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The Fifth U.S.-Japan Seminar on

## *Dielectric and Piezoelectric Ceramics*

### PROGRAM AND EVALUATIONS

General Chairman:	K. Wakino ( <i>Murata</i> )
	J. P. Dougherty ( <i>Penn State/MRL</i> )
General Vice Chairman:	T. Shiosaki ( <i>Kyoto University</i> )
<i>Program Committee</i>	
Chairman:	K. Sakata ( <i>Tokyo Sci. University</i> )
Vice Chairman:	T. Takenaka ( <i>Tokyo Sci. University</i> )
<i>Financial Committee</i>	
Chairman:	F. Hamano ( <i>Kyocera</i> )
Secretary:	M. Adachi ( <i>Toyama Pref. University</i> )
	Y. Sakabe ( <i>Murata</i> )

December 11-14, 1990

Kyo-Dai Kaikan

Kyoto, Japan

91-17575



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## U.S. Chairman's Report

### Abstract

The Fifth US-Japan Workshop on Dielectric and Piezoelectric Materials was held in Kyoto, Japan on December 11-14, 1990. The local organization was from Kyoto University and Murata Corp. U.S. organization was from the Materials Research Lab, Penn State University. There were 123 participants: 84 Japanese and 39 U.S.; 67% of the Japanese were from industry, while 38% were from U.S. industry (up 5% from the last workshop in Japan).

The technical topics most discussed were: (1) thin film ferroelectrics and dielectrics, (2) relaxor ferroelectrics, and (3) piezoelectric materials and processing. Barium titanate materials for multilayer capacitors received little attention. There were no USA papers on microwave dielectrics and no Japanese papers on single crystals.

There were no USA papers on piezoelectric devices, while many Japanese papers were device related.

Dielectric materials for electronic packaging were few from both sides.

### Participants

During the organization of this U.S.-Japan Seminar, we tried to increase the number of U.S. industry participants. With an aggressive telephone lobbying campaign, I was able to increase the U.S. industry participation in the workshop to 38% of the attendees. This increase was noted in several of the evaluations. However, comments by Gene Haerding (Clemson) and Jim Scott (Colorado) cited the even greater Japanese industry participation. The breakdown is given in Table 1.

It should be noted that in the organizing phase that I had only 1/3 of the companies that I contacted send participants. In contrast, the Japanese organizers turned away nearly as many companies as they allowed to participate!

(A listing of the workshop participants follows the evaluations.)

**TABLE 1**  
**Participants**  
**5th U.S.-Japan Seminar**  
**on**  
**Dielectric and Piezoelectric Ceramics**

	U.S.A.	JAPAN	TOTAL
University	18	26	44
Industry	15	56	71
Government	6	2	8
Total	39	84	123

**Technology Emphasis**

I have shown in Table 2 a classification matrix to help identify the technologies being emphasized. From Table 2 the significance of processing and properties is apparent; Tom Shrout (Penn State) wrote a very lucid evaluation of the processing work. This emphasis illustrates the strong Japanese commitment to materials research in ceramics. (A part of this emphasis is also due to the materials research backgrounds of many of the workshop organizers.)

**TABLE 2**

**Paper Classifications**

USA/Japan	Processing & Properties	Modeling	Devices	Reliability	Total
Crystals	2/0		1/0		3/0
Piezo	3/6	2/1	0/3	2/2	7/12
Relaxors	2/9	1/0	0/2		3/11
High K	3/5			1/0	4/5
MLC	1/3		0/0	4/3	5/6
Thin Films	6/13		1/1	1/0	8/14
Low K	1/4	1/0	1/1		3/5
Varistor PTC Pyro & Misc.	0/5		0/1		0/6
	18/45	4/1	3/8	8/5	33/59
<b>Totals</b>	<b>63</b>	<b>5</b>	<b>11</b>	<b>13</b>	<b>92</b>

Packaging dielectric ceramic material, was just barely mentioned in spite of its importance. Of particular note was the fact that work on low temperature ceramic cofire multichip modules were not presented in proportion to the amount of work I personally know is being done. This might mean that commercialization is in progress and both sides have to keep quiet.

Also of note was that there were no U.S. papers on microwave materials. (I would recommend that purchasing agents for these materials program dollar to yen conversions into their business calculators.)

**Poll of Japanese Interests**

For the first time at a U.S.: Japan Dielectric Workshop, we asked the Japanese participants to write down their assessments. Table 3 shows the form that was used.

The Japanese were initially surprised by my request, but they cooperated very well in completing the "form" I created. The results were given to R. Pohanka and S. Freiman, who completed an ancillary report.

### TABLE 3

#### 5th U.S.: Japan Seminar

Please list the most significant new ideas or results that you saw at this meeting.

[The list should be short.]

[The order of the list is not important.]

1.

2.

3.

4.

5.

6.

7.

(optional)

Name:

*The Fifth U.S.-Japan Seminar on  
Dielectric and Piezoelectric Ceramics*

**PROGRAM and ABSTRACTS**

General Chairman : K.Wakino (Murata)  
J. P. Dougherty (MRL/PSU)  
General Vice Chairman : T. Shiosaki (Kyoto Univ.)  
Program Committee :  
Chairman : K.Sakata (Tokyo Sci. Univ.)  
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Chairman : F. Hamano (Kyocera)  
Secretary : M. Adachi (Toyama Pref. Univ.)  
Y. Sakabe (Murata)

December 11-14, 1990  
Kyo-Dai Kaikan  
Kyoto, Japan



## Schedule Table

	Morning	Afternoon	Evening
Dec.11 (Tue)		15:00  Registration  <i>(CO-OP INN)</i>	18:00-21:00  Reception Cocktail Party  <i>(CO-OP INN)</i>
Dec.12 (Wed)	9:00 Registration  9:30-10:00 Opening Remarks  10:00-12:00 Overviews  <i>(Kyo-Dai Kaikan)</i>	13:30-17:00  Poster Session I  <i>(Kyo-Dai Kaikan)</i>	17:30  Bus Tour  <i>(Kyoto City)</i>
Dec.13 (Thu)	9:15-12:30  Poster Session II  <i>(Kyo-Dai Kaikan)</i>	13:30-17:00  Poster Session III  <i>(Kyo-Dai Kaikan)</i>	19:00  Party (Courtesy of DuPont Japan)  <i>(CO-OP INN)</i>
Dec.14 (Fri)	9:15-12:30  Poster Session IV  <i>(Kyo-Dai Kaikan)</i>	12:30-14:00 Ferewell Party  14:00-15:00 Committee Meeting  <i>(Kyo-Dai Kaikan)</i>	

# PROGRAM

## Wednesday Morning / Dec. 12

### 9:30- 10:00 Opening Remarks

K. Wakino (Murata Mfg. Co., Ltd.)  
J. P. Dougherty (Pennsylvania State University)

### 10:00-12:00 Overviews

Chairman T. Shiosaki (Kyoto University)  
S. K. Dey (Arizona State University)

### Plenary Lecture

- P-1 "Physics and Engineering of Ferroelectric Memories."  
J. F. Scott (University of Colorado)
- P-2 "Chemical Processing of Electronics Ceramics."  
S. Hirano (Nagoya University)

## Wednesday Afternoon / Dec. 12

### 13:30-17:00 Poster Session I:

#### Dielectric Ceramics and Applications (23)

Chairman I. Burn (E. I. du Pont de Nemours & Co., Inc.)  
N. Ichinose (Waseda University)

- I-1 "Phase Distribution and Dielectric Properties of Lead Magnesium Niobate-Pyrochlore Diphasic Mixtures" J. Chen and M. P. Harmer (Lehigh University)
- I-2 "La-Pb Partition between Perovskite and Pyrochlore Phases"  
T. Yamaguchi and M. Saitoh (Research & Development Center, Mitsubishi Mining & Cement Co., Ltd.)
- I-3 "The Glassy Nature of Relaxor Ferroelectrics"  
L. E. Cross, D. Viehland, S. Jang and M. Wutting (Pennsylvania State University)
- I-4 "Interaction between Relaxor and Glass in the Low-Temperature Fireable Ceramics"  
K. Onda, S. Ohtomo and S. Nishigaki (Narumi China Co.)
- I-5 "MLC's with Copper Electrodes for High Frequency Applications"  
I. Burn and W. C. Porter (Du Pont Electronics)
- I-6 "Multilayer Ceramic Capacitor with Ni Electrodes"  
H. Chazono, H. Kishi and N. Yamaoka (Taiyo Yuden Co., Ltd.)
- I-7 "A Study on the Degradation of Ni Electrode Ceramic Chip Capacitors"  
T. Nomura, S. Sumita, Y. Nakano and K. Nishiyama (Materials Research Center, TDK Co.)
- I-8 "Y5V Capacitor Based on Hydrothermally Prepared Barium Titanate Zirconate Solid Solution Powder" T. Shikida, K. Hidaka, K. Fukai, M. Aoki and K. Abe (Sakai Chemical Industry Co., Ltd.)
- I-9 "Lead Perovskite Relaxor based Low Loss Ceramic Dielectric for High Voltage Ceramic Capacitors" O. Funakawa, Y. Yamashita and M. Harata (Research and Development Center, Toshiba Co.)

- I-10 "Y5U Multilayer Ceramic Capacitors with High Specific Capacitance"  
T. Mori, A. Ochi, M. Nakanishi, K. Utsumi, S. Abe\* and T. Yoshimoto (NEC Co., \*Nippon Soda Co., Ltd.)
- I-11 "Dielectric Properties of  $(\text{Pb}_{1-x}\text{Ba}_x)(\text{Mg}_{1/3}\text{Nb}_{2/3})_{1-y}\text{Ti}_y\text{O}_3$  Ceramics"  
S. Tanaka, Y. Matsushita, K. Maeda and K. Takahashi (Hitachi Res. Lab., Hitachi Ltd.)
- I-12 "Chemical and Physical Effects on the Aging of FMN Relaxor Materials"  
M. S. Chu and J. E. Carminati (TAM Ceramics Inc.)
- I-13 "Microstructure and Dielectric Properties of Lead-Based MLC Fabricated by Alternative Lamination of 2 Kinds of Dielectric Layers"  
H. Igarashi, S. Tashiro and Y. Mizukami (The National Defense Academy)
- I-14 "Oxidation and Reduction of Pd in the Presence of Ag"  
J. P. Dougherty, S. F. Wang and W. Huebner (Materials Research Laboratory, Pennsylvania State University)
- I-15 "Unusual Dielectric Anomalies in  $(\text{Pb},\text{La})\text{TiO}_3$  Ceramics"  
M. Kuwabara, K. Goda and K. Oshima (Kyushu Institute of Technology)
- I-16 "Influence of Chemisorbed Oxygen to the PTCR and VDR Effects in Perovskite Pceramics"  
N. Ichinose, Y. Nakano and T. Takahashi (Waseda University)
- I-17 "Microstructure of High-Purity, Fast-Fired, Near-Stoichiometric Barium Titanate"  
W. A. Schulze and R.C. VanAlstine (New York State College of Ceramics)
- I-18 "A Study on Non-Oxide Ceramics for Electronic High-Performance Applications"  
H. Nakahara, C. H. Pai, K. Koumoto and H. Yanagida (University of Tokyo)
- I-19 "SiC as a Dielectric Ceramic" G. R. Love and T.K. Gupta (Alcoa Technical Center)
- I-20 "Dielectric Properties of Nonstoichiometric Barium Zinc Niobium Tantalate Ceramics"  
H. Ohuchi (Shonan Institute of Technology)
- I-21 "Dielectric Properties and Micro-Structure of  $(\text{Sr},\text{Ca})_3\text{Ti}_2\text{O}_7$  Ceramics"  
A. Yamada, T. Honda, Y. Utsumi and H. Watarai (Mitsubishi Electric Co.)
- I-22 "Microwave Characteristics of BaO-TiO<sub>2</sub>-Rare Earth Oxide Ceramics"  
K. Fukuda, A. Takasugi, M. Fujinaga and R. Kitoh (Ube Industries Ltd.)
- I-23 "Third Harmonic Distortion of Dielectric Resonator Materials"  
K. Wakino, Y. Ishikawa, T. Nishikawa and H. Tamura (Murata Mfg. Co., Ltd.)

## Thursday Morning / Dec. 13

### 9:15-12:30 Poster Session II:

#### Reliability of MLC's, Substrates and Semiconductors (12)

Chairman A. S. Bhalla (Penn State University.)

Y. Sakabe (Murata Mfg. Co., Ltd.)

- II-1 "Surge Test Characteristics of High K Multilayer Ceramic Capacitors"  
H. C. Ling and D. D. Chang (AT & T Bell Laboratories)
- II-2 "Use of Highly Accelerated Life Test (HALT) to Determine Reliability of Multilayer Ceramic Capacitors" J. P. Canner and R. J. Confer (Murata Erie N. A., Inc.)
- II-3 "Accelerated Life Tests of Multilayer Ceramic Capacitors with Thin Dielectric Layers"  
G. H. Maher, T. I. Prokopowicz and G. A. Shim (MRA Laboratories Inc.)

- II-4 "Role of Minor Phase Additions on Aging and Dielectric Stability Characteristics in BaTiO<sub>3</sub>"  
R. C. Buchanan, T.R. Armstrong and R.D. Roseman (University of Illinois)
- II-5 "Acoustically Aided Board Flexure Testing of Surface Mount Capacitors"  
K. Ritchie, C. Nies and T. Rutt (AVX Corp.)
- II-6 "Control of Mass Transport and the Attendant Point Defects - The Next Generation of Reliability in Dielectrics" N. G. Eror (University of Pittsburgh)
- II-7 "Cordierite Glass-Ceramic Substrate for IBM's Mainframe Computers" R. R. Tummala (IBM)
- II-8 "The Reaction between AlN Ceramics and Ti Film after High Temperature Treatment"  
T. Yasumoto, K. Yamakawa, N. Iwase and N. Shinozawa\* (R & D Center, \*New Materials Engineering Lab., Toshiba Co.)
- II-9 "Interaction between Glass and CaTiO<sub>3</sub> Ceramics" T. Yamaguchi and H. Ayaki (Keio University)
- II-10 "Interfaces of Electronic Ceramics / Electrodes and Wetting Properties"  
S. Sugihara and K. Okazaki (Shonan Institute of Technology)
- II-11 "Formation Mechanism of Potential Barrier of ZnO Varistor" S. Shirasaki, J. Tanaka, H. Haneda, S. Hishita and K. Sato (National Institute for Research in Inorganic Materials)
- II-12 "Relationship between Degradation Phenomenon and Trap Levels in ZnO Varistor"  
M. Takata, Y. Watanabe, K. Nakajima and S. Shichino (Nagaoka University of Technology)
- II-13 "Fabrication of Jointed Type PTC Ceramics with Different Composition and Their Evaluation"  
T. Yamamoto, S. Ohashi and S. Tomono (National Defense Academy)

### Processing (11)

Chairman T. Yamaguchi (Keio Univ.)

W. A. Schulze (New York State College of Ceramics)

- II-14 "Sol-Gel Processing of Pb(Mg,Nb)O<sub>3</sub> Dielectrics"  
K. Kikuta, W. Sakamoto and S. Hirano (Nagoya University)
- II-15 "Fabrication of Porous Electroceramic Structures by Reactive Calcination"  
T. R. Shrout, Y. S. Kim and J. Fielding, Jr (Materials Research Laboratory, Pennsylvania State University)
- II-16 "Mechanism of PNN Based Perovskite Ceramics Formation"  
T. Yoshimoto, Y. Sasaki and A. Nagai (Odawara Research Center, Nippon Soda Co., Ltd.)
- II-17 "Application of Attrition-Milling to Lead-based Relaxor" J. Kato, H. Kagata and Y. Yokotani\*  
 (Materials & Devices Res. Lab., \*Central Res. Lab., Matsushita Electric Ind. Co., Ltd.)
- II-18 "Chemical Precipitation and Coating Processes for Synthesis of Next Generation Dielectric Powders"  
D. K. Swanson, S. A. Bruno, I. Burn, K. Sasaki, H.E. Bergna and P.J. Jesson (E. I. du Pont Co., Inc.)
- II-19 "Synthesis of Metal-Vetivenoxide Compounds and Its Application for Thin Films and Powders"  
K. Hayashi (Research and Development Lab., Shoen Chemical Inc.)
- II-20 "The Growth and Characterization of Tungsten Bronze Sr<sub>x</sub>Ba<sub>1-x</sub>Nb<sub>2</sub>O<sub>6</sub> Single Crystal Fibers"  
A. S. Bhalla and J. K. Yamamoto (Materials Research Laboratory, Pennsylvania State University)
- II-21 "Sol-Gel Processing of Ferroelectric Fibers" S. C. Choi\*, S. I. Aoki, M. Miyayama, D. A. Payne\*\*  
 and H. Yanagida (RCAST, University of Tokyo, \*Ajou University, \*\* University of Illinois)

- II-22 "Processing of Stoichiometric and Ti Doped LiNbO<sub>3</sub> Films with Preferred Orientation from Metal Alkoxides" S. Hirano, K. Kikuta and K. Kato (Nagoya University)
- II-23 "A New Method for the Preparation of A<sup>a+</sup>{(B<sub>Ix</sub><sup>b+</sup>, B<sub>II1-x</sub><sup>b+</sup>)<sub>n</sub> B<sub>III1-n</sub><sup>c+</sup>} O<sub>3</sub> (a+b(1-n)+cn=6) Type Solid Solutions" K. Kakegawa (Faculty of Engineering, Chiba University)
- II-24 "Solubility and Phase Stability of Barium Titanate in Aqueous Suspension" James H. Adair, Benjamin L. Utech, K. Osseo-Asare and Joseph P. Dougherty (Pennsylvania State University)

## Thursday Afternoon / Dec. 13

### 13:30-17:00 Poster Session III

#### Piezoelectric Ceramics and Applications (16)

Chairman H. Banno (NGK)

R. Y. Ting (U. S. Naval Research Laboratory)

- III-1 "Temperature Dependence of Anisotropy in Piezoelectric Properties of Modified Lead Titanate Ceramics" T. R. Gururaja and L. E. Cross\* (Hewlett Packard, \*MRL, Pennsylvania State University)
- III-2 "Effects of Silica on the Microstructure and Electrical Properties of Lead Zirconate Titanate" A. H. Gabor, B. G. Koepke, J. C. Kyonka and F. C. Wallenhorst ( Honeywell, Inc. )
- III-3 "Reliability-based Consideration on the Fatigue Degradation Process of Piezoelectric Ceramics" T. Tanimoto and K. Okazaki (Shonan Institute of Technology)
- III-4 "Modeling and Optimization of Piezoelectric Composites" B. A. Auld and J. A. Hossack (E. L. Ginzton Laboratory, Stanford University)
- III-5 "A New Flexible Piezoelectric Composite Material: Woven PZT Ceramic Fiber/Polymer Composites" A. Safari and D. J. Waller (Rutgers-The State University of New Jersey)
- III-6 "Piezoelectric Properties of 0-3 Composite of Polymer and Ceramic Powder Mixture of PZT and PbTiO<sub>3</sub>" H. Banno and K. Ogura (NTK Technical Ceramics Div., NGK Spark Plug Co., Ltd.)
- III-7 "Modeling 1-3 Piezocomposites -- The Simple Perspective" W. A. Smith (Office of Naval Research, Materials Division)
- III-8 "Hydrophone Characteristics of Porous PZT Ceramics" H. Sato, K. Ayusawa, T. Arai, K. Kawamura, T. Miyata and K. Kobayashi (Oki Electric Ind. Co., Ltd.)
- III-9 "Effect of Thermal Aging on the Piezoelectric Properties of PVDF and Piezocomposites" R. Y. Ting (U. S. Naval Research Laboratory)
- III-10 "Preparation of Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-PbTiO<sub>3</sub> (PMN-PT) Relaxors and Evaluation of Their Electrostrictive Properties" T. Ando, R. Suyama K. Tanemoto (Nippon Steel Co.)
- III-11 "Electrostrictive Materials for Ultrasonic Probes (I): Material Properties" H. Takeuchi, H. Masuzawa, Y. Ito and S. Jyoumura\* (Central Research Laboratory, Hitachi Ltd., \*Magnetic & Electronic Materials Research Laboratory, Hitachi Metals Ltd.)
- III-12 "Electrostrictive Materials for Ultrasonic Probes (II): Applications" H. Masuzawa, C. Nakaya and H. Takeuchi (Central Research Lab., Hitachi Ltd.)
- III-13 "Numerical Calculation of Dielectric and Piezoelectric Constants and 3-dimensional Vibration Simulation of Piezoelectric Device" M. Nishimura and D. A. Sagala (Central Research Lab., Kyocera Co.)

- III-14 "Phase Relations in the  $(\text{Bi}_{0.5}\text{Na}_{0.5})\text{TiO}_3\text{-PbTiO}_3$  Solid Solution Ceramics and Its Piezoelectric Properties" K. Sakata, T. Takenaka and Y. Naitou (Science University of Tokyo)
- III-15 "A High Sensitivity IR Detector Using Modified PZT Ceramics"  
J. Lian and T. Shiosaki (Faculty of Engineering, Kyoto University)
- III-16 "Effects of Partial Oxygen Pressure During Heat Treatment of Multilayer PZT Ceramics"  
M. Kahn (Naval Research Laboratory)

### Actuator, Mechanical Properties (7)

Chairman S. W. Freiman (NIST)

H. Igarashi (National Defense Academy)

- III-17 "A Piezoelectric-Ceramic Hollow-Cylinder Torsional Vibrator"  
H. Shimizu and T. Yoshida\* (University of Electro-Communications, \*Tokin Corporation)
- III-18 "Auto-Focusing System Utilizing Electrostrictive Actuators"  
R. Suyama, K. Tanemoto, Y. Kobayashi\* and M. Yajima\* (Nippon Steel Co., \*Sankyo Seiki Mfg.)
- III-19 "A Small-Size Ultrasonic Linear Motor" J. Toyoda and K. Murano (Sony Corporate Research Lab.)
- III-20 "Piezoelectric Actuator Properties of  $\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-KNbO}_3\text{-PZT}$  Ceramics"  
T. Takenaka, M. Satou and K. Sakata (Science University of Tokyo)
- III-21 "A Quantitative Theory of Microstructure in Polycrystalline Ceramics: Application to Calculation of the Effective Elastic Constants and Internal Stresses"  
S. K. Kurtz, S. Kumar, J. R. Banavar, K. Kunz, D. Steich, S. S. Chang, M. Leffler and M. Cieplak\* (Penn State University, \*Polish Academy of Science)
- III-22 "Effects of Poling on the Mechanical Properties of Piezoelectric Ceramics"  
H. Yamaoka and Y. Ushida (Mitsui Petrochemical Industries)
- III-23 "Fracture Behavior of Cyclically Loaded Lead-Zirconate-Titanate (PZT)"  
G. S. White, A. S. Raynes and S. W. Freiman (National Institute of Standards and Technology)

### Friday Morning/ Dec. 14

9:15- 12:30 Poster Session IV:

#### Dielectric and Ferroelectric Thin Films, Electrooptics (21)

Chairman I. Ueda (Matsushita Electric Industrial Co.)

R. R. Neurgaonkar (Rockwell Science Center)

- IV-1 "Electrical Degradation Mechanisms in Ferroelectric Thin Film Memories"  
D. M. Smyth (Lehigh University)
- IV-2 " $\text{Ta}_2\text{O}_5$  Thin-Film Capacitor for Ultra High Frequencies"  
S. Yamanaka and H. Yoshino (Sumitomo Electric Industries, Ltd.)
- IV-3 "Electrical Characterization of Ferroelectric Thin-Film Capacitors"  
P. J. Schuele and S. D. Traynor (Ramtron Co.)

- IV-4 "Advances in Perovskite Thin-Films: Chemical Processing, Properties, and Electrical Applications" S. K. Dey (Arizona State University)
- IV-5 "Preparation of  $\text{PbTiO}_3\text{-La}_2\text{O}_3$  System Thin Films by Sol-Gel Processing"  
T. Okamura, H. Nagata, M. Watanabe, N. Sakai and H. Yamamura (Tosoh Corporation)
- IV-6 "Preparation of  $\text{Pb}_x\text{M}_{1-x}\text{TiO}_3$  (M= Sr, Ba) Thin Films by the Thermal Decomposition of Organometallic Compounds"  
K. Saegusa, M. Sasaki and K. Yamada (Tsukuba Research Lab., Sumitomo Chemical Co.)
- IV-7 "Synthesis of  $\text{SrTiO}_3$  and  $\text{CaTiO}_3$  Thin Films by Hydrothermal Electrochemical Method"  
Y. Sakabe, Y. Hamaji, M. Hayashi, Y. Ogino, N. Ishizawa\* and M. Yoshimura\* (Murata Mfg. Co., Ltd., \*Research Laboratory of Engineering Materials, Tokyo Institute of Technology)
- IV-8 "Effect of Pulsed Laser Irradiation in Preparation of  $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$  Films by Laser Ablation"  
T. Minamikawa, Y. Yonezawa, K. Segawa, A. Morimoto\*, T. Shimizu\*, S. Otsubo\*\*, H. Kidoh\*\*\* and T. Ogawa\*\*\* (Industrial Research Institute of Ishikawa, \*Kanazawa University, \*\*Shibuya Co., Ltd., \*\*\*Murata Mfg. Co., Ltd.)
- IV-9 "Thin Film Growth of  $\text{PbTiO}_3$  by Photo-MOCVD"  
M. Shimizu, T. Katayama and T. Shiosaki (Faculty of Engineering, Kyoto University)
- IV-10 " $\text{PbTiO}_3$  Thin Films by Chemical Beam Deposition"  
A. I. Kingon, K.Y.Hsieh, L. L. H. King and S-H. Rou (North Carolina State University)
- IV-11 "Preparation and Dielectric Properties of Ferroelectric  $\text{PbTiZrO}_3$ ,  $\text{BaTiO}_3$  and  $\text{SrTiO}_3$ "  
I. Ueda, K. Iijima and K. Kugimiya (Central Res. Lab., Matsushita Electric Industrial Co., Ltd.)
- IV-12 " $\text{PbTiO}_3$  Thin Film Growth by Multi-Metal Target Sputtering"  
K. Terao, S.P. Leiphart\* and S. B. Krupanidhi\* (Sumitomo Metal Industries, Ltd., \*Penn State University)
- IV-13 " $\text{Pb}(\text{Zr,Ti})\text{O}_3$  and  $\text{KNbO}_3$  Thin Films by Ion Beam Sputter Deposition"  
A. I. Kingon, T.M. Graettinger, S-H. Rou, H. N. Al-Shareef, K. D. Gifford, P. D. Hren, M. S. Ameen and O. H. Auciello\* (North Carolina State University, \*Also Microelectronics Center of North Carolina)
- IV-14 "Polar Axis Oriented Ferroelectric  $\text{Pb}(\text{Zr}_{0.9}\text{Ti}_{0.1})\text{O}_3$  Film by Sputtering"  
T. Shiosaki and T. Okamura\* (Faculty of Engineering, Kyoto University, \*Kyocera Corporation)
- IV-15 "Preparation of C-Axis Oriented  $\text{AlN}$  Thin Films by Low-Temperature Reactive Sputtering"  
H. Okano, K. Shibata and S. Nakano (Functional Materials Res. Center, Sanyo Electric Co., Ltd.)
- IV-16 "Properties of  $\text{Bi}_4\text{Ti}_3\text{O}_{12}$  films by ECR Plasma Sputtering Deposition" Y. Masuda, A. Baba, H. Masumoto\*, T. Goto\*, M. Minakata\*\* and T. Hirai\* (Hachinohe Institute of Technology, \*Institute for Material Research, Tohoku University, \*\*Institute of Electrical Communication of Tohoku University)
- IV-17 "Linear and Nonlinear Optical Properties of Tungsten Bronze Crystals"  
R. R. Neurgaonkar, W.K. Cory, J. R. Oliver and Edward J. Sharp\* (Rockwell International Science Center, \*Center for Night Vision and Electro-Optics)
- IV-18 "Tungsten Bronze Crystals for Photorefractive Applications"  
L. N. Durvasula (DARPA DSO)
- IV-19 "Selected Properties of Acetate-Derived PLZT Thin/Thick Films"  
G. Haertling (Clemson University)
- IV-20 "Properties of Electro-Optic PLZT Ceramics Fabricated by Partial Coprecipitation Method"  
K. Nagata, T. Kiyota\* and M. Furuno\* (The National Defense Academy, \*Tamura Corporation)
- IV-21 "Excimer Laser Ablated PZT Films"  
S. B. Krupanidhi, D. Roy and J.P. Dougherty (Materials Research Laboratory)

# EVALUATIONS

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## U.S.:JAPAN SEMINAR ON DIELECTRIC AND PIEZOELECTRIC CERAMICS

December 11-14, 1990  
Kyoto University  
Kyoto, Japan

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### BACKGROUND

In addition to the conference in Kyoto, universities and companies visited in Japan included the following:

- Shonan Institute of Technology, Fujisawa
- Nippon Soda, Odawara
- NEC/R&D, Kawasaki
- Toshiba/R&D, Kawasaki
- Marcon Electronics, Nagai

Along with representatives from the above, Japanese participants at the U.S.:Japan meeting included affiliates from numerous Universities\* (~ 15) and industrial companies (~ 24). This is in reverse contrast to the U.S. participants at the seminar, 23 and 16 for universities and industry, respectively.

### RESEARCH TOPICS (DIRECTIONS)

The topic areas presented below have been broken down into material families and whether in bulk or thin film form.

#### Relaxors

In the area of relaxor ferroelectrics a considerable amount of effort in R&D is being performed. From Table I, the fundamental work is being performed at U.S. universities (namely Penn State) whereas the work in Japan is primarily related to processing and applications such as MLCs and actuators. Much of the processing is performed at chemical companies, e.g., Nippon Soda, Mitsubishi Materials, Narumi China, as well as large MLC related companies, NEC, Toshiba, Marcon, Matsushita, etc. Processing work was related to optimum mixing, etc., using state-of-the-art milling (attrition) and wet-chemical (alkoxide) methods. Also, much of the work on relaxors was in reality redundant in that the formulations studied are obviously selected on the premise to avoid patent infringement. It is interesting to point out that now 6 M/MLC manufacturers, including multilayer actuators, are currently in production of relaxor-based materials.

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\* Encompassed with universities are government and related R&D facilities.

**TABLE I**  
**Materials, # of Papers as Related to U.S. and Japan Interests**

Topics	BULK		THIN FILM	
	U.S.	Japan	U.S.	Japan
Relaxor Ferroelectrics	3 [F,P]	14 [P,A]	---	1
BaTiO <sub>3</sub> and Related	10 [F,P,A]	2 [F,P,A]		1
PZT/PT and Related	10 [F,P,A]	14 [I,F,P,A]	8	6
Sensors (PTCR/Varistors)	---	3 [I,F,P]		
Microwave	---	4		1
Packaging	2 [A]	1-2 [I,P]		
Tungsten Bronze and Crystals	2 [F,A]	1 [P]		---
Miscellaneous	(LiNbO <sub>3</sub> ) 1	(Ta <sub>2</sub> O <sub>5</sub> )		1 [I,P]
Reliability	3 [F]			
General	[F]			
	(TiNTiO <sub>2</sub> ) and Bismuth Titanate			1

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I—Innovative; F—Fundamental; P—Processing; A—Applications

They are in order of chronological implementation:

- TDK
- NEC
- Mitsubishi Materials
- Marcon (Toshiba)
- Murata
- Nippon Soda (powder manufacturing only)

Generally, the relaxor formulations are based on PMN. The market is reportedly about the same as Ni-electroded MLCs and only includes large capacitance parts ( $\geq 10 \mu\text{f}$ ).

To date, little has been reported on reliability of relaxors, though the Japanese are strongly interested in this area. They feel reliability is the key to success and not performance.

Only a minimal amount of work in relaxor thin films has been reported. They appear to be good for capacitor type devices and perhaps electrostrictive micro-motors.

### **BaTiO<sub>3</sub> and Related Materials**

A considerable amount of work on BaTiO<sub>3</sub> and related materials comes from the U.S. Fundamentally, electrode interfaces and defect chemistry is being examined at both universities and industries. Perhaps, the best work comes in processing. DuPont utilizes a chemical precipitation and coating process to minimize the level of required fluxes/additives, resulting in enhanced dielectric behavior and overall performance.

In Japan, very good fundamental work on electrode "wetting" comes from the Shonan Institute of Technology. Processing: the Sakai Chemical Company's hydrothermally derived material is attractive and commercially available.

### **Packaging**

Interestingly, the papers from U.S. companies (no universities) were applied presenting new SiC (ALCOA) and established cordierite-glass (low K—IBM) substrate materials. Work in Japan is heavy on AlN (high thermal conductivity) with Sony doing AlN thin films by reactive sputtering. Interface AlN/Ti studies are being done at Toshiba.

### **Microwave Dielectrics**

No work was reported by U.S. participants with most of the research being reported by Japanese industries, Murata, Mitsubishi, and UBE. Nothing significant was reported.

### **Sensors (PTCR/Varistors)**

Good fundamental work on ZnO varistors and PTCR ceramics came from the Japanese universities of the Nagaoka University and Tsukuba (Science) City (ZnO)-defect chemistry. Again, no U.S. participants presented work in these areas. Though varistors represent a significant market in Japan and the U.S., no R&D from associated industries was reported.

### **Thin Films**

Perhaps the best work in PZTs comes in the area of thin films. The U.S. and Japan are quite active in this area. Both have developed state-of-the-art techniques with emphasis on grain orientation. Excellent work comes from Colorado, Ramtron, Penn State, N.C. State, and Arizona.

More work, however, in the area of domain theory, stress effects, interface effects, is still needed. This is especially true in Japan where less fundamental work has been done. Thin films is probably the only area where I feel the Japanese are behind the U.S. in development.

### **Thin Films (Miscellaneous)**

In terms of practical importance, the Ta<sub>2</sub>O<sub>5</sub> thin film work by Sumitoma Industries may surpass any potential for using perovskites particularly for high speed decoupling capacitors where high capacitances are not required.

In thin films, the novel hydrothermal method performed by Murata is very interesting for both capacitors and perhaps varistors.

### **PZT/PT-Piezos/Pyros**

The U.S. and Japan are quite active in the area of PZT and PT related materials. However, most of the work presented by U.S. participants was outdated/redundant and not significant. The processing of PZT fibers may have some merit. In Japan much of the same is true. Some good work in mechanical properties comes from the Defense Academy. Kyocera's transducer modeling by finite element analysis was also of importance.

In terms of piezo composites, good fundamental work comes from Stanford.

In applications, some new designs were introduced by Japanese companies, including the cylindrical actuator from the University of Electro-Communications and Tokin and linear motors by Sony.

New non-Pb-based piezoelectrics developed at the Science University of Tokyo appear interesting, but the argument that they are non-PbO and hence, non-toxic, is totally a misconception, since bismuth is in the same EPA class as PbO.

Fabrication of very large optically transparent PLZT using tape casting and optimum chemically derived powders was a fine piece of work from the Japanese Defense Academy.

### **Miscellaneous**

The paper by Kurtz, et al. (Penn State) on ceramic simulation was fundamental and of significant importance for the understanding of elastic behavior in polycrystalline materials.

### **SUMMARY OF MEETING**

- Little innovative or breakthrough science was presented.
- U.S. more fundamental than Japan (universities only).
- Japan—excellent in processing to insure reliability. Applied R&D at both the university and industrial level.
- Most promising Japanese university—Shonan Institute, a recently founded MRL for electronic ceramics. Most universities have insufficient staffs to be effective. This includes the U.S. universities.
- U.S. is ahead in thin films, particularly in the fundamental understanding.
- Japan good at developing devices, e.g., piezo motors, actuators.
- Much of what was presented was redundant and old. Perhaps, industrial people cannot present up to date work. This is no excuse for university representatives.
- Meeting had excellent U.S.:Japan interaction.

### **Shonan Institute of Technology**

Newly found MRL for electronic ceramics. Have university:industry meetings every two months. More than 50 industrial companies and other institutes are invited.

#### Topics Include:

- Mechanical properties of composites including ferroelectrics.
- Metal-glass-ceramic interactions.
- Crystal growth.

- HIP and HUP.
- RF sputtering of films.
- Piezo composites for hydrophones.
- Nano heterocoagulation/ceramic processing.
- Surface characterization (ESCA).
- Defect chemistry of electronic ceramics (ZnO, PTCR).

Approximately 8-10 associate and full professors. State-of-the-art processing, characterization, testing, etc. equipment. This institute will be a strong force in electronic ceramics in Japan. Conceptually, U.S. universities need a certain size of disciplinary effort to perform innovative and high quality research.

#### Nippon Soda

A chemical company who diversified in the manufacturing of electro-ceramic powders, namely relaxors.

- Chemical method using alkoxides as well as state-of-the-art chemical processing equipment.
- No U.S. company making relaxors by chemical methods.(?)
- The powder is not fundamentally superior, nor highly advanced. However, the powder meets the Japanese requirement of obtaining high reliability.
- Approximately 2-3 man years of effort.
- Strong collaboration with NEC.

#### NEC (R&D)

##### Topics:

- MLCs
  - Actuators
- } Relaxors
- Packaging (AlN), Glass-Cordierite (low K)  
Artificial Superstructures
- 10-12 man years.
  - State-of-the-art characterization and processing equipment.  
Was not shown any manufacturing facilities.

#### Toshiba (24 Billion Dollar Company)

Over the last ten years the % of gross sales expended for R&D went from 6 → 8% and is expected to go to 10%.

##### Electro-Ceramic Topics:

“Functional ceramics.” Note: The term used in Japan for high tech ceramics.

- Packaging (AlN, hybrids).

- Piezo transducers for bio-medical applications.
- MLCs.
- ZnO varistors.
- Thin film capacitors (PMN-based).

No breakthroughs, but extremely high reliability. Will only invest in "large" volume components due to large capital investment costs. Perhaps only ~ 10-12 man years of effort.

Key to success is: 1) large % investment to R&D and 2) long term planning—5+ years.

#### Marcon Electronics

Capacitor company (Al, tantalum, MLCs, and varistors). Very open, was shown complete production line of relaxor MLCs and ZnO varistors including multilayer varistors.

- Relaxor MLCs > 10  $\mu$ f.
- Goal is to replace tantalum capacitors with MLCs.
- Use automated state-of-the-art, processing "no problem with Pb containing materials."
- All equipment commercially available.
  - clean rooms
  - attrition mills and mixing
  - tape casters, etc.

#### NEW TOPIC AREAS

In summary of my trip to Japan, R&D areas needed for U.S. universities and industry are presented in the following:

#### R&D Areas Needed

##### University

- Surface chemistry/powder processing/nano-heterocoagulation and characterization (ESCA).
- Reliability of relaxors.
- Mechanical properties of relaxors and related dielectrics.
- Domain theory, interface analysis and modeling of thin films.
- New thin film approaches, e.g., hydrothermal.
- New thin film materials, e.g., Ta<sub>2</sub>O<sub>5</sub>.
- Need long term funding (> 1 year).

##### Industry

- Need large capital investment to obtain state-of-the-art processing equipment (not thin films).
- Need to work harder and demonstrate dedication.
- Should stress more on reliability and less on performance.
- Need larger % gross R&D investment and long term planning.

REPORT ON FIFTH U. S.-JAPAN SEMINAR ON DIELECTRIC AND  
PIEZOELECTRIC CERAMICS, AND ON RELATED LABORATORY VISITS

Kyoto, Japan, December 11-14, 1991

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## INTRODUCTION

This fifth in a series of biannual joint meetings between U. S. and Japanese scientists and engineers offered another outstanding opportunity to learn about the recent and current activities on dielectric and piezoelectric ceramics in Japan. The personal relationships that have developed from these periodic meetings have contributed to easy communication between the two groups. This meeting was particularly well attended by the Japanese side, and it is apparent that this has become an important and prestigious affair for them. This report will be divided into three main sections: those presentations that were of specific interest to me, those that were of general interest, and my visits to industrial laboratories.

### PRESENTATIONS OF SPECIFIC INTEREST

I have always been very much interested in the development of base metal electrode (BME) multilayer ceramic capacitors (MLCs) because they are based on principles of doping and equilibration that derive directly from defect chemistry. There were two presentations that dealt with this area:

Multilayer Ceramic Capacitor with Ni Electrodes

H. Chazono, H. Kishi, and N. Yamaoka

Taiyo Yuden

At the Joint U. S.-Japan Seminar four years ago, Yamaoka presented a poster on a Ni-electroded MLC. It included a pre-reacted  $\text{Li}_2\text{O-SiO}_2\text{-CaO}$  glass as a sintering aid, and was fired in a reducing atmosphere to avoid oxidation of the Ni. The glass had to be present, and it had to contain Ca, so I concluded that the dielectric must absorb Ca



from the glass to serve as the acceptor dopant that is required to maintain an insulating dielectric after firing in a reducing atmosphere. The system was thus closely related to the Ca-doped composition used by Murata for their BME units.

This year, modifications were described that produced BME units that meet X7R specifications. This seems primarily to involve the addition of around 0.5 mol% MgO which served to depress the dielectric constant at the 125°C Curie point, giving a flatter temperature dependence that falls within the X7R requirements. The precise mechanism for this is not clear. Mg will also act as an acceptor dopant that can help to maintain an insulating dielectric after firing in a reducing atmosphere. The resulting life-test results were outstanding for a BME unit. After questioning this, it was stated that this was a very recent laboratory result that had not yet been verified for mass-produced units.

These capacitors are fired at 1200°C in a  $N_2-H_2$  "controlled atmosphere". This is inherently an uncontrolled system, since the oxygen activity will depend on such things as the water content of the gas mixture. Whether or not this is controlled is not clear. In any case, it is a very reducing atmosphere, and I cannot believe that the dielectrics come out of it in an insulating state. Apparently to correct this, there is a subsequent anneal at 1000°C in  $N_2$ . This is also an uncontrolled situation where the oxygen activity will depend on the accidental oxygen content of the commercial  $N_2$  gas. It appears that the units are sintered to a semiconducting state and then oxidized back to an insulating condition. The annealing step could either be a re-equilibration of the dielectric at an oxygen activity that will still not oxidize the Ni, or it may depend on kinetic control whereby the dielectric reoxidizes faster than the Ni oxidizes. My impression is that the latter is the case.

The Taiyo Yuden people continue to be very clever in an intuitive way in developing BME capacitors of high quality.

#### A Study on the Degradation of Ni Electrode Ceramic Chip Capacitors

T. Nomura, S. Sumita, Y. Nakano, and K. Nishiyama

TDK

Once again, this appears to involve firing in a highly reducing atmosphere that gives a semiconducting dielectric that is restored to an insulating state by an oxidizing anneal. The life-time under highly accelerated life-test conditions (HALT), 200 Vdc at 200°C, was measured as a function of the oxygen partial pressure during sintering, and the temperature during annealing. The normal conditions were 1340°C,  $10^{-12}$  atm, and 2 hours for sintering, and 1100°C,  $10^{-5}$  atm, and 3 hours for annealing. The dielectric composition was essentially that pioneered by Murata, an alkaline earth rich, Ca-containing  $\text{BaTiO}_3$ , with small amounts of MnO and  $\text{SiO}_2$ .

It was observed that the life-time under HALT conditions increased steadily as the oxygen partial pressure during sintering was reduced from  $10^{-5}$  to  $10^{-13}$  (problems arose at lower pressures because of anomalous sintering). In comparing chips sintered at  $10^{-8}$  and  $10^{-12}$  atm, it was noted that for the higher pressure, the "grain boundaries" contained substantial amounts of Ca, Mn, Fe, and Ni, all acceptor impurities. These concentrations were greatly reduced in the chips sintered at the lower pressure, and a Mn-rich region was noted adjacent to the Ni electrodes. These results leave a lot of questions, but it is tempting to suggest the following scenario. At the higher oxygen pressures, the acceptors are highly soluble, and are incorporated in the dielectric. After the oxidizing anneal, they are compensated by oxygen vacancies, whose migration

results in short life-times. The solubilities of the acceptor impurities are suppressed at the lower partial pressures, and they are segregated in impurity-rich layers near the electrodes. We have observed such a suppression of the solubility of acceptor impurities at low oxygen partial pressures, which occurs because the high oxygen vacancy concentration that results from reduction interacts through a common-ion effect with the vacancies that result from the acceptor content. When the solubility of the acceptor impurities has been suppressed by highly reducing sintering conditions, they cannot enter the lattice during the annealing step, because of very low diffusivities, so the oxygen vacancy content of the dielectric remains low, and the life-time increases accordingly.

It was also observed that the life-time increased as the annealing temperature was increased from 800 to 1100°C, and then fell off again. The initial rise can probably be attributed to increasingly complete reoxidation of the dielectric to an insulating state, while the drop above 1100°C reflects excessive oxidation of the Ni electrode layers.

This investigation was based on some good ideas, but was not pursued in enough detail to reach firm conclusions.

Chemical Processing of Electronic Ceramics

S. Hirano

Nagoya University

This plenary lecture by Professor Hirano was an excellent overview of processing routes. He does consistently excellent work and is clearly a leader in this field. His subsequent poster presentations on sol-gel processing of PMN and LiNbO<sub>3</sub> were also of great interest to me.

## OTHER PAPERS OF INTEREST

There were two papers describing the preparation of alkaline earth titanates by hydrothermal techniques, one from Sakai Chemical Industry, and the other a joint effort by Murata and the Tokyo Institute of Technology. This is an interesting new approach that we are hearing more and more about, primarily from Japan. The Sakai poster described the preparation of a very fine-grained powder, while the Murata team used an electrochemical technique to form thin films on a Ti anode.

Because of our intensive study of Ba zinc niobate, I was particularly interested in two papers on related materials. A paper from Toshiba described a Pb zinc niobate (PZN) dielectric for low-loss, high voltage applications. The microwave properties of BZN with partial replacement of Nb by Ta was studied as a function of compositional variables in a paper from Shonan Institute of Technology.

## LABORATORY VISITS

### Murata Manufacturing Company

On December 10, I visited the Research Laboratories of Murata for discussions with Drs. Sakabe and Wakino, and to give a seminar on ionic transport in ferroelectrics. Their production of Ni electroded multilayer capacitors continues to grow and is now approaching 200,000,000 per month, approximately ten times their production rate at the time of my last visit in 1986. They have explored two different approaches for BME capacitors: a low-fired composition with Cu electrodes, and the acceptor(Ca)-doped composition that can be fired in a reducing atmosphere with Ni electrodes. They have concluded that the latter is the best approach and have abandoned the low-fired,

Cu-electroded system. As stated above, it appears to me that they are now firing under reducing conditions that give a semiconducting dielectric, and are then restoring the insulation resistance by means of an oxidizing anneal. This differs from the original approach in which they fired the capacitors within a very narrow window of oxygen partial pressures that would not oxidize the Ni but would still give an insulating dielectric. The acceptor doping moved the insulating state below the oxygen activity of the Ni/NiO equilibrium. It was subsequently found that an additional oxidizing anneal relieved a problem with the hot leakage current. This anneal has to oxidize the dielectric without oxidizing the Ni, and the process then becomes kinetically controlled. Once this step was added, there is less reason to try to keep the dielectric insulating before the anneal. The acceptor dopant may now be serving to increase the rate of oxidation of the dielectric by enhancing the oxygen vacancy content.

It was also indicated that there had been problems with life-test stability which does not surprise me given the high oxygen vacancy concentrations in these acceptor-doped materials. When we first prepared Ca-doped BaTiO<sub>3</sub> with excess BaO so that the Ca substituted for the Ti as an acceptor dopant, they had the poorest accelerated life-test behavior of anything we had ever tested. Murata somehow managed to improve on that, but the problem has reappeared. They indicated that they had obtained some improvement by changing the annealing conditions or by Nb-doping. The latter is only going to undo some of the acceptor-doped effect, since Nb is a donor impurity. That should indeed improve life-test behavior, but should cause problems in maintaining an insulating dielectric after sintering. This is additional evidence that they are no longer trying to do the latter, but are depending on the subsequent oxidizing anneal to restore the oxidized, insulating state. In other words, they have changed their approach from using an equilibrium condition during sintering, to depending on the relative oxidation states of the dielectric and the Ni electrodes during the lower temperature oxidizing anneal.

On each of my visits to Japan, in 1982, 1986, and now in 1990, I have had a tour of the Murata Research Laboratories. Each time I have been extremely impressed by the size and quality of this operation. The laboratories are extremely well-equipped. I was particularly interested to see experimental facilities for measuring high temperature equilibrium conductivities and ionic transport numbers by oxygen concentration cells.

#### Sumitomo Metal Mining Corporation

On December 6 and 7, I visited laboratories of the Sumitomo Metal Mining Company. These visits were arranged by Mr. Ko Takada, who spent two years in our laboratory as a Visiting Scientist in 1985-86. On December 6, I visited the Electrical Materials Research Laboratory and gave a seminar on the defect chemistry of  $\text{LiNbO}_3$ . They have a special interest in this material for low-loss optical wave guides. They have developed a process for growing large single crystals of very high quality. On December 7, I visited the Central Research Laboratories, where the mutual interest was primarily on thick film resistors based on  $\text{RuO}_2$ . They have a substantial effort in this area with a very well-equipped laboratory. We had some interesting discussions on the possible conduction mechanisms in these complex structures.

#### SUMMARY

The joint seminar was very well organized and included an extremely large number of presentations. The format of very brief oral descriptions followed by a poster presentation seems to work very well, especially as a means of accommodating large numbers of papers. The interest and friendliness of the Japanese delegation led to open discussions and laboratory visits that were extremely valuable.

**5TH US - JAPAN MEETING ON DIELECTRIC AND PIEZOELECTRIC  
CERAMICS**

**December 11 - 14, 1990  
Kyoto University, Kyoto, Japan**

**Ahmad Safari  
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**In addition to the main Conference in Kyoto, the companies visited in Japan include the following:**

- (1) Mitsubishi Mining and Cement Company, Chichibu (December 10)**
- (2) Hitachi Central Research Laboratories, Kokubunji (December 17)**
- (3) Nippon Soda Inc., Odawara (December 18)**
- (4) Mitsubishi Materials Inc., Kanagawa (December 19)**

**Visit to Mitsubishi Mining and Cement Company**

**T. R. Gururaja accompanied me on this visit. A MMC representative picked us up from the Washington Hotel in the Tokyo area. Dr. Hikita, a senior researcher and T. Ueyama, assistant general manager, received us at the train station in Chichibu. After about an hour of touring around the city and shrines, we had lunch, finally arriving at the plant at 2:00 PM. We had a very fast and brief tour of two laboratories. They are actively working on PTC devices and piezoelectric bimorphs. MMC is not investigating porous piezoelectric composites.**

They showed us some excellent fine powder-transparent substrates. Unfortunately, we didn't have a chance to see their MLC Laboratory. The rest of the afternoon T.R. Gururaja gave a lecture on the processing and properties of Sm doped  $\text{PbTiO}_3$  (mainly some of his work at PSU) and I summarized the electronic ceramic research at Rutgers University. In the evening the general manager, assistant general manager and three other senior members acted as hosts for the whole evening. We had an excellent dinner and they picked up the entire hotel bill at Chichibu for that night.

We were eager and wanted to see some more activities but, unfortunately, the way they scheduled us we had less time to tour the facilities and discuss their research activities. Substrate work was very impressive.

#### December 11, 12, 13, 14 U.S. Japan Seminar

- Returned from Chichibu to Tokyo and traveled to Kyoto. Dr. Takenaka picked us up at the Tokyo station and traveled with us (T.R. Gururaja and me) to Kyoto.
- We registered that evening. The reception was excellent. Both the US and Japanese general chairmen, Dr. Wakino and Dr. Dougherty did an excellent job putting the program together, including all of the US and Japanese papers.
- Two plenary lectures by Professors Scott (US) and Hirano (Japan) were excellent. The important topic areas discussed at the meeting were relaxor



ferroelectric material, barium titanate ceramics, lead zirconate titanate piezoceramics and composites, electronic packaging and ferroelectric thin films.

- Relaxor Ferroelectrics

Japanese dominated in this area. Fourteen papers were presented by Japanese participants vs. three papers by the US. The Japanese researchers are concentrating on the processing and applications such as actuators, motors and, most recently, on the electrostrictive ceramic and ceramic-polymer composites. Excellent work was presented by Nippon Soda on the chemical processing of PMN-PNN ceramic powder (alkoxide method). One paper on relaxor thin films was presented by the Japanese. I believe that in the future they may concentrate on this area, since thin film relaxors may be good for capacitors and actuator applications.

- Barium Titanate

About eleven papers were presented by US participants in this area vs. two papers by the Japanese. An excellent paper was presented by the du Pont company on coating of additives and fluxes on the surface of  $\text{BaTiO}_3$  powder via chemical processing. As a result of this,  $\text{BaTiO}_3$  could be sintered uniformly and enhanced properties are expected.

- Lead Zirconate Titanated Ceramics and Composites

Both the US and Japan presented an equal amount of papers (25 papers

overall). Drs. Takanaka and Sakuta from the Science University of Tokyo presented a good paper on Pb free piezoceramics  $(\text{Bi,Na})\text{TiO}_3\text{-PbTiO}_3$ . The US needs to work on this area in the future. Excellent papers were given on processing of PZT fiber from sol-gel by the Japanese and fine structure PZT fiber-polymer composites by the US (Rutgers work). Also, an impressive paper was presented by Dr. Takauchi of Hitachi regarding 1-3 PMN-PT ceramic rods/polymer composites for controlled coupling coefficients of the ceramics and ceramic/polymer composites. This is an area in which we should start to do some research in the US.

- Ferroelectric Thin Films

Both the US and Japanese researchers are active in the ferroelectric thin film area. The US may have a slight edge in this area. A considerable number of papers were presented in the processing of PZT, PT and bismuth titanate thin films by different techniques such as sol-gel, laser ablation, sputtering.

- Electronic Packaging

Only four papers were presented on electronic packaging for substrate applications. There were notable papers presented by US participants (IBM, Cordierite glass ceramics for substrates). Apparently, the Japanese are concentrating on aluminum nitride.

### Visit to the Hitachi Central Research Laboratories at Kokubunji

T.R. Gururaja and R. Bucannon accompanied me. Mr. Matsuzda, a Hitachi representative picked us up from the hotel. Dr. Takauchi's activities were discussed and several of his laboratories were toured. Approximately eight persons are working in his group. Recent advancements of interest follow:

#### -Electrostrictive Ceramics and Ceramic Polymer Composites for Transducer Applications

Electrostriction is an excellent way to control the electromechanical coupling coefficient. They are using PMN based ceramics for this purpose. The exact composition of the ceramic is 0.91PMN-0.09PT. Experimenting with bulk ceramics, they were able to obtain 40% thickness coupling coefficient with the bias voltage of 400 (V/mm). Both  $K_t$  and  $K_p$  varied linearly with bias field. By dicing sintered PMN-PT ceramics and making PMN rods-polymer composites with 1-3 connectivity, they were able to decouple  $k_p$  and  $k_t$ . As a result,  $K_t$  was improved to about 55% with an applied voltage of 300 V/mm.

#### -Thin Films

Very good work on epitaxial growth of ZnO and PbTiO<sub>3</sub> thin films by sputtering and sol-gel. These films were made for ferroelectric memory applications.

### Visit to Nippon Soda at Odawara

Mr. Kato, manager of the ceramic group, picked me up from the hotel. After about two hours of traveling by train, we arrived at Odawara City. He gave me a brief tour of the city prior to arriving at the Research Center. This is a chemical company which is in very strong collaboration with NEC. In fact, NEC obtains most of the powders from Nippon Soda.

### Major Activities

#### Processing of Relaxor Ferroelectric Materials

- Namely PMN-PNN composition prepared by alkoxide method and also by high energy attrition milling. Three engineers are working full time on this project. The powder prepared by this company meets the requirements high reliability MLC's.

#### Tape Casting

- Two engineers working full time on the effects preparation and property evaluation of thin layers of tape relaxor of the materials.

#### Superplasticity of Transformation Toughened Ceramics

- The goal is to enhance the creep rate at which large superplastic deformations take place. They are studying the effect of the additives such as Fe, Mn, (Fe+b),  $V_2O_5$ ,  $Bi_2O_3$  to 3Y-TZP which may yield a liquid grain

boundary phase at low temperature. In the evening, Dr. Isehi, the executive vice-president of the company plus three other top people in managerial positions members of the company spent the whole evening with me.

Visit to Mitsubishi Materials Inc. at Kanagawa

- In the morning of December 19, I visited Dr. Sato's group briefly.
- Mainly interested in ferroelectric and superconducting thin films. Very good work on superconducting thin films by MOCVD and laser deposition but in ferroelectric thin films they are far behind.

SUMMARY

The 5th US - Japan Seminar was organized extremely well. There were very good interactions and productive discussions between the US and Japanese participants. Visiting the Japanese industries was very useful and a good experience for me. I think the new "Pb free" piezoelectric ceramics, piezoelectric fiber and electrostrictive ceramics and composites for controlled  $K_t$  were the highlight of the meeting. I believe that this seminar series should continue and participation from US industry should be expanded. I would like to take this opportunity to thank the Office of Naval Research for sponsoring my trip.

**DISCUSSION  
OF THE  
5TH UNITED STATES-JAPAN SEMINAR  
ON  
DIELECTRIC AND PIEZOELECTRIC CERAMICS**

**December 11-14, 1990  
Kyoto, Japan**

**Prepared by**

**Walter A. Schulze  
of  
New York State College of Ceramics  
at  
Alfred University**

## **1.0 Introduction**

This report will be divided into three sections. The first is general observations from the meeting and discussions with participants that do not fit with the evaluation of specific posters in the second section. The third portion is a brief review of a visit to the Shanghai Institute of Ceramics that occurred directly after the meeting.

## **2.0 General Observations**

My general observation from the meeting four years ago still seems valid; that is, the Japanese companies, in general, still seem to be committing a considerably larger fraction of their gross revenues into research and development, especially development, than their United States counterparts. The emphasis is still on products and the Japanese companies are not unwilling to consider very new and advanced processing techniques to gain the ability to produce advanced devices before a market has assured a sales advantage. Part of the perceived Japanese dominance in the research and development area may also come from the willingness of Japanese companies to present what appears to be relatively current development work. It was very interesting to hear that only about two thirds of the Japanese companies that wanted to attend were allowed to participate in the meeting. It was stated that about one half as many as we met were turned away. At this point, it is interesting to observe that Dr. Kenji Uchino was conspicuously absent with rumors ranging from a fall out with the organizers because of his strong industrial connections to exhaustion. I am not strong in my information, but I believe the facts lie toward the latter complicated by a possible career change. Also, it is interesting that the proximity of the meeting still did not warrant a showing from Kyocera. This may be because of Murata's traditionally strong support of the seminar series. Even the AVX (Kyocera) representative from the United States failed to show.

One of the highlights of the meeting was to see 15 United States industrial participants in the book. This is a major increase from four years ago and especially so in the light of our "recession". Figures presented by Joe Dougherty indicated United States-Japanese attendance figures as 18:26 for universities, 15:56 for industry, and 6:2 for government representatives. This good United States turnout is the result

of efforts by Joe and others to involve industry (lacking four years ago) and the Navy funding that aids university personnel like myself.

### **3.0 Comments on Selected Papers**

**P-1** Excellent presentation and shows the very good film work progressing in the United States. This demonstrates how military funding can boost a very marketable technology, if only United States industry will take the advantage.

**P-2** The selection of Dr. Hirano by the Japanese for their lead position just adds more emphasis to my feeling that he is a researcher to watch for innovations in processing.

**I-3** As usual, Dr. Cross is getting to the heart of understanding relaxor materials as many of us only wish we could.

**I-4** Dr. Nishigaki again uses a complex chemical approach, even for Japanese, and makes it work. This demonstrates the willingness to commit a large effort to develop a product even for a saturated market.

**I-6** I do not remember Taiyo Yuden having previous Ni electrode technology. It appears they are stabilizing resistivity with Mg on a B site as is done with Ca.

**I-8** Looks like excellent hydrothermal preparation of barium titanate.

**I-9** I believe using the Curie Weiss tail of a traditional relaxor is a new approach to developing a high-voltage dielectric.

**I-13** It is interesting to see a worker of Dr. Igarashi's caliber push multicompositional multilayers. The idea is sound; but until now, industry has been skeptical of processing problems.

**I-23** Dr. Wakino continues to display a very sophisticated approach to the development of high-frequency dielectrics and their characterization.

**II-2 and 3** The Prokopowicz formula continues to show



usefulness in life time projection.

**II-5** IBM is becoming more secure in their low-fire copper substrate technology and seems to have overcome the removal of organics at low temperature in a nonoxidizing system.

**II-9 and 10** These show the recognition of the need for more basic studies to develop better adhesion in thick-film materials.

**II-13** The connection between Yamamoto and Igarashi is clear here with both working on multicompositional multilayers to average or sum properties.

**II-14** Dr. Hirano is another to watch for excellent organic preparation techniques.

**II-15** Tom Shrou's smart calcining is an inexpensive alternative to organically-prepared powder.

**II-18** DuPont's coating process gives a view of next-generation-powder preparation for rheology control.

**II-19** Fibrous single crystals are impressive from theoretical and device perspectives.

**III-3** Interesting work. Apparently the field resulting from a short circuit boundary condition can actually increase cycle lifetime.

**III-4** Always good to see Bert Auld working in piezoelectric composites.

**III-9** Does this mean ceramic-containing composites are winning against PVDF in the hydrophone race?

**III-10** I sometimes wonder about so many players in the PMN game. I also wish I had asked more about lead safety and lead scrap disposal in Japan.

**III-11 and 12** Good to see a serious player like Takeuchi in the biased-electrostrictor development. This is a transducer area that should

have taken off years ago.

**III-14** Very interesting low-lead-piezoelectric system that should be explored. How is the patent coverage of this one?

**III-17** I do not remember previously seeing this type of rotational displacer.

**III-18 and 19** More interesting motion devices from Japan.

**III-22** As a result of this meeting I finally understand, from a ferroelastic-domain-reorientation concept, why the indent cracks are different lengths; but it came from discussions, not from the poster.

**IV-2** Thin film work is obviously being explored in Japan for all market areas.

**IV-6 and 8** Many normal and less obvious film-forming techniques are being explored.

**IV-9** This is a most impressive film-forming technique. The ability to follow substrate contours is excellent.

**IV-20** The PLZT samples by Nagata were physically very impressive and may represent an increase in possible device size if they are spatially consistent.

#### **4.0 Shanghai Institute of Ceramics**

My visit to Shanghai Institute of Ceramics was hosted by Dr. Chude Feng, who spent a year as a visiting scholar at the New York State College of Ceramics. The rationale for using the short period of time available for visitation after the United States-Japan Seminar was that I know essentially nothing about the conditions for ceramic research and development in China. Also, this is the only time I could afford to make the visit. I found it most educational!

Probably the most important realization was the level of living and working conditions. I knew China was not a rich country, but I also was

aware of the expensive equipment in operation in Shanghai. The workers there are to be commended for the very good research conducted in an old, unheated, concrete facility, which was not really constructed for research with poor maintenance and janitorial services. The difficulties seem to be overcome by skilled, patient individuals and careful work.

As would be expected, much of the equipment is imported with the dominant share from Japan and the United States. The choice seems to show careful and cost-conscious selection. The choice of projects seems to be driven by careful analysis of external literature and internal need. The Institute is very conscientious about developing profitable products and eliminating the need for foreign purchase of ceramic materials. As a project reaches the prototype level, it is transferred to the Research and Development facility about 30 miles away. This group of two-story buildings has a much more campus feeling, and the buildings seem to be constructed for the purposes.

At the Institute I was greeted by Drs: Yin and Guo. This was followed by visits to a host of departments. The visit to the structural materials area was conducted by the section leader, Dr. Jiang Dong Liang. Material projects ranged from hydrothermally-prepared, nano-size powder of stabilized zirconia to a pressurized furnace for preparing silicon nitride.

The mechanical properties testing facilities were supervised by Ting Rong Lai and included high-temperature three-point bend, indentation, and a computer-monitored group of high-temperature creep furnaces.

The single crystal area was described by Deputy Director, Fan Shiji. This area was impressive from the old crystal pulling equipment use for development to the incredibly sophisticated BGO crystals produced for CERN. Development has now turned to fluoride systems. Diamond production still seems to be a strong cash product. The work ranges from seeded large crystal production to sintered diamond coating for cutting tools.

In the electronic ceramics area, I was told that lead-based capacitor formulations had been successfully transferred to a production facility. The current thrust was demonstrated by a new, very clean area dedicated

to the production of PTCR barium titanate heater material. The barium titanate is produced from barium carbonate and titania very similar to older United States commercial systems.

I was honored by an evening banquet which was attended by Professors Guo, Yin and Feng. I was very happy to meet Chen Zhili again after a number of years. The professors were not sure she would make the dinner since she is ranked either third or fourth in Shanghai as Head of the Ministry of Propaganda. It is a good feeling to realize that a competent scientist and good person can rise to such a position.

Trip Report—Fifth U.S.—Japan Seminar  
on Dielectric and Piezoelectric Ceramics

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Structure of the Meeting

The meeting was exceptionally well organized and the hospitality of our Japanese hosts was extraordinary. Indeed, such hospitality presents a challenge to the organizers of the next meeting in the United States.

This is my second attendance at this series of meetings and my clear impression is that progress has been made since the Toyama meeting. This type of meeting (a small group, intensive meetings, and social gatherings in the evenings) is very effective in promoting information exchange. In this particular meeting the facilities for the technical sessions at the university and the social events at the hotel greatly enhanced the interactions and discussions, both international and intranational. For me, a very pleasant surprise was the degree to which I had extremely fruitful conversations with American colleagues and new acquaintances. I made several valuable contacts, to be followed up in the future. Also, the Japanese talked much more freely than ever before in my experience.

The use of three minute introductions by the principal poster authors is an excellent idea. This approach provided an information focus and a first contact with the authors that simplified the later viewing of the posters, and made the whole procedure more dynamic. The Japanese presentations were much improved over the rapporteur style of presentation at Toyama. Someone has clearly spent time coaching their younger scientists on how to summarize concisely and how to highlight their viewgraphs with colored cues—underlining, arrows, etc. Some of the Americans could well have taken a few lessons from some of the Japanese visual aids. The two plenary lectures were excellent although, perhaps, a little long. They were so effective that the next meeting could well include a greater number of review lectures. I realize that time is limited and that the essence of this

meeting lies in the poster sessions. But it would be useful to consider having four thirty minute lectures at the next meeting.

### Technical Content

The plenary lectures on "Physics and Engineering of Ferroelectric Memories" and "Chemical Processing of Electronics Ceramics" properly placed emphasis on the technology of ferroelectric thin films. It is impressive to see the advances made in this area since the Toyama meeting. In the Friday session on thin films and electrooptics the U.S. and Japanese effort was quite well balanced (9 versus 11 posters, plus one joint effort). Not unexpectedly there was a significantly greater contribution from industry among the Japanese and from universities among the Americans. This partly reflects the research-industrial structure in the two countries. But there remains also a problem in attracting American industrial scientists to these meetings. Efforts to correct this situation should continue. It was surprising to find so few Japanese ultrasonic and acoustic device engineers at this meeting. Less surprising because of the travel distance involved, was the small representation of Americans working in both ultrasonics and actuators. An effort should be made next time to invite selected attendance from the medical ultrasound and general sensors industries.

In my particular speciality, piezoelectric composites, the high points of the meeting were the poster presented by M. P. Harmer and J. Chen from Lehigh University on modeling the dielectric properties of diphasic mixtures, and the poster presented by H. Banno and K. Ogura of NTK on four-phase 0-3 composites. The first of these provides an excellent formal base for our work on modeling 0-3 composites, and the second led to stimulating conversations with H. Banno about the present status and future trends of the Japanese effort. Lively discussions were also held with M. Kahn of the Naval Research Laboratory on the subject of 3-0 composites. These highlights emphasize the intensive intranational and international contacts that constitute for me the strength of these meetings.

## Fifth US-Japan Seminar on Dielectrics and Piezoelectrics

Trip Report

Gene Haertling

Monday, December 10, 1990

A visit was made to Nippon Mining Company, Ltd. Nippon Mining is a \$5.1B company located about 1 hour from downtown Tokyo. It has assembled the largest concentrated effort in Japan on PLZT. Although they have focused on bulk materials for most of their work, they have also now initiated a program in thin film ferro-electrics utilizing a MOCVD technique. The abstracts of two papers (one not yet published) are attached to this report.

Accompanied by Dr. K. Nagata of the National Defense Academy (who claims to be the Japanese coordinator for PLZT in Japan), we spent the first part of the visit in presentations and discussions of the research work on PLZT at Nippon Mining. Dr. Hideo Sagawa, the general manager and host, presented an overview of Nippon Mining and then left the technical presentations to the scientists, most of which were very young. Five individuals gave talks on their areas of expertise - these included (1) T. Tsukada on powder preparation by chemical coprecipitation, (2) A. Nakajima on tapecasting, (3) R. Suzuki on sintering and hot pressing, (4) S. Okubo on evaluation and (5) A. Katoh on devices. The last part of the visit included a plant tour of the facilities dedicated to the PLZT work.

### Points of interest:

1. The PLZT development work was started three years ago.
2. It was funded with government seed money and a 3-year effort to establish a PLZT capability. Very good equipment was evident everywhere on the plant tour. This equipment is on loan from the government to establish this capability and will need to be returned within the next year. Examples of special equipment include large batch chemical coprecipitation processing equipment, a vacuum/oxygen uniaxial hot press, a hot isostatic press (HIP), a diamond surfacing grinder, lapping and polishing equipment, a computer-controlled slotting saw for electrodes, optical and IR spectrometers and E/O measuring apparatus.
3. The central research lab at Nippon Mining involves five separate labs and 900 people. Subdivisions include: (1) inorganic materials, (2) organic materials, (3) ceramic materials, (4) petroleum products and (5) resins. Their research and development of the PLZT materials is part of (3).
4. The PLZT effort involves a group of 10 researchers working in the specialty areas mentioned above.

5. They employ a different chemical preparation process for their PLZT powder, using  $PbO$ ,  $La_2O_3$ ,  $ZrO(NO_3)_2$  and  $TiCl_4$ .

6. The atmosphere sintering process for PLZT was pretty standard but good, and the PLZT polished ribbon (1" x 12" x 0.1") produced from it is of excellent quality. It was prepared from tapecast/laminated material and was obviously fabricated for printer heads.

7. There was nothing new on devices - mostly shutters. I saw both shadow evaporated electrodes and plated/slotted electrodes for their devices. The shutters I observed were of marginal quality because they exhibited a residual space charge problem which was obviously not addressed.

### Overall Assessment

Nippon Mining now has the capability of producing good, optical quality PLZT ceramics in quantity by either hot pressing or sintering and intends to supply the markets in Japan and elsewhere. Their progress in the last three years, from start-up to product development, has been very impressive. The issue of whether they have a cost effective product remains to be seen. This will be evident after the government funding goes away.

Tuesday, December 11, 1990

Tuesday was spent visiting NGK/NTK in Nagoya. Our host, Dr. H. Banno, introduced our group of US visitors to NGK and then took us on a tour of the manufacturing facilities for piezoelectric filters. The tour only included the last part of the production process; i.e., (1) slicing/dicing of the filter blanks from the fired plates, (2) fired-on Ag electroding, (3) auto-poling, (4) auto-tuning and (5) final testing. Although we were not shown the materials batching and processing areas, it was surmized that they were using a PZT formulation (probably 52/48) with both Cr and Mn additives.

In the manufacturing process shown to us, there was a large amount of automation in place. Extensive use of computer-interactive data collection and quality control were evident. According to Banno, their yields were very high because computer-interactive tuning of each individual filter assured them of these results.

In general, the line they showed us appeared to be very efficient and well run. It was more automated than other lines that I have seen at other companies in the US which deal with similar products. Much of their automation was their own; i.e., it was designed in-house and made in-house for their own use, exclusively.

Late afternoon was spent traveling to Kyoto.



Wednesday, Thursday, Friday, December 12-14, 1990

Personal Impressions of the Symposium

The format for the symposium seemed to be the best possible one - a good compromise between the presentation format which brings attention to the paper and the poster format which allows one to concentrate on the papers of real interest to the individual.

Applied technologies were emphasized in regard to processing, properties and products.

Contributions (papers) were of equally good quality from both US and Japanese participants.

First day papers were not as new and innovative as 2nd and 3rd days.

Some statistics:

- \* 91 total contributed papers
  - 35 US papers
  - 56 Japanese papers
  
- \* 50 papers on bulk materials
  - 18 papers on thin films
  - 23 miscellaneous
  
- \* 6 papers on modeling/theory
  - 12 papers on materials
  - 28 papers on processing
  - 18 papers on properties
  - 19 papers on phenomena
  - 5 papers on products
  - 4 papers on testing
  
- \* 16 papers on PMN
  - 10 papers on BT
  - 18 papers on PZT
  - 4 papers on PLZT
  - 3 papers on PT
  - 2 papers on  $\text{LiNbO}_3$
  - 2 papers on tungsten bronzes
  - 2 papers on PTCs
  - 3 papers on varistors
  - 4 papers on microwave ceramics
  - 2 papers on substrates
  - 6 papers on composites
  - 2 papers on fibers
  - 16 miscellaneous

Most popular materials at this symposium were PZT and PMN, and the most popular devices were actuators. Microwave ceramics and BT capacitors were also prominent along with thin films.

Most innovative modeling concept was the tessellation-derived microstructures of Penn State (S. Kurtz, et.al.)

Product papers were exclusively Japanese.

### Summary

Making new contacts and renewing old ones was a pleasant experience and of value, however, they were not critical to any new work that I plan in the near future. Although I did not feel that there were any breakthroughs reported at this symposium, there were some papers which in my mind stood out. These included:

1. Paper No. III-14 dealing with the composition  $\text{Bi}_{.5}\text{Na}_{.5}\text{TiO}_3\text{-PbTiO}_3$ . This system contains less less Pb and could be a substitute for PZT when lead compounds are restricted in the US in the future. In addition, it can be fired in an open furnace which would be a distinct advantage.
2. Paper No. II-18 involving chemical precipitation and coating processes for dielectric powders. This coating process can be important for possible lower temperature sintering and new boundary-layer capacitors.
3. Papers III-17, III-18 and III-19 utilizing PZT and PMN materials in actuator devices as torsional vibrators, auto-focusing systems and linear motors.

In general, I observed a high level of interest in the symposium and felt that it was worthwhile, albeit, more from the Japanese side than the US side. It appears that Okazaki's statement at the 1st Symposium in 1982; i.e., "Japan is too much in practical improvement and not enough in scientific work" is still true but it noticeably changing. On the other hand, it can also be said that the US is still heavy on scientific research and not enough in technology and product applications. This may be changing, but if it is, it is do so very slowly. As has been observed before and is still true, "in the long run, we (the US) will be the losers." Thin film ferroelectrics is now the new frontier which has opened up a myriad of possibilities for new science, technology and applications, especially integrated applications. Let's hope that the US maintains a balanced approach to this new initiative.

## DEPENDENCE OF ELECTRO-OPTICAL AND DIELECTRIC PROPERTIES OF PLZT CERAMICS IN PHASE BOUNDARY COMPOSITION

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For determining the composition region suitable for PLZT optical shutter utilizing second-order electro-optical effect, various PLZT compositions of phase boundary region compositions were prepared, and dielectric, electro-optical characteristics were examined. As the result, it has been found that the value of ratio of remanent polarization to spontaneous polarization is suitable as the factor for determining whether the electro-optical effect is good or not, and the composition of 8/70/30 is suitable when applied to optical shutter.

## PREPARATION AND ELECTRICAL PROPERTIES OF PZT THIN FILMS BY MOCVD

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Strongly [001] oriented lead zirconate-titanate ( $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ ) thin films with tetragonal structure ( $0 < x < 0.52$ ) have been successfully grown on (100)Pt/(100)MgO substrates by using metal-organic chemical vapor deposition (MOCVD). The metal-organic precursors were  $\text{Pb}(\text{C}_2\text{H}_5)_4$ ,  $\text{Zr}(\text{DPM})_4$  and  $\text{Ti}(\text{i-OC}_3\text{H}_7)_4$ . Scanning electron micrographs showed dense and noncolumnar growth with good surface morphology. The relative dielectric constants at room temperature were 200-350, and appeared to have less dependence on composition  $x$  than PZT bulk ceramics. Typical D-E hysteresis loops which occur with PZT bulk ceramics were observed. Remanent polarizations were  $30\text{-}40\ \mu\text{C}/\text{cm}^2$ . The coercive field decreased from  $65\text{ kV}/\text{cm}$  to  $40\text{ kV}/\text{cm}$  with increases in Zr content. The pyroelectric coefficients without poling treatment were about  $3 \times 10^{-8}\ \text{C}/\text{cm}^2\text{K}$ , showing almost the same value as that of poled PZT bulk ceramics.

## **EVALUATION OF THE FIFTH U.S.-JAPAN SEMINAR ON DIELECTRIC AND PIEZOELECTRIC CERAMICS**

**PAUL SCHUELE, RAMTRON CORPORATION**

### **FORMAT:**

- The format worked very well. Both the extended abstracts and the 3-minute introductions made information transfer effective.
- Because of the diversity of subjects, more plenary sessions would be very helpful.
- Number of participants should be limited to about 100.

### **IMPORTANT POINTS**

- The diversity of methods for producing thin films of useful quality (RF, ion beam and reactive sputtering, Sol-Gel, laser ablation, and CVD). The benefits of each technique for improved film properties must be investigated.
- The step coverage for MOCVD PT was very good, which is promising for FE-semiconductor integration with CVD.
- The papers modeling material properties as a function of grain structure using tessellation (Kurtz) and percolation (Harmer) indicates a direction for much more work, particularly in light of the issues for MLC's with thin layers (Maher).
- Preferred orientation for thin films seems to be easier to change than I would have believed (Shiosaki & Hirano). This can be important for optimization of electrical properties.
- The study of interaction between ceramics and other elements in the system, i.e. glass (Yamaguchi, Onda) or Ni (Nomura).
- Composite capacitors (Igarashi and Yamamoto) suggests using thin-film deposition to micro engineer capacitor performance.
- No breakthroughs but a lot of solid work aimed at higher performance and higher reliability devices. Clear trends toward better microscopic understanding and more controlled device fabrication.

### **THINGS MISSING:**

- Detailed electrical characterization of thin films deposited by various methods.
- Study of the ceramic/electrode interface in depth. Only one paper (Nomura).
- Very few papers on optoactive applications and characterization.

Department of Physics

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27 Dec 1990


REPORT ON THE 5th US-JAPAN SEMINAR ON DIELECTRIC AND  
PIEZOELECTRIC CERAMICS

As a researcher whose work is almost equally divided between academic university programs and industrial R&D, I found the mix of people and papers at this conference particularly pleasant. My own special area of interest is in ferroelectric thin film memories. Most of the papers in this area were presented on the third day, and some were quite innovative: I particularly liked the excimer-laser ablation-deposition of PZT reported by Krupanidhi; his dep rate of 1 micron in 20 minutes was quite impressive. The result of Dey on high-voltage switching of sol-gel PZT films was also quite noteworthy; by constructing a high-voltage, ns risetime driving circuit for his PZT RAMs, he was able to show two distinct characteristic activation fields for switching, one of which "turns on" at 300-350 kV/cm. (I suspect that this is the threshold between inhomogeneous nucleation at the electrode interfaces and homogeneous nucleation throughout the film.) On the more basic side, Prof. Cross' study showing the analogous relationship between magnetic spin glasses and relaxor ferroelectrics appears quite profound and is likely to be the seed for a very large number of studies in the immediate future.

On the Japanese side the paper by Prof. Shiosaki showing virtually perfect submicron step coverage of PZT on Si via CVD deposition was startling in the quality of coverage produced. It was without question the Japanese paper of greatest interest to me. Second would rank the plenary review by Hirano, which was delivered in excellent English and full of good new information.

As in any good conference, the really important information is transmitted in the hallways or after dinner. Such was the case in Kyoto. I was particularly interested in understanding the level of interest and activity within Japanese industrial laboratories for the application of ferroelectric thin films in both DRAMs (for higher capacitance to minimize trenching problems 64Mb DRAMs) and for ferroelectric RAMs. Surprisingly, this activity is not limited to companies like NEC and Matsushita, where it might be expected, but also spills over into corporations that are not into high-density memories, such as the capacitor manufacturers (Murata and Kyocera). There is not such interest in vertical integration among US corporations of the same size and technical orientation.

After the conference I was fortunate to be invited to give a plenary lecture at a meeting of JEIDA, the Japanese Electronics Industry Association, in Tokyo. At this talk there were technical representatives from the following Japanese corporations: Oki Electric Co., Canon, Sharp, Seiko, Sony, TDK, Toshiba, Nishin Denki, Japan Signal Co., NEC, JVC, Hitachi, Fuji-Xerox, Fujitsu, Matsushita, Mitsubishi, Murata, Yokagawa, Ricoh, Sumitomo Chemical, and Fuji Electric Co., among others. For me this was a unique opportunity to present the results of my efforts over the past five years to develop ferroelectric memories in Colorado, and to exchange technical information with leaders of Japanese microelectronics industries. There is, however, a sad comparison to be made with the level of interest in this technology found in US industries, where only a handful are doing work (Westinghouse, TRW, McDonnell-Douglas, Raytheon) and almost entirely with DOD funds rather than in-house IR&D. As it stands, these US-Japan symposia are excellent, but unless more US industries can be coerced into participation, they risk being technology-transfer avenues between US universities and Japanese industries, which is not altogether healthy.

  
Prof. J. F. Scott  
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FIFTH U.S. - JAPAN WORKSHOP ON DIELECTRIC  
AND PIEZOELECTRIC CERAMICS

Report by

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On 11-14 December 1990 I attended the Fifth U.S.- Japan Seminar on Dielectric and Piezoelectric Ceramics in Kyoto, Japan. Prior to the meeting I visited Professor Niihara at Osaka University to learn about their work on "nanocomposites". They reported that the incorporation of small amounts (5-10 vol%) of "nano" - sized (0.3 micron diameter) SiC particles into various ceramics could significantly enhance the strength. Obtaining a uniform dispersion of the SiC particles is crucial to the attainment of high strength. In confirmation of their results, we have since obtained strengths in our laboratory of over 1000 MPa on hot pressed alumina containing 5 vol % SiC particles. For comparison, the undoped alumina had an average strength of 560 MPa. I am not aware of other groups in the U.S. pursuing this approach. The seminar itself covered a wide exchange of information on several topics and I will just make some general observations here. Firstly, it was clear that there has been significant growth in the area of ferroelectric thin films for use in non volatile computer memory and DRAM applications. Researchers from Murata described a novel hydrothermal electrochemical technique for producing high quality thin films at very low temperatures (100 - 200 degrees C). Overall, the U.S. and Japan seemed to be making comparable progress in this field. Piezoelectric composites appear to have reached a mature stage of development. Piezoelectric motors and high strain actuators are being developed extensively in Japan for a variety of applications, whereas the U.S. activity in this area is very small. U.S. capacitor companies have been reluctant to develop lead based relaxor ferroelectrics for capacitor applications, whereas Japanese companies have steadily been introducing relaxors into the capacitor business. Both in Japan and the U.S. the technology for reducing the thickness of the dielectric layers in multilayer capacitors has been improved. Minimum thicknesses in the range of 5 - 10 microns are now routinely achievable.

## LIST OF U.S. PARTICIPANTS

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