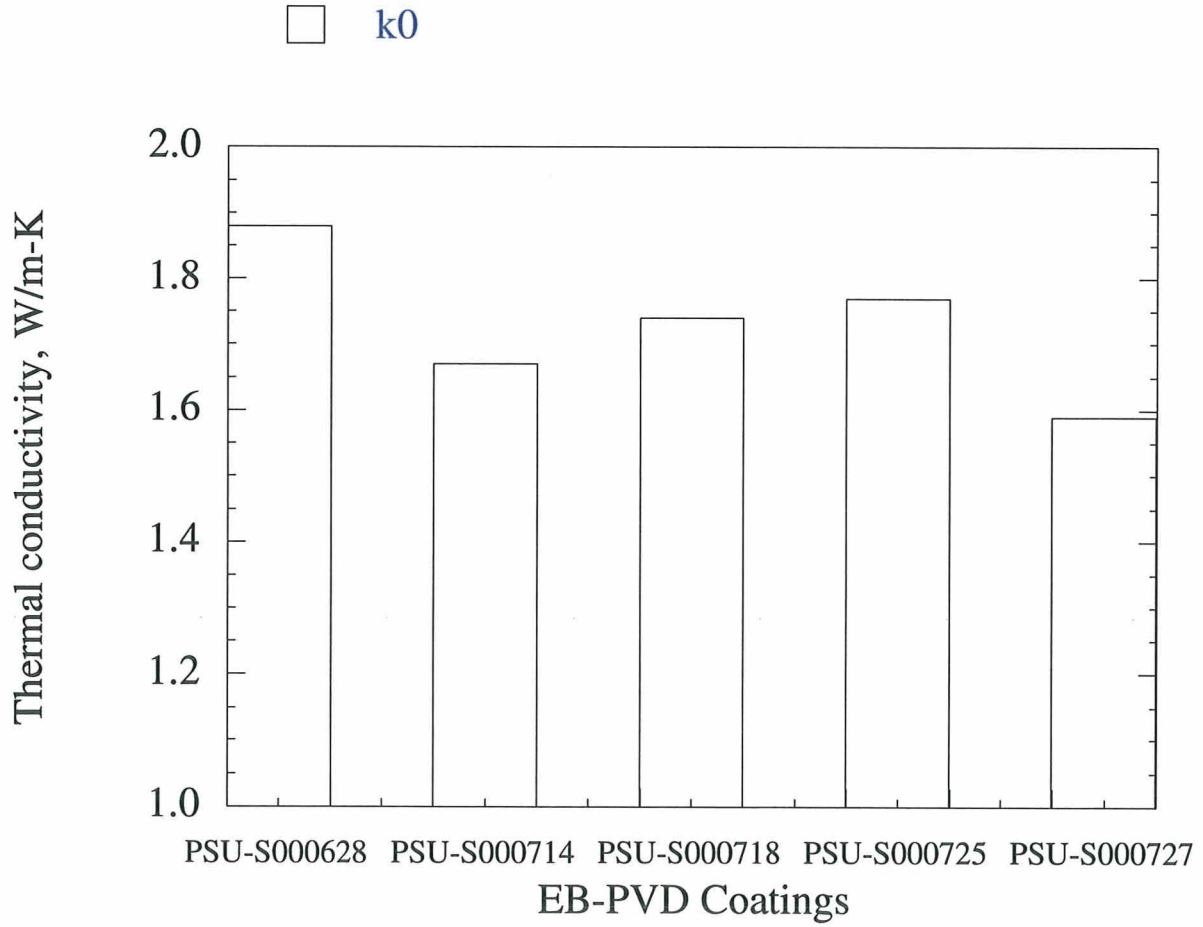
S000727-1



Thermal Conductivity of EB-PVD ZrO_2- and HfO_2-Based Thermal Barrier Coatings

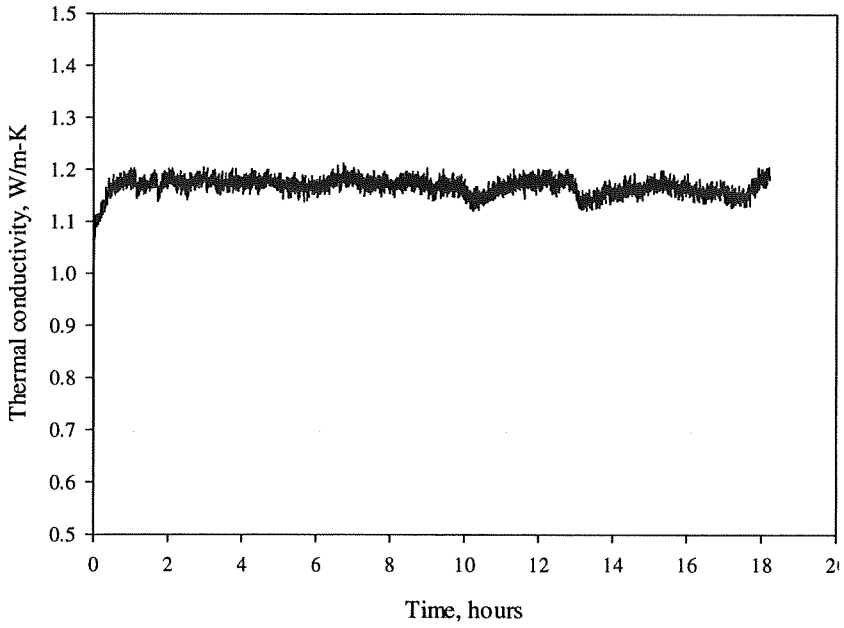


Summary of Thermal Conductivity EB-PVD Thermal Barrier Coatings

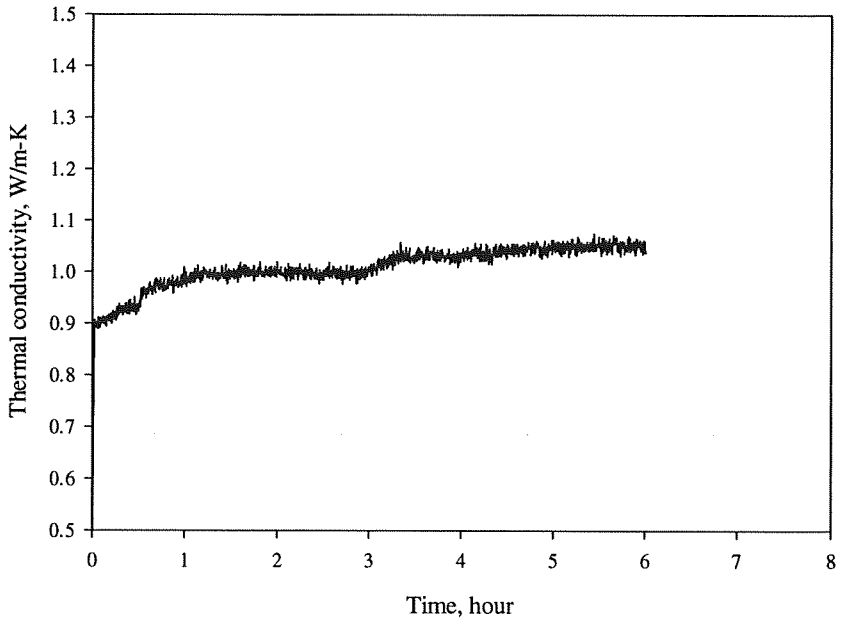


PSU Coatings: Laser Steady-State Conductivity Test

psu27yshf-1L-4b2d-010904



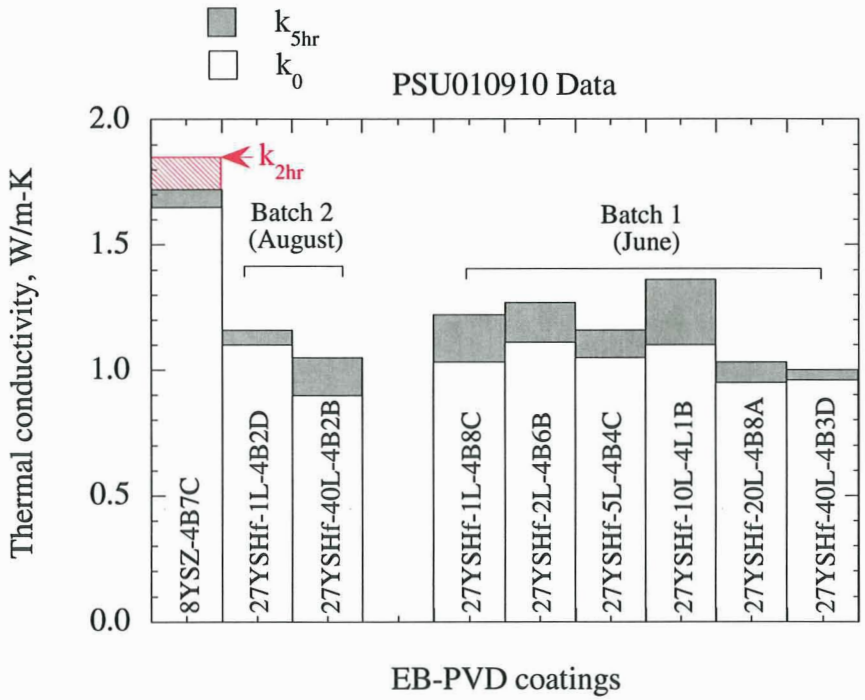
psu27yshf-40L-4b2b5-010904a

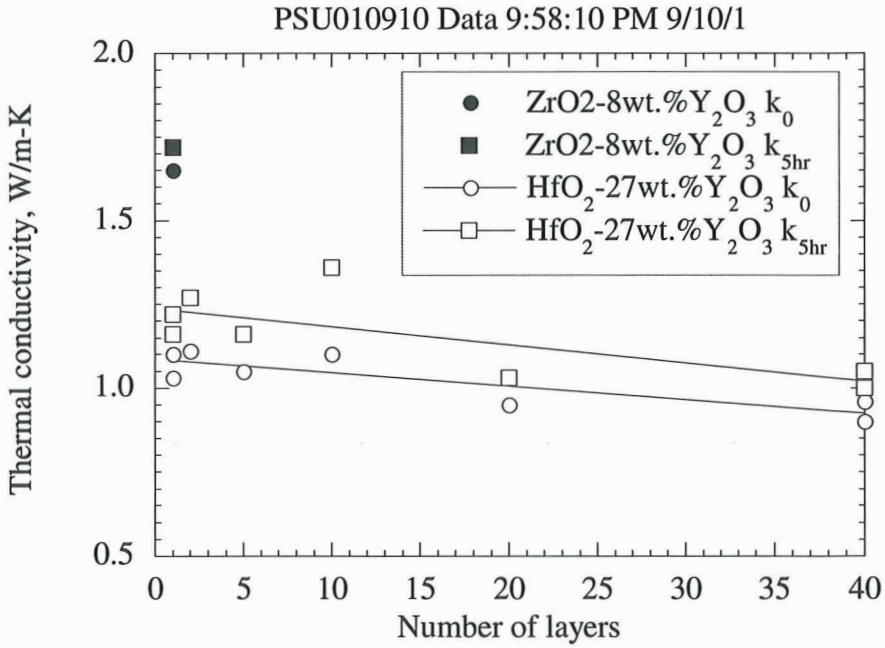


Thermal Conductivity of Penn State EB-PVD Coatings

— Laser tested at $T_{surface}$ ~2400°F

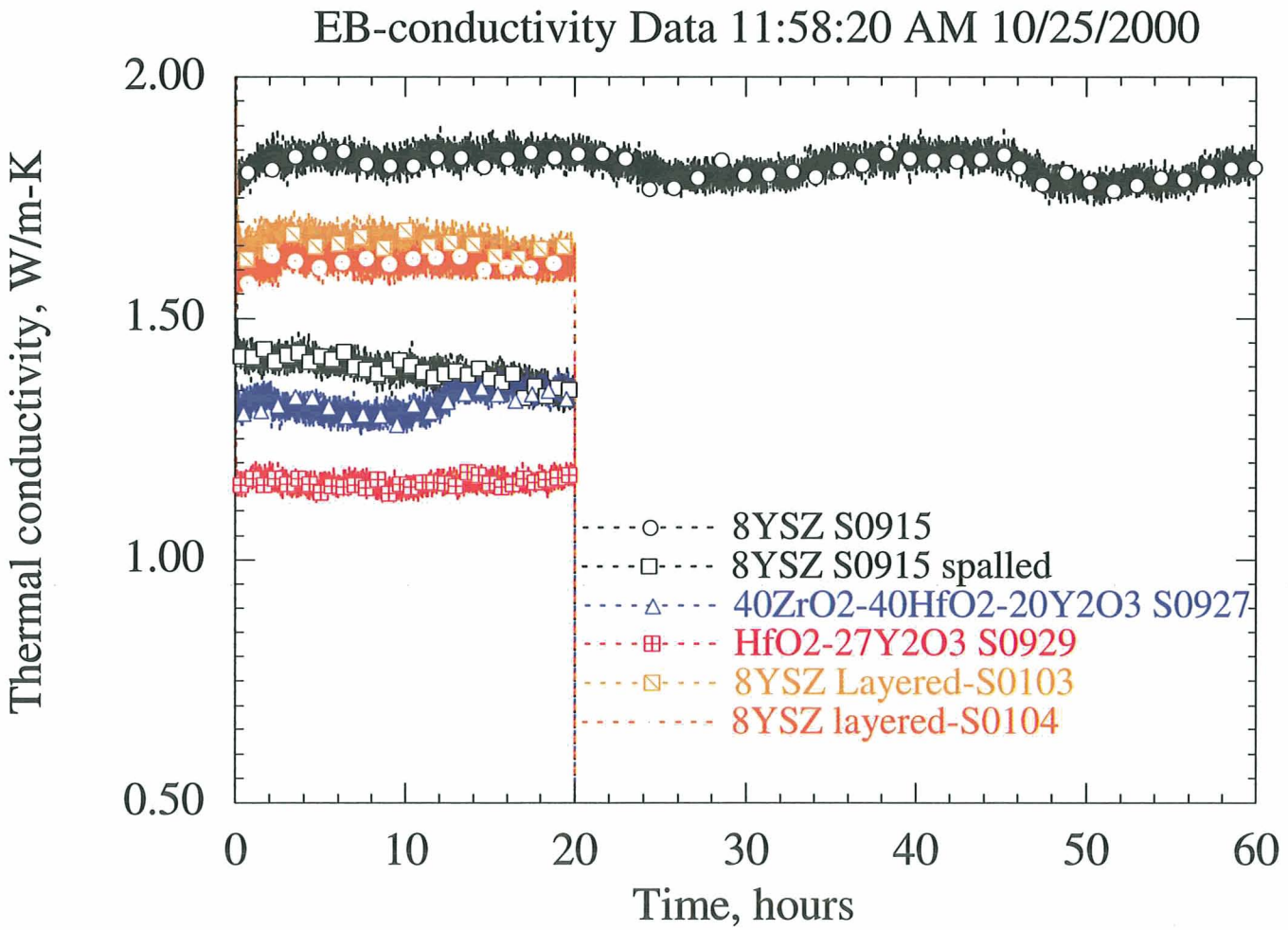
— Initial conductivity k_0 and 5 hr conductivity k_{5hr} values are shown





Thermal Conductivity of EB-PVD ZrO_2- and HfO_2-Based Thermal Barrier Coatings

— Tested at 2400°F using laser steady-state heat flux rig



SUMMARY

Summary of Layered 8YSZ

- **Reduction in thermal conductivity by 12-15%**
- **Reduction in emissivity**
- **Reduction in compressive stresses on components**
- **Improvement in thermal cyclic life 40-50%**

Summary of Layered HfO_2-based TBC

- **Differences in coating performance between different ingot suppliers**
- **Low cyclic life of HfO_2-based TBC primarily due to dense microstructure**

HfO_2 -27% Y_2O_3 Ingot supplier: Norton Inc.

- **Theoretical Ingot Density 43% (desirable 60-65%)**
- **Deposition conditions: Ts: 1000 C, Oxygen supply: 150 SCCM**

Layer #	Grain size	Microstructure	X-ray
1	faceted	individual columns	<200>
2	faceted	individual columns	<200>
5	faceted	individual columns	<200>

Melt pool condition: very unstable contributed to additional unexpected layered structure.

HfO_2 -27% Y_2O_3 Ingot supplier: Phoenix Coatings Research

- **Theoretical Ingot Density 66%**
- **Deposition condition: Ts: 1000 ^{0}C, Oxygen supply: 150 SCCM**

Layer #	grain size	Microstructure	X-ray
1	unchanged	Dense fractionated columns	<220>
10	unchanged	Dense fractionated columns	<200> ?
20	unchanged	Dense fractionated columns	<220>
40	unchanged	Dense fractionated columns	<220>

- **Deposition condition: Ts: 1150 ^{0}C, Oxygen supply: 150 SCCM**

Layer #	grain size	Microstructure	X-ray
1	unchanged	individual columns	<220>
40	unchanged	individual columns	<200>

Future effort for NASA-GRC

- Deposit 5 HfO_2 alloyed TBC at 1150 ^{0}C on:
  - Pt-Al bond coated Rene N5
  - CoNiCrAlY bond coated Rene N5
  - 8YSZ (1 mil)/Pt-Al bond coated Rene N5

- Repeat: HfO_2 –27% Y_2O_3 at 1150 ^{0}C on:
  - 8YSZ (1 mil)/Pt-Al and CoNiCrAlY, bond coated Rene N5

- Layered 8YSZ with and without ion source on Rene N5 samples
  - Pt-Al bond coated
  - CoNiCrAlY bond coated

- Characterization of TBC
  - Thermal cyclic tests, SEM, X-ray, thermal conductivity, emissivity

•Project funding was cut short so additional research could not be performed