201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Superconducting YBCO MOCVD Technology at SMI

Summary of superconducting YBCO thin film technology at Structured Materials Industries, Inc. (SMI)

January 2011



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Superconducting YBCO MOCVD Technology at SMI

### **Presentation Outline:**

- Introduction to SMI
- Process Development for Undoped YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> on SrTiO<sub>3</sub>
- Process Development for Ho Doped YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> on SrTiO<sub>3</sub>
- Process Development for CeO<sub>2</sub> Buffer Layers for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>
- Process Development for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> on Buffer Coated Metal Tape
- Hardware Development for Reel-to-Reel MOCVD of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> on Kilometer Scale Metal Tape
- Next Generation Hardware for MOCVD of YBCO Tapes



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# **SMI/GNM Founder**

Dr. Gary S. Tompa: Materials Science Expert and Serial Entrepreneur



- -President and CEO of SMI,
- Former Head of Systems Research, Engineering and other positions at Emcore Corp. (TurboDisc group Veeco subsequently purchased that group)
- Contracted by Applied EPI 2001 (\$10M\$ budget -build 4 MOCVD tools (Veeco bought them out)
- Co-Founder of multiple companies— exited three successfully, still participates in 3: CoB/co-founder of NEI Corporation (www. NEICorporation.com); Board member of UNTPL (joint venture of NEI and UNPL of India to manufacture battery electrode material, Board member American Nanomite)
- Managed >\$40M in development contracts
- >\$10M in commercial sales to Companies, Universities and Government Laboratories
- Came to SMI to develop/implement Si QNC light emitters moved it to MOCVD niches and ultimately to "organically" grow with customers and spinouts.
- Understands what can be done, how to build a team and how to build a business
- >100 technical publications, ~>100 conference papers, several patents
- Member of advisory board to multiple universities



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Structured Materials Industries Inc. (SMI)

### **Core Business:**

New Devices are Built in New Materials
New Materials Need (New) Production Tools

SMI's produces Metal Organic Chemical Vapor Deposition (MOCVD) tools that

**Enable Next Generation Devices** 

**Examples: Wide Bandgap Materials** 

**Memory Materials, Graphene** 

**Transparent Conductive Materials Superconductors, Other Materials** 

### **Business Model:**

Development Funded with SBIR's/Service Contracts
Sell MOCVD Systems, Components, Services
Grow Organically or With Scale-up Investment
Spin-off Companies (4 to date)





Tibbets Award Winner







201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

## **SMI** is a Sound Company

Incorporated: 1992

Privately held: 1 principle with ~ 90% ownership

Financial: Self-funded: no long-term debt, loans, warrants, liens,

or other financial obligations

**Defense Contract Auditing Agency** 

annual accounting audit passed every year

**Profitable operation since 1995** 

Retained earnings are used for self-funded working capital

Legal: No known negative issues: No past, present or

known / anticipated legal actions -- by us or against us



## **SMI** is Presently "Two Businesses"

- 1. Government/Commercial contract R&D services research of processes, tools, materials, and devices
- 2. Equipment developer / provider R&D scale by infrastructure and resources

"Organic" growth with customers for production



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

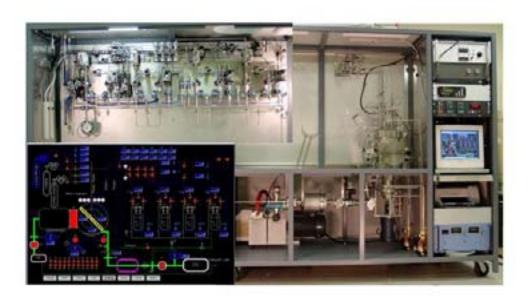
Selected Major Government Contracts @ SMI		
Si nanocrystals for light emission		
ZnO TCO	Re-Programmable FPGA	
PZT CVD	Prompt Dose FPGA	
PLT/PZT pyroelectric	Rad Hard COTs	
Al2O3 H passivation	Graphene tools	
CMO NDRO EEPROM	2DEG LaAlO ALD	
Sb HBT	InGaN PV I	
YBCO TAPE TOOL	Carbon Nitride	
p-type ZNO	CF-CVC nanopowd	
Radiation Sensor	TPV InGaSb	
Phase Change memory material	LNBO waveguide	
Pyrophoric Coatings	FBCVD particle coating	
InGaN HOVPE	InGaN PV II	
WPT	Si Photonic waveguide	
Thermoelcctrics	2DEG films	
BST variable dielectric	Si resonator LIDAR	



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# MOCVD tools that SMI has developed and implemented cover the range from R&D to Cluster Tool Production.

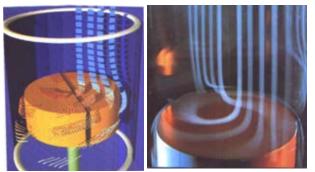






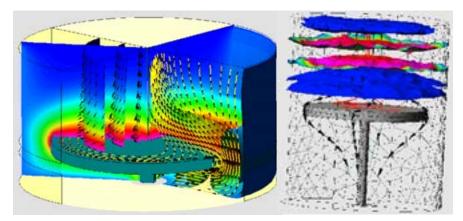
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Tools Designed by Modeling

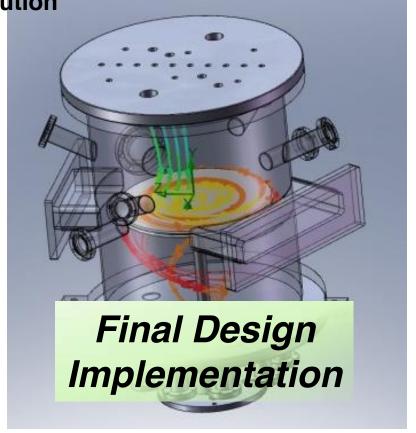


Parallel modeling effort will help converge on optimal solution

**Courtesy of Sandia National Laboratories** 



Uniformity & Nanolayer (quantum well) Control



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# SMI Has aVery Broad Range of Materials Expertise

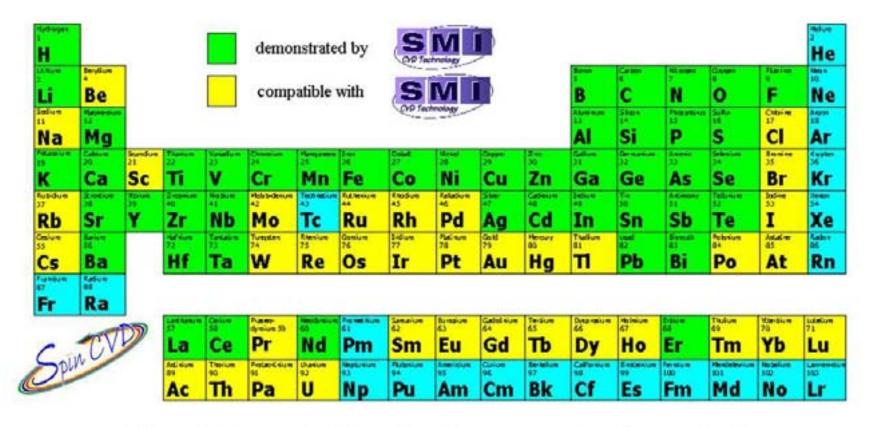


Table of elements showing the range of Elements that SMI's MOCVD technology has or can produce.



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### Facilities I (7,500 sq. ft.) – more on the way

# Table F1: SMI In-House Applications Laboratory: MOCVD Tools from RESEARCH to PRODUCTION



MOCVD: p-type ZnO



MOCVD: Static Optical



MOCVD: Electro-Optical



MOCVD: TCOs



MOCVD: NanoCVD



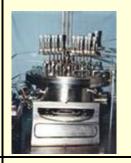
MOCVD: GaN



Production Cluster Tool: SiGe/GeSbTe



Production Cluster Tool: Ferroelectric



MOCVD: Stand-Alone Ferroelectric



Production Cluster Tool: Dielectrics

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854

Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### **Facilities II**

### Table F2: SMI Analytic and Related Capabilities



MEI Wedge Bonder



Rigaku X-Ray Diffraction System



Hall Effect Measurement station



Inert atmosphere glove box (<1 ppm H<sub>2</sub>O, O<sub>2</sub>)



1500x Polylite Inspection Microscope



Filmetrics Thin Film Reflectance Spectrometer



JOBIN YVON Optical Spectrometer



HP Parametric and Impedance Analyzers, and Wafer Probe



8" wafer photoresist spinner



Dektak profilometer



Differential Scanning Calorimeter

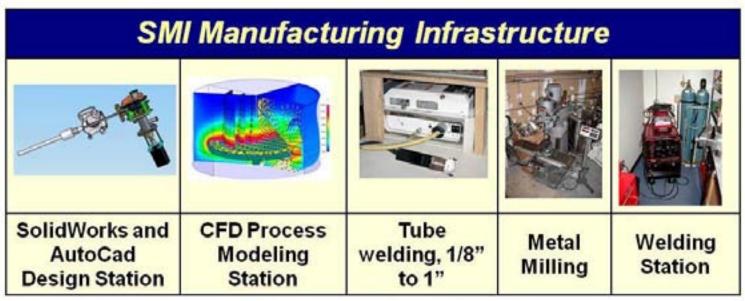


Annealing Furnace



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

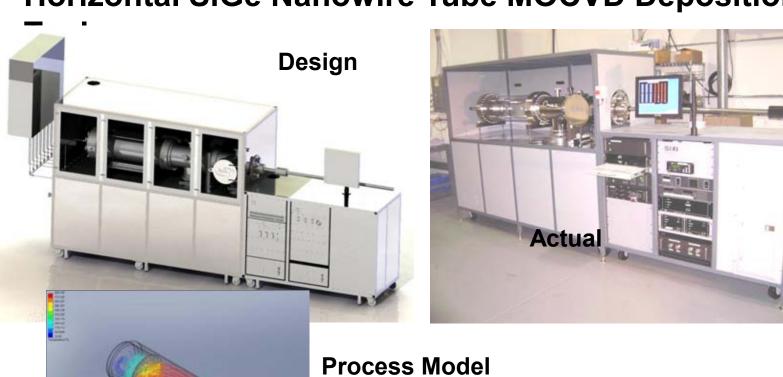
### **Facilities III**





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### Horizontal SiGe Nanowire Tube MOCVD Deposition

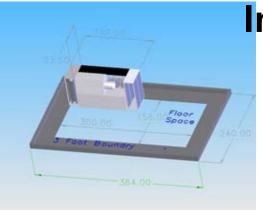




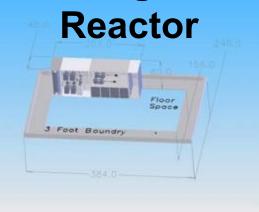
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# **Facility**

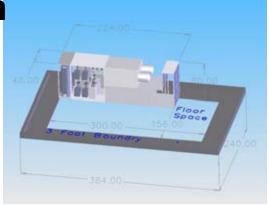
**I**ntegration



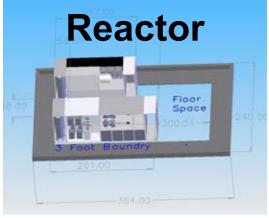
Single



Dual Vertical Reactor



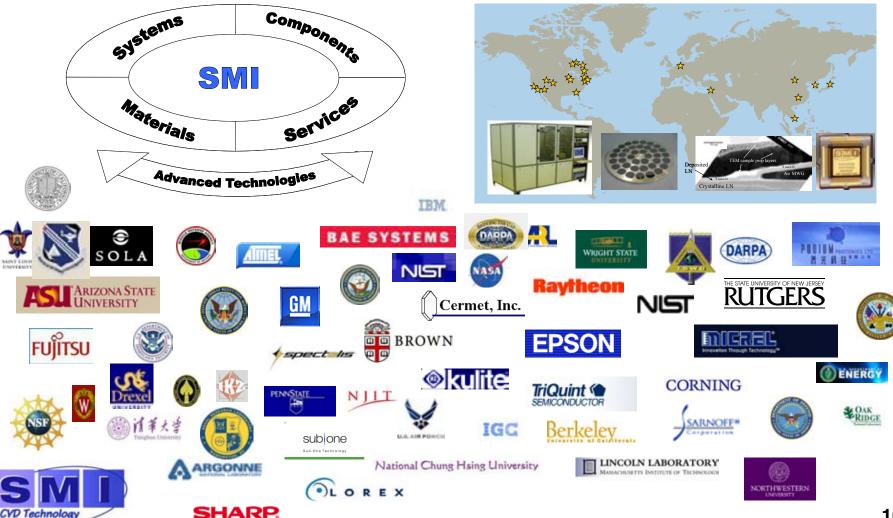
**Dual Horizontal** 



**Two Systems** 

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# SMI International Commitment to Customer Excellence (selected logos)



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

				UTo – Graphene
vernment Contracts:	Closed, Active, Awarded:	>\$30,000,00	0.00	NRXX – 2 HWCVD
vernment Contracts:	Pending:	100000000000000000000000000000000000000	>\$3,500,000.00	UFX - Nitride/oxide
mmercial contracts: 0	Closed, Active, Awarded::	>\$7,000,000	.00	
mmercial contracts: I	Pending with >50% proibability:	7.000	>\$4,000,000.00	UCX – III-V
uasar Essas III	20/4%	>\$30,000,00		CXX – TCO
Il Spinouts - Raised &	Sold	0.00		AFXX – GaN
Some	2004 - 2004 Onjekt - 1 (1400 on	~\$4,000,000		
SMI Spin	-Out Joint Venture in India	.00		New Orders in Jan 2011
				11011 0101010 111 0011 2011
nmary of Major Sale	s/Services			XXR – GaN
TOOLS	Does not include ~10 in-house tools	Subsystems		Major in-house tool
			Multiple showerheads, some	addition starts:
	GaN Reactor Assembly		Control systems multiple (A	
	GaN system			Graphene (2)
	GaN test system	Coatings		Subl;imation tool (1)
	GaN heater assembly		Oxide coatings, LiNbO, PZT,	ALD tool (2)
	Dielectric/Ferroelectric System		Transistor, chloride	GaN HOVPE Tool (1)
	Dielectric oxide system		Oxide coatings luminescent	
	GaN Tube tool (in process)		Calcoginides/CBRAM/PCRAM	dep services
	PV HWCVD tool (2 on a cluster - in process)		ZnO tool use / services	
	ZnO System		Special Nitride Foil Depositio	ns
	ZnO system		InGaSb InGaAs ErSb	y-1,-
	SiC Bulk material growth		High temperature lamp coati	ng
	Superconductor tape tool, Tape tool rebuild, Deposition services		PZT evaquation	
	Ferroelectric System		Several others	
	SiGe Nanowire Tool			
	High Temperature Oxide MOCVD system	System Rebu	ailds Conversions	
	NanoH Si/SiO2		SiC, GaN System rebuilds, Ga	
	DeskTop CVD™ oxides		And the state of t	GaN to TCO, Plasma dep to PZT+C30
	Thin Flim Battery PECVD Tool		GaN Shaft heater conversion	1
	Graphene DeskTop CVD™			
	NanoV CNTs	Other Tools		
	Vacuum nanopowder system		GFHC	23
			DESIGN PV coater and PV And	AND AND ASSOCIATION OF THE PARTY OF THE PART
			Design Applied Epi MOCVD to	ools (sold out to Veeco)

New Orders: - 2010

ND - Graphene NR - Graphene

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854 Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### **EMCORE DEPOSITION SYSTEMS DESIGNED** Principally by Dr. G. S. TOMPA

**ORGANIZATION** 

**EASTMAN KODAK CORPORATION** ZnO/SiO2 OXIDE MOCVD

ROCHESTER, NY

FLORIDA STATE UNIVERSITY HTC OXIDE MOCVD

TALLAHASSE, FL

**MOTOROLA\*** 

**AUSTIN, TX** 

NASA, GODDARD\*\*

**GREENBELT, MD** 

NATIONAL INSTITUTE OF STANDARDS

**BOLDER, CO** 

**NAVAL RESEARCH LABORATORIES;** 

**WASHINGTON D.C.** 

**NORTH AMERICAN PHILLIPS** 

**BRIARCLIFF, NY** 

STEVENS INSTITUTE OF TECHNOLOGY

HOBOKEN, NJ

DUPONT

**DELAWARE** 

UNIVERSITY OF CONNECTICUT

STORRS, CT **EMCORE** 

SOMERSET, NJ

SYSTEM TYPE

OXIDE PEMOCVD GASPANEL

III-V/OXIDE MOCVD

**III-V CBE** 

HTC OXIDE MOCVD

II-VI MOCVD

HTC OXIDE MOCVD

HTC OXIDE MOCVD

II-VI MOCVD

II-VI VTE(CBE) IV VTE(CBE)

YBCO OXIDE-PROTOTYPE MOCVD

**II-VI MOCVD** 

3600 PROTOTYPE (E400 forerunner) **ENTERPRISE SYSTEM (1st ONE)** 

\*GAS PANEL TO MATE WITH PLASMA QUEST GROWTH REACTOR SYSTEM

\*\* OXIDE PORTION OF DUAL SYSTEM



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### **Example Installed Customer Tools**



Examples of SMI's commercially installed MOCVD reactor products that span the range from research to production, in sizes ranging from small stand-alone units up to cluster tools for high-volume production; producing materials from oxides to GaN to SiC.

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# SMI Projects on Superconducting YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD

- "High Current Capacity YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Coated Conductors" Sponsor DOE
- "Thick YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films for Coated Conductors with Improved Critical Current" Sponsor Air Force
- "Effective Flux Pinning In YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Coated Conductors by Continuous MOCVD" Sponsor Air Force
- Delivery and Implementation of Reel-to-Reel MOCVD Tool for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Superconducting Tape Production to Air Force Research Laboratory at WPAFB
- "Multi-Kilometer Superconducting Tape Production Tool" Sponsor MDA/Air Force



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### **SMI Core Superconducting Team:**

Dr. Gary S. Tompa, Corporate and Technical Leadership

Dr. Nick Sbrockey, PI on Several Government Programs, YBCO process and characterization leader

Mr. Tom Salagaj, Sr. Process & Tooling Development Engineer

Mr. L. Gary Provost, Sr. Design and Manufacturing Engineer

Several outside consultants and manufacturing service providers as needed.



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

#### Dr. Nick Sbrockey:

Structured Materials Industries, Piscataway, NJ (2002 - Present): Principal Scientist

Develop new process and hardware technologies for SMI's line of thin film deposition equipment. Initiate, manage and conduct R&D programs, including development of strategic partnerships with companies, universities, government laboratories and funding agencies. Dr. Sbrockey has managed R&D programs at SMI on graphene and diamond based devices, tunable RF devices based on ferroelectric and multiferroic materials, high-k dielectrics, optical coatings, laser damage resistant optical coatings, photovoltaics, thermophotovoltaics, electro-optic and photonic crystal devices, laser wireless power transfer and superconducting YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> coated conductors.

Axcelis Technologies/Eaton Corporation, (1986 - 2001): Various Positions

Develop new technologies and new products for Axcelis/Eaton, including ion implantation, thermal processing, photoresist processing and cluster tool equipment. Dr. Sbrockey managed R&D programs for Axcelis/Eaton on plasma immersion ion implantation, flat panel display processing tools, broad beam ion source development, microwave plasma processing, diamond CVD, CVD of tungsten and tungsten silicide and unbalanced magnetron sputtering of hard coatings. Dr. Sbrockey's responsibilities also included Thin Films Research Group Manager (1986 - 1991) and Technical Liaison between Eaton's R&D Center and Semiconductor Equipment Divisions (1994 - 1997).

Balzers High Vacuum Systems and Components, Hudson, NH (1984 - 1986): Process Scientist Develop new process and hardware technology to support Balzers line of plasma etching and thin film deposition equipment products.

Synergistic Activities:

Member - Board of Directors for Electro-Optic Alliance at Penn State University: 2005 - 2006.

Member - Organizing committee for AVS Spring Meeting: 1998 - 1999.

Session Chairman at AVS Int. Conf. on Microelectronics and Interfaces: 1996 - 1999.

Session Chairman at Int. Materials Research Congress: 2010.

Member - Materials Research Society and American Vacuum Society: since 2000



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### Mr. Tom Salagaj:

### Structured Materials Industries, Piscataway, NJ (2010-Present): Project Manager

Coordinate the design and process development for custom designed MOCVD/CVD deposition systems used in the Nano, Solar, and Semiconductor markets. Provide technical support to customers and to the "In-House" research team in the SMI applications lab.

First Nano/CVD Equipment, Ronkonkoma, NY (2006- 2010): Applications Laboratory Manager Manage the daily operations of the 15,000sqft laboratory facility. Oversee a team of scientists and engineers for the development of Carbon Nanotubes, ZnO & GaN nanowires, and Si and ZnO epitaxial layers. Coordinate the schedules and provide technical support to the customers.

### IGC-SuperPower, Schenectady, NY (2003-2006)\_Senior Process Specialist

Transition the Metal Organic Chemical Vapor Deposition (MOCVD) process for the deposition of YBaCuO superconducting materials from development into full scale production. Responsible for the "Scale-Up" of the MOCVD reactor from a research test stand to a kilometer length production scale system. Awarded a patent for the reactor and tape handling system design for the "Method of making a superconducting conductor"

Uniroyal Optoelectronics, Tampa FI (2001-2003) Senior Epi Manufacturing Engineer Oversee daily operation and maintenance of Metal Organic Chemical Vapor Deposition (MOCVD) reactors, for the growth of

Emcore Corporation Somerset, NJ (1986-2001) Senior Manufacturing Engineer (1996-2001)

Gallium Nitride UV (400nm), Blue (470nm), and Green (505nm) Light Emitting Diodes (LEDs).

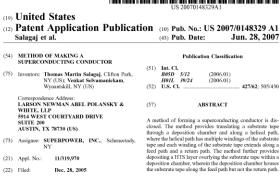
Assist in the transition of processes from R&D into production for the Electronic Materials Division. Create related documentation (i.e. work instructions, test plans, delivery schedules, etc.) to maintain compliance to ISO 9000 guidelines. Sustain the manufacturing of a product through a high volume production line. Managing and maintaining yields, resources, and deliverables for a 24 hour 7day a week operation. Increase overall throughput of product by utilizing Cycle Time Reduction and Failure Mode Effects Analysis (FMEA) techniques.

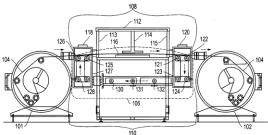


201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854

Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### Tom Salagaj: Key Technology Patents and Publication





Co-Author of over 30 technical publications and presentations.

Selected Publications relevant to the project:

- "Scale-up of Applications-ready Practical Y-Ba-Cu-O Coated Conductors." IEEE Trans. Appl. Supercond. 15, pp. 2596-2499 (2005), V. Selvamanickam, A. Knoll, Y. Xie, Y. Li, Y. Chen, J. Reeves, X. Xiong, Y. Qiac T. Salagaj, K. Lenseth, D. Hazelton, C. Reis, H. Yumura, and C. Weber,
- 2. "Progress in Scale-Up of Second-Generation High-Temperature Superconductors at Superpower" Physica C. 426-431, pp. 849-857 (2005), Y.-Y. Xie, A. Knoll, Y. Chen, Y. Li, X. Xiong, Y. Qiao, P. Hou, J. Reeves, T. Salagaj, K.Lenseth, L Civale, B. Maiorov, Y. Iwasa, V. Solovyov, M. Suenaga, N. Cheggour, C. Clickner, J.W. Ekin, C. Weber, and V. Selvamanickam
- 3. "Progress towards Application Readiness of Coated Conductors at SuperPower" Proc. Intl. Workshop on
- 4. Coated Conductors for Applications, Oct. 3' - Nov. 2, Kanagawa, Japan (2004), Y.-Y. Xie, A. Knoll, Y. Li, X. Xiong, Y. Qiao, Y. Chen, P. Hou, J. Reeves, T. Salagaj, K. Lenseth, C. Weber, and V. Selvamanickam



US 6,197,121 B1

Mar. 6, 2001

(12)	United	States	Patent
	Gurary et	al.	

(54) CHEMICAL VAPOR DEPOSITION

CALL A CAMACA	
Inventors:	Alexander I. Gurary, Bridgewater;
	Richard A. Stall, Belle Mead; Rober
	F. Karlicek, Jr., Flemington; Peter
	Zawadzki, Martinsville: Thomas
	Salagaj, South Plainfield, all of NJ

Subject to any disclaimer, the term of this

(21) Appl. No.: 09/345,032 (22) Filed: Jun. 30, 1999

#### Related U.S. Application Data

	**
(63)	Continuation of application No. 08/757,909, filed on Nov. 27, 1996, now abandoned.
(51)	Int. Cl.7 C23C 16/00
(52)	U.S. Cl
(58)	Field of Search 118/715, 719,
	118/724, 725, 730

	U.S. PATI	ENT DOCUMENTS
3,645,230	2/1972	Huggle .
4,446,817	5/1984	Crawley .
4,607,591	8/1986	Stitz .
4,771,730	9/1988	Tezuka .
4,794,220	12/1988	Sekiya .
4,997,677	3/1991	Wang et al
5,024,748	6/1991	Fuiimura .
5.063.031	11/1991	Sato .
5,136,975	8/1992	Bartholomew et al
5,173,336	12/1992	Kennedy .
5,174,825	12/1992	White .

5,344,492	9/1994	Sato et al
5,370,739	12/1994	Foster et al
5,421,892	6/1995	Miyagi .
5,500,256	3/1996	Watabe .
5,595,606	1/1997	Fujikawa et al.
5,653,806	8/1997	Van Buskirk .

(10) Patent No.: (45) Date of Patent:

#### FOREIGN PATENT DOCUMENTS

61-101020	5/1986	(JP).
1-177228	7/1989	(JP).
1-294868	11/1989	(JP) .
8-291385	11/1996	(JP) .

OTHER PUBLICATIONS

G. Tompa et al., "Design and applications of large area RDRs," III-Vs Review, vol. 7, No. 3, 1994.

H. Hitchman and K. Jensen, "Chemical Vapor Deposition Principles and applictions," Academic Press, 1993; pp 59-65.

D. Fotiadis, A. Kremer, D. McKenna, K. Jensen, "Complex phenomena in vertical MOCVD reactors: effefts on deposi-tion uniformity and interface abruptness," *Journal of Crystal Growth* 85 (1987) 154–164.

Primary Examiner-Richard Bucker (74) Attorney, Agent, or Firm—Lerner, David, Littenberg, Krumholz & Mentlik, LLP

#### ABSTRACT

ABSTRACT
Reactors for growing epitaxial layers on substrates are disclosed including rotatable substrate carriers and injectors for injecting gases into the reactor towards the substrates on the carriers and including a gas separator for separately to the carriers and including a gas separator for separately to the carriers and including a gas separator for separately to. Various reactor embodiments are disclosed including removable gas separators, and particular injectors which midule cooling chambe, as well as flow restrictors mounted within the carrier of the carriers of the carri

58 Claims, 3 Drawing Sheets





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

### L. Gary Provost

**Under Development** 



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Process Parameters for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD

<b>MOCVD Process Parameters</b>	
Metal Organic Precursors	$Y(thd)_3 / Ba(thd)_2 / Cu(thd)_2$
Precursor Ratio	10% / 53% / 37%
Solvent	Tetrahydrofuran
Molar Ratio Precursor / Solvent	0.06 moles/liter
Feed Rate	0.25 ml/minute
Flash Evaporator Temperature	260 C
Gas Line Temperatures	280 C
Shower Head Temperature	280 C
Susceptor Temperature	750-850C (775C typical)



26

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Process Parameters for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD

<b>MOCVD Process Parameters</b>	
Uniform O <sub>2</sub> Flow	300 sccm
Uniform Ar Flow	300 sccm
Flash Evaporator Push (Ar)	500 secm
Chamber Push (Ar)	500 sccm
Chamber Pressure	2.50 Torr
Flash Evaporator Pressure	~ 40 Torr
Deposition Rate	0.30 μm/hr

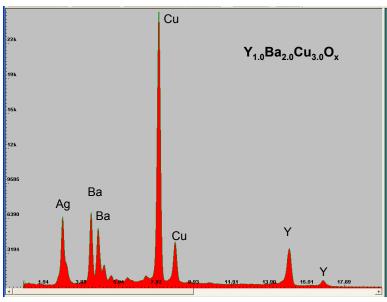


27

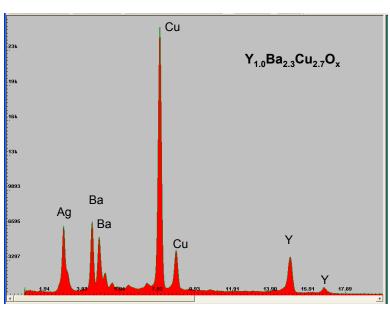
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

X-Ray florescence (XRF) provides for easy *qualitative* analysis of YBCO films on silicon witness substrates.

Most of the common substrates for YBCO (i.e. SrTiO<sub>3</sub>, LaAlO<sub>3</sub>, YSZ) have overlapping peaks with YBCO, which interfere with *quantitative* XRF analysis.



Bulk YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Standard



**MOCVD YBCO Film on Silicon** 

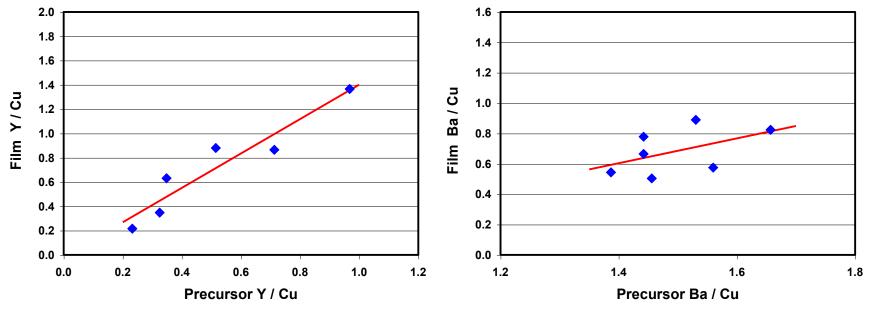
X-ray Florescence using Silver (Ag<sub> $K\alpha$ </sub>) primary beam.



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

Quantitative analysis of YBCO films is done by Rutherford Backscattering Spectroscopy (RBS).

RBS is used for calibration of the YBCO Composition relative to Precursor Concentrations.

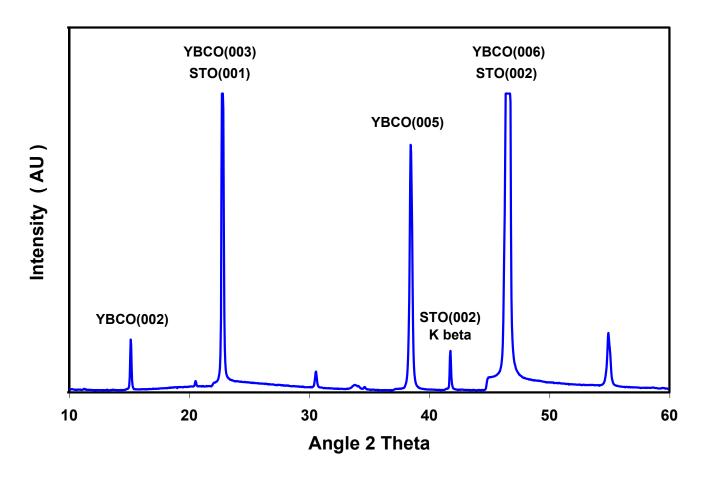


YBCO film composition determined by RBS.



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

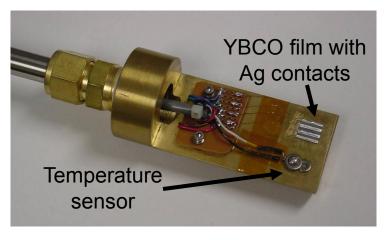
# Epitaxial YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films on SrTiO<sub>3</sub>





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# SMI developed in-house capabilities for T<sub>c</sub> and J<sub>c</sub> characterization of superconducting YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> films.



**Unpatterned YBCO film** 

Mounted for 4-point probe measurement of T<sub>c</sub>



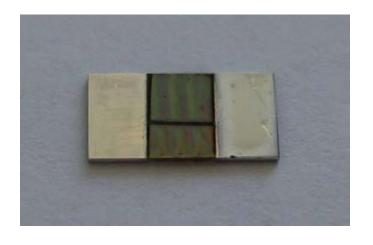
YBCO film with laser patterned microbridge

For 2-point probe measurement of J<sub>c</sub> at 77 K



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# J<sub>c</sub> Characterization of Superconducting YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films at SMI





YBCO films on 4 mm x 10 mm SrTiO<sub>3</sub> Crystal by MOCVD. Thickness  $\sim 0.5 \mu m$ .

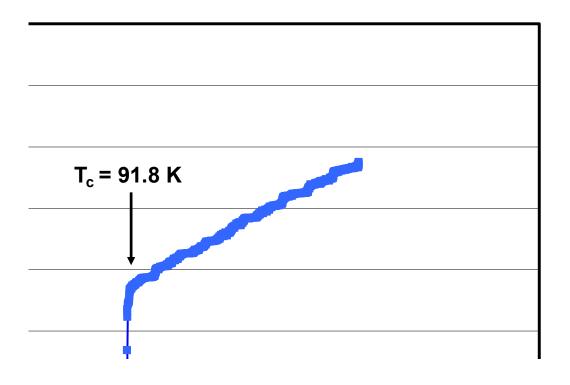
Silver contacts applied by sputtering through a shadow mask.

YBCO Patterned to ~ 250 µm wide micro-bridge by laser ablation for Jc measurements.



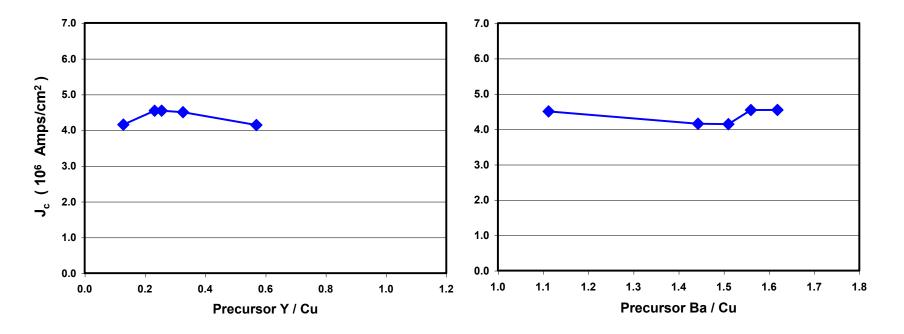
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Epitaxial YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films on SrTiO<sub>3</sub>





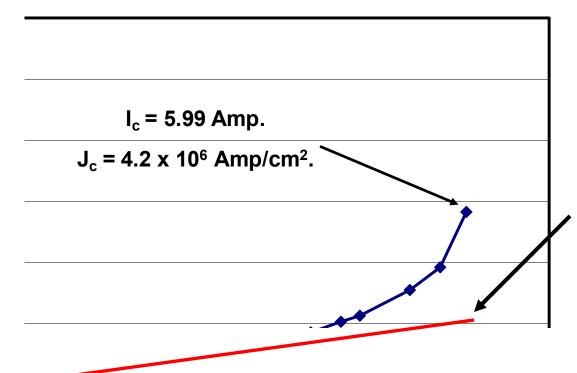
Critical current density ( J<sub>c</sub> ) is relatively insensitive to composition, for films near the  $Y_1B_2C_3O_x$  stoichiometry.



YBCO films on SrTiO<sub>3</sub> single crystals



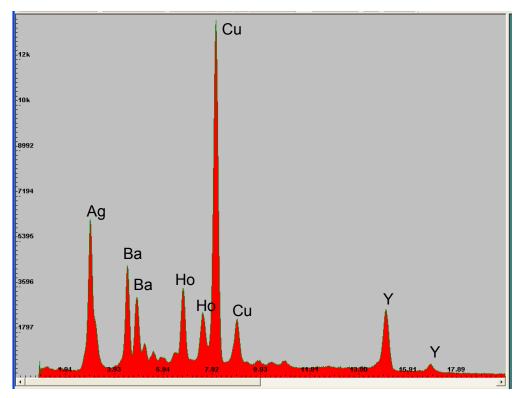
# Epitaxial YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films on SrTiO<sub>3</sub>



The resistance versus current data contains a contribution from the contact resistance, since we use a 2-point measurement.



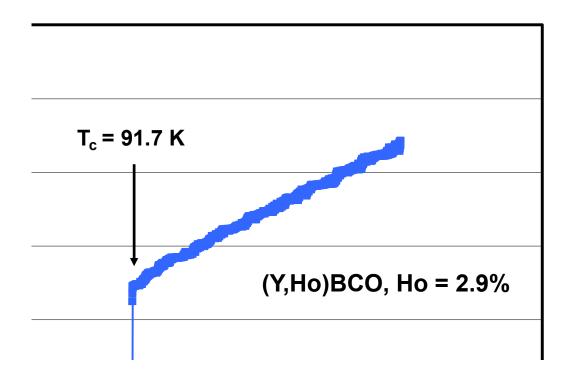
### Rare Earth (Ho) doped YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films by MOCVD



X-ray Florescence using Silver (Ag<sub>K $\alpha$ </sub>) primary beam. XRF qualifies films; RBS quantifies composition.

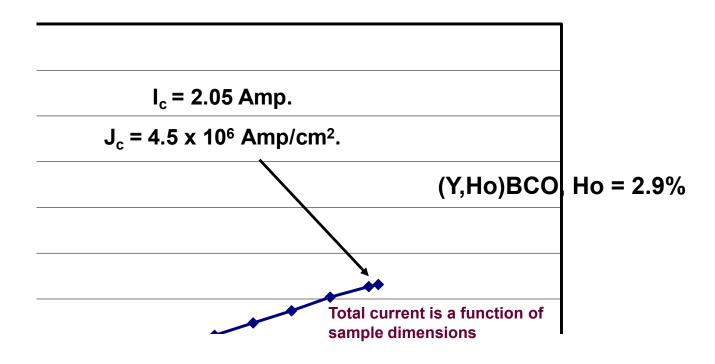


# Rare Earth (Ho) doped YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films by MOCVD



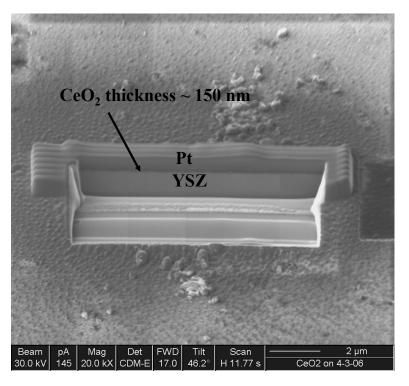


# Rare Earth doped YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> Films by MOCVD





# CeO<sub>2</sub> MOCVD on Single Crystal Substrates



Typical results for CeO <sub>2</sub> films deposited at 725 C to 800 C	
Deposition Rate	2.7 nm/minute
Deposition Efficiency [per mole of Ce(thd) <sub>4</sub> ]	840 to 930 microns/mole
Measure Index of Refraction (n)	2.20 to 2.39

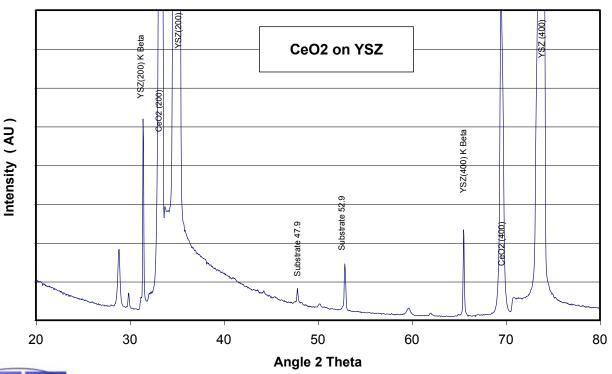
**Focused Ion Beam Microscopy Image** 



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# CeO<sub>2</sub> MOCVD on Single Crystal Substrates

Epitaxial CeO<sub>2</sub> films were deposited by MOCVD on (100) single crystal YSZ substrates at all temperatures in the range 725 C to 850 C.

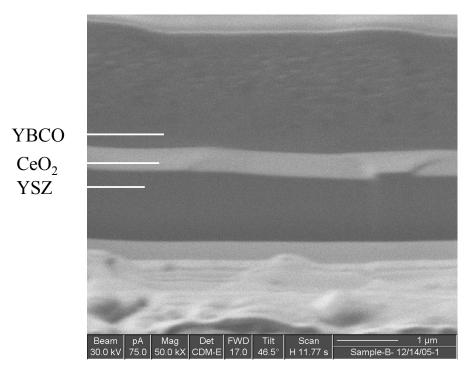


X-ray diffraction results for a CeO<sub>2</sub> film deposited on (100) YSZ by MOCVD at 725 C.

Ni filtered Cu  $K_{\alpha}$  radiation.



# YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD on CeO<sub>2</sub> Coated YSZ Substrates



**FIB Microscopy Image** 



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD on Buffer Coated Metal Tape

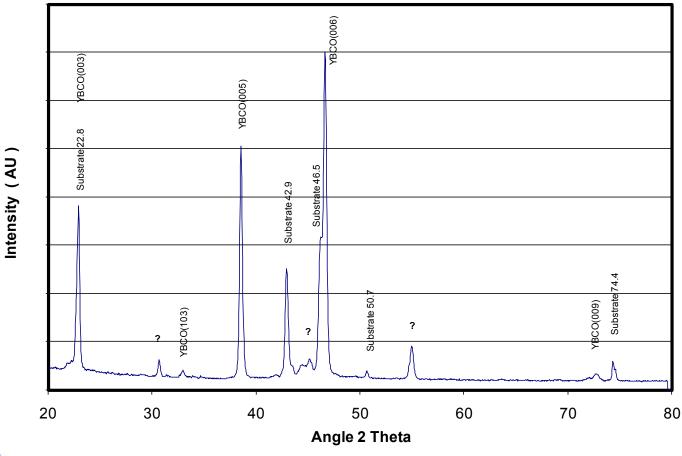


IBAD coated flexible metal tape obtained from Los Alamos National Laboratory

Initial, limited trial depositions



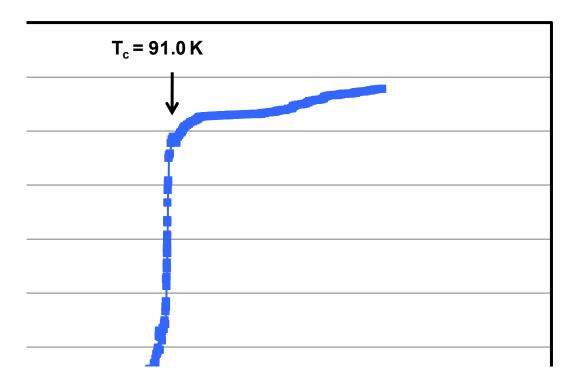
# YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD on Buffer Coated Metal Tape





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> MOCVD on Buffer Coated Metal Tape





# Intermediate Development of Reel-to-Reel MOCVD System for Kilometer Scale Coated Conductors

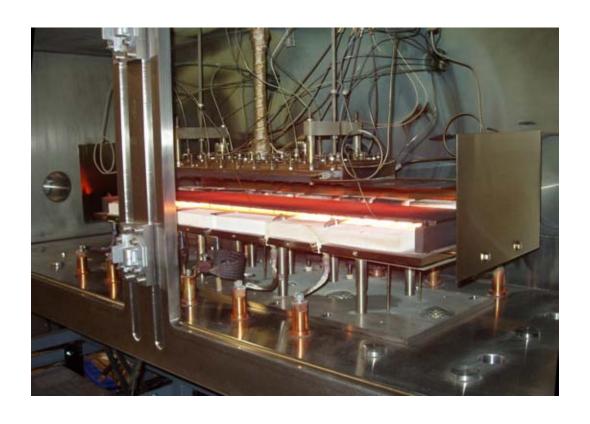
- Large chamber accommodates internal or external tape drive system.
- Expandable to multiple chambers.
- Multiple ports for additional process enhancements or process monitors.





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# MOCVD System Internals: Substrate Heater and Gas Showerhead



Substrate hot zone = 36" x 6".

Showerhead deposition area = 22" x 5".

The large area deposition zone provides for high tape throughput and for simultaneous deposition on multiple tapes.

Multiple showerheads can be installed for multi-layer films.

Internal tape drive not shown.



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# **Kilometer Scale Tape Drive System**

The tape drive system is shown mounted on a test stand. The tape drive can accommodate a single tape or multiple tapes running in parallel. A proprietary tension control system enables balanced forward or reverse tape motion.





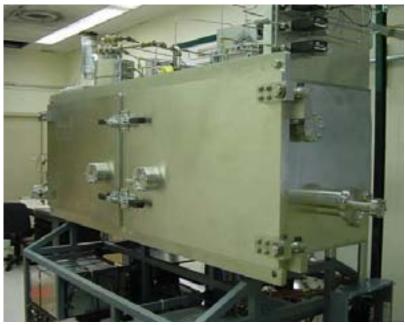


47

201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Reel-to-Reel MOCVD System Delivered and Installed at AFRL





System developed by SMI and presently installed at the Air Force Research Laboratory at WPAFB.



# MOCVD Reel-to-Reel (R2R) System for Kilometer Scale YBCO (rare earth doped) Tapes

# Major Features:

- MOCVD Reactor and optional stations (Anneal, Alt. Layer, Etc.)
- >100 meter tape reel-to-reel chambers (2) with Tensioners & Aligners (With loaders)
- Optional In-situ Monitors: XRD, XRF, RGA, others possible
- Chemical Delivery System(s); liquid flow controllers (or pumps)
- Control System & Electronics
- Exhaust System
- Framing and utilities



# MOCVD Reel-to-Reel (R2R) System for Kilometer Scale YBCO Tapes

- YBCO Deposition Station
  - Pre-heat, Post anneal and/or cool
  - 1 or More Process Zones
    - Primary YBCO 0.5m
    - Optional– Clean, CeO, Anneal, Passivation, or additional YBCO stations



# MOCVD Reel-to-Reel (R2R) System for Kilometer Scale YBCO Tapes

# Control System

- Computer to PLC Control System
- Real Time Graphical User Interface
- Automatic Control, manual override
- All Analog and Digital Inputs and Outputs Controlled
- Safety Features Interlocked
- Program/Data Logging



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Proposed Reel-to-Reel MOCVD System for Scale 100 Meter Coated Conductors

- Basic Pricing
  - Complete System

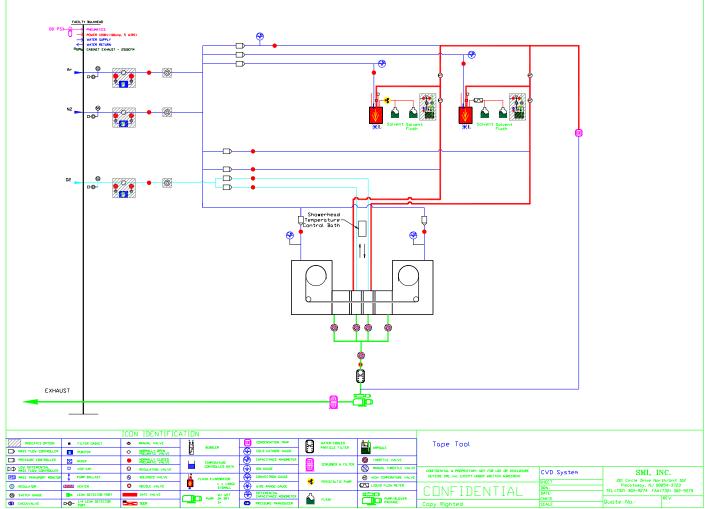
**\$\$TBD\$\$** 

- RGA option
- In-Situ XRD or XRF options
- Spare Parts Kit
- Service Agreement



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

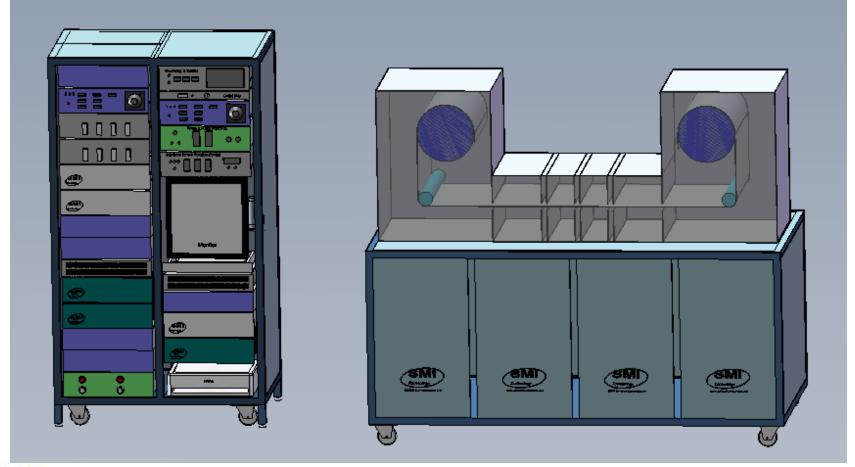
# MOCVD Gas Panel Reel-to-Reel (R2R) System for Kilometer Scale YBCO Tapes





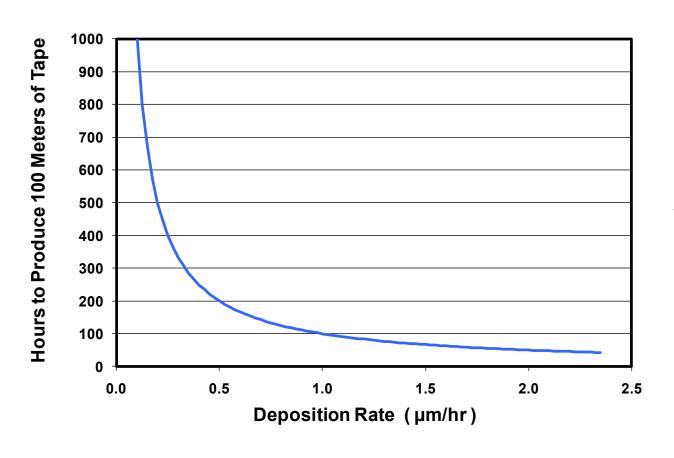
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Next Generation Reel-to-Reel (R2R) Production MOCVD System for Kilometer Scale Coated Conductors





# **Tape Production Yield Estimates**



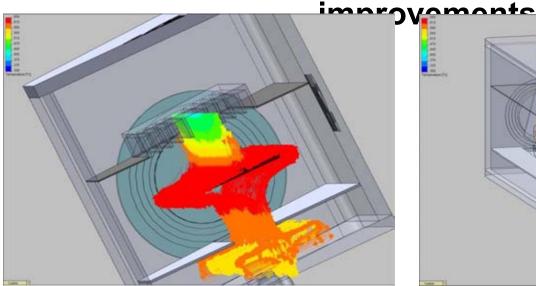
Deposition Area = 50 cm in Length.

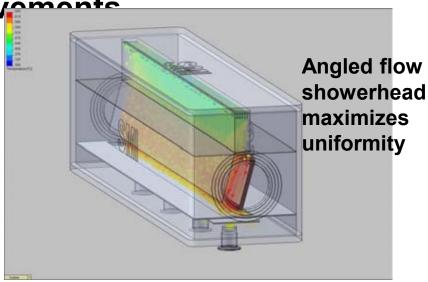
YBCO Thickness = 0.50 μm.

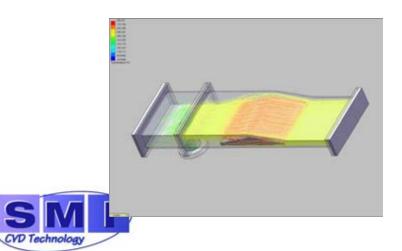


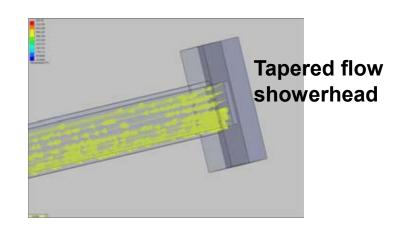
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

## Flow/Thermal and Chemical modeling support design



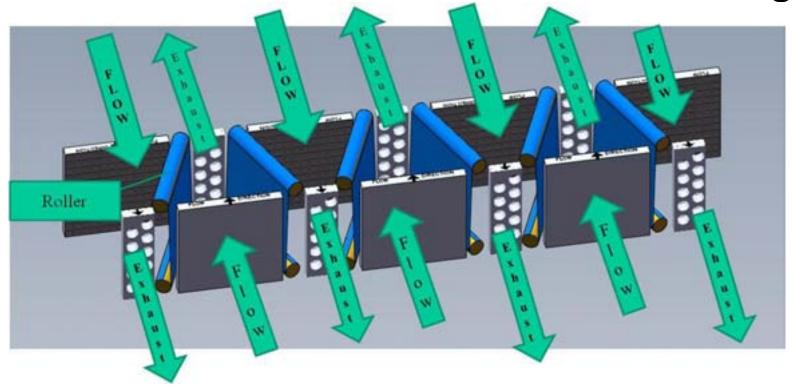






201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Patent Pending Future Production Yield Enhancement Design



Simple Patent Pending innovation can essentially halve production costs



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Some of SMI's External Design and Manufacturing Support



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

## P Subcontracted Capabilities

# Quality You Can Count On: Operating in modern facilities covering more than 100,000 square feet with stateof-the-art equipment, clean room facilities and assembly services – P utilizes all of its resources to provide OEM build to order manufacturing services for fixed and tape drive systems.







201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# **IL Subcontractor Capabilities**

## Engineering

- SolidWorks software with structural finite element analysis add-ons
- Robust CAD stations
- Significant standard part CAD model database assists in efficient and accurate design
- Document and version controls
- ERP/MRP system

## Manufacturing

- Conventional machine shop tools and staff machinists
- Assembly tables, hand tools, precision measurement tools
- Additional equipment:

TIG welder, 9000lb forklift and rigging equipment, 16' tandem axle trailer.

Relationships with many US vendors for manufacture of:

- Vacuum chambers
- Fabricated parts
  - CNC mills and lathes with very large capacity available
- Specialty brazed, welded, or heat treated parts
- Ceramics





201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# **AV Subcontractor Capabilities**

THIN FILM SOLAR (CIGS)

#### **Sputter-based layers**

- Back Electrode
- CulnGa absorber layer with proprietary co-selenization
- CdS integrated into the absorber layer system
- Window Layer
- •WEB CIGS co-sputtering technology offers the following advantages vs co-evaporation process:
  - Higher web speed
  - Easier process control
  - Less power consumption
  - Easier maintenance
  - Lower operating cost
- WEB's three-tool set has matched outputs
- •Custom web widths and number of magnetrons
  - 5 to over 30 magnetrons per system
- Common payout & take up system

#### **•SUPER CONDUCTOR MATERIAL**

#### Broad Process Expertise

- Sputtering/Reactive (Proprietary Technologies for Fast Line Speeds)
- CVD (Proprietary Injection Design)

#### Continuous Flow Processing

- Roll to Roll (Flexible Substrates)
- 5 cm to >100 cm web
- Extensive Range of Web Materials: Metals (Al, Ti, SS, Cu, etc.)
- Load locked Architecture
- Independent Process Zones
- High Throughput
- Pre-cleaning/conditioning



201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# Many Engineering services may be hired for temporary development efforts



## **Contract Engineering Services**

Engineered Support - for Innovators

All Services ▼

#### Overview

Contract Engineering Services is a unique resource. We "speak the language" of manufacturers, inventors, researchers and product developers who need electromechanical engineering support. For over 15 years, we have provided engineered solutions to a diverse clientele of businesses, inventors, and professionals working in medicine, energy, optics, advanced materials, and consumer products.

We work closely with our clients, to truly understand and clarify their problems and objectives. Then we apply our SolidWorks CAD, FEA, Rapid...

Read More »

Contract Engineering Services



Ceseng.elance.com

#### **Engineered Packaging Example**

Illustrated is a lightweight, ruggedized, Fuel Cell Power Supply designed for a Military Client. The entire case was composed of plastic components fabricated by a Rapid Prototyping process called Selective Laser Sintering (SLS). Strong, functional components can now be obtained by SLS directly from CAD files and with no tooling or molds required. Additionally, SLS can produce parts with internal cavities and other features unproducible by other methods.



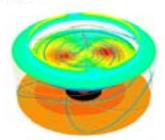
201 Circle Drive North, Unit 102/103, Piscataway, NJ 08854
Phone: 732 302-9274 Fax: 732 302-9275 www.structuredmaterials.com

# In addition to in-house Multiple MOCVD Modeling Services Available



#### Epitaxy/MOCVD

Organo metallic vapor phase epitaxy, also known as NOCVO, is a primary technique to grow thin film III-V compound semiconductors such as gallium nitride (GaN), gallium arsenide (GaAs), and indium phosphide (InP) based materials. These materials are used in devices such as light-emitting diodes, solid-state lissers, photovoltaics, IR detectors, and heterojunction bipolar transitors. MOCVO is the critical enabling technology with several advantages including highly uniform thickness, excellent repeatability, for maintenance, high throughput and low cost of ownership. Fluent's CFD software combines physically accurate transport models with proposed gas-phase and surface chemistry mechanisms, and predicts uniformity of film thickness and composition variation during selective growth.



Flow path lines, concentrations of AsK3, GaAs deposition on a substrate in a MOCVO system.

Courtesy of Emcore Corporation fluent



