SYMPOSIUM DD

Materials Issues for Tunable RF and Microwave Devices II

November 29 - 30, 2000

Chairs

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Symposium Support
Army Research Office
DARPA
Office of Naval Research

^{*} Invited paper

SESSION DD1: OVERVIEW AND OPPORTUNITIES

Chair: Stephen K. Streiffer Wednesday Morning, November 29, 2000 Room 206 (Hynes)

8:30 AM *DD1.1 FREQUENCY AGILE MATERIALS FOR ELECTRONICS: AN UPDATE OF THE DARPA PROGRAM. Stuart A. Wolf, ${\rm DARPA/DSO},\ {\rm Arlington},\ {\rm VA}.$

The Frequency Agile Materials for Electronics (FAME) program has as its goal the improvement of tunable materials for RF and microwave applications. The challange is to develop methods for lowering the loss while increasing the tuneability of materials whose permitivity and permeability can be changed with the application of electric or magnetic fields. In this talk I will review the various projects in the DARPA FAME program and indicate the applications that have been identified for the developments to date.

9:00 AM *DD1.2

IMPROVED TECHNIQUES FOR MEASUREMENT OF LOSS TANGENT OF FERROELECTRIC THIN AND THICK FILMS IN THE MICROWAVE REGION. Stanley Toncich, KWC, Inc., San

The accurate determination of ferroelectric(f-e)thin and thick film loss tangent in the microwave region (1 - 10 GHz) is critical for two reasons. One is to guide the improvement of film quality, the other is to assist the successful introduction of these materials into the commercial market. A common technique used to obtain loss tangent information is to make quality factor ("Q") measurements in either a resonant cavity or circuit. To date, much of the loss tangent data has been obtained from Q measurements made on interdigital capacitors (IDC's). In doing so, it is critical to correctly remove contributions to the overall loss due to not only the metalization but to the geometry of the IDC. Accounting for IDC loss is complicated by the fact that the required geometry for high tunability (narrow gap spacing) also increases the loss in the IDC exponentially. Thus even if an accurate model for IDC loss is used, removing its effect can introduce large errors in the resulting loss tangent values even if small errors are made in accounting for IDC loss. Another complication is that measured loss tangent is somewhat dependent on the geometry of the circuit used to measure it as well. This paper will discuss ways to correctly account for IDC and geometry-based loss contributors using analytical data and electromagnetic simulation tools. It will also present a measurement technique that may mitigate, if not eliminate many of the sources of error. It is based on Q factor measurements of gap coupled open circuit microstrip resonators. Thus measurements can be easily be done at the frequency of interest and the geometric contributors to loss can be more easily limited and extracted. Data will be presented as well.

10:00 AM *DD1.3

SYSTEMATIC EVALUATION OF FERROELECTRIC BSTO THIN FILMS DRIVEN BY A SPECIFIC TUNABLE MICROWAVE APPLICATION. Felix Miranda, Carl Mueller, Fred Van Keuls, Robert Romanofsky, Samuel Alterovitz, Joseph Warner, NASA Glenn Research Center, Cleveland, OH.

The application of ferroelectric thin films for the development of frequency and phase agile microwave components has been the primary reason behind very encouraging demonstrations of tunable microwave devices in recent years. Based on these results, it appears that tunable thin film ferroelectric technology can have a vital role in specific applications. However, it has become increasingly apparent that a detailed understanding of how film microstructure impacts device performance is largerly unknown, and furthermore, that the "ideal" film microstructure may be different for different applications. In this paper, we will discuss the material evaluation methodology implemented at NASA Glenn Research Center aimed at identifying and optimizing the most relevant parameters. We report film microstructure and quantitatively characterize the effects of film crystallinity, lattice parameter, film thickness, and film strain on microwave tunability, loss and robustness for a specific microwave application (in our case, phase shifters for reflectarray antennas). Results of our material analysis and how these properties correlate with microwave performance for the aforementioned targeted circuit will be presented.

10:30 AM DD1.4

NOVEL ROOM TEMPERATURE ZnMgO/ZnO RESONANT TUNNELING DEVICES FOR MICROWAVE APPLICATIONS. Aravind Inumpudi, Agis A. Iliadis, University of Maryland, Dept of Electrical and Computer Engineering, College Park, MD; Supab Choopun, T. Venkatesan, University of Maryland, CSR, Dept of Physics, College Park, MD.

The development of resonant tunneling devices in the ZnMgO/ZnO system is reported for the first time. Negative differential resistance (NDR) peaks have been observed at room temperature in these structures that consist of ZnMgO barriers and ZnO quantum wells grown by pulsed laser deposition (PLD) on C-plane sapphire substrates. The structures consist of a double ZnO quantum well with well widths of 6 nm and ZnMgO barrier widths of 8 nm. Multiple (five) well structures were also developed demonstrating multiple negative differential resistance (MNDR) peaks. A thin indium-tin oxide (ITO) layer is deposited first on the sapphire substrate followed by 150 nm of a ZnO buffer layer upon which the resonant structure is grown. The ITO layer serves as the back contact electrode for the structure. Secondary ion mass spectroscopy (SIMS) analysis was employed to verify the structure composition, and electrical characterization was performed both at room temperature and low temperatures. The resonant tunneling characteristics of the system will be reported. The tunability and merits of such metal oxide system in microwave applications will be discussed.

BISMUTH PYROCHLORE DIELECTRIC FILMS. Ryan Thayer, Wei Ren, Nathanael Bennink, Clive A. Randall, Thomas R. Shrout, Susan Trolier-McKinstry, Materials Research Laboratory, Pennsylvania State University, University Park, PA.

Bismuth pyrochlore ceramics have modest temperature coefficients of capacitance (TCC), good microwave properties, and can be prepared at relatively low temperatures. This work focuses on thin films in this family. A MOD procedure was used to prepare $(\mathrm{Bi}_{1.5}\mathrm{Zn}_{0.5})$ $\rm (Zn_{0.5}Nb_{1.5})O_7$ and $\rm Bi_2(Zn_{1/3}Nb_{2/3})_2O_7$ films. In both cases, crystallization occurred by 550°C into the cubic pyrochlore structure. At 750°C and above, Bi₂(Zn_{1/3}Nb_{2/3})₂O₇ adopts a monoclinic pyrochlore structure. Cubic bismuth zinc niobate films show a permittivity of 150, $tan \delta$; 0.005, negative TCC, and a 30% field tunable permittivity at 1.8 MV/cm. Monoclinic films show a smaller permittivity (~ 80), $\tan \delta$; 0.005, positive TCC, and little field dependence of the permittivity at room temperature. Crystalline $\mathrm{Bi}_{2}(\mathrm{Zn}_{1/3}\mathrm{Nb}_{2/3})_{2}\mathrm{O}_{7}$ films could be deposited at 400°C by pulsed laser deposition. This makes this family of materials interesting for capacitors on polymer substrates and on fully processed Si devices Support for this work was provided by the TDK Corporation, and by Intel Corporation.

SESSION DD2: DIELECTRIC CHARACTERIZATION Chair: Felix A. Miranda Wednesday Morning, November 29, 2000 Room 206 (Hynes)

11:00 AM *DD2.1

INVESTIGATION OF LOCAL FERROELECTRIC PHASE TRANSITIONS IN $Ba_xSr_{1-x}TiO_3$ THIN FILMS USING CONFOCAL SCANNING OPTICAL MICROSCOPY. Oleg Tikhomirov, Jeremy Levy, Univ of Pittsburgh, Dept of Physics and Astronomy, Pittsburgh, PA; Hua Jiang, NZ Applied Technology, Inc., Woburn, MA.

We employ temperature-dependent confocal scanning optical microscopy (CSOM) to investigate local ferroelectric phenomena near the cubic-tetragonal phase transition in $Ba_xSr_{1-x}TiO_3$ thin films. The local ferroelectric hysteresis loop changes shape and disappears as the temperature is increased over a narrow range of temperatures $(\sim 10 \text{ K})$. The local Curie temperature $T_c(x,y)$ varies significantly with position, and the distribution of T_c is consistent with the broad temperature dependence of the dielectric permittivity measured through conventional methods. These results directly confirm the hypothesis that the ferroelectric phase transition in thin ferroelectric films is inhomogeneously broadened.

The work was supported by the Officie of Naval Research (N00173-98-1-G011) and the National Science Foundation (DMR-9701725).

11:30 AM DD2.2

DIELECTRIC ANISOTROPY AND TUNABILITY OF (BaSr)TiO3 FILMS STUDIED BY SCANNING NEAR-FIELD MICROWAVE MICROSCOPY, Y.G. Wang, M.E. Reeves, Department of Physics, The George Washington University, Washington, DC; W.J. Kim and J.S. Horwitz, Naval Research Laboratory, Washington, DC.

We have studied the dielectric anisotropy and tunability of (BaSr)TiO $_{3}$ films prepared by pulsed laser deposition (PLD) using a scanning microwave microscope at ambient conditions. The film dielectric permittivity and tunability increases with deposition oxygen pressure from 35 mT to 50 mT, and then decreases for oxygen pressures between 50 mT up to 500 mT. The dielectric anisotropy, i.e., the ratio of out-of-plane vs in-plane permittivity, decreases also

with background oxygen pressure for films grown between at O pressure of 50 mT. Theoretical calculations reveal that the in- vs. out-of-plane component of the probe field changes with tip size and dielectric anisotropy. These results agree with the measurements and x-ray diffraction analysis.

11:45 AM DD2.3

FREQUENCY-DEPENDENT CHARACTERIZATION OF VOLTAGE-TUNABLE DIELECTRIC THIN FILM MATERIALS USING WAFER PROBING TECHNIQUES. James C. Booth, Leila R. Vale, Ronald H. Ono, NIST, Boulder CO; Ichiro Takeuchi, University of Maryland, College Park, MD.

We use wafer-probing techniques to determine the frequency dependent dielectric properties of voltage-tunable thin film materials over the frequency range 30 kHz to 40 GHz. Determination of the relevant dielectric properties over such a broad frequency range is accomplished by measuring a number of different planar devices all fabricated on the same thin film sample. We combine lock-in amplifier measurements of lumped-element interdigitated capacitors (30 kHz 30 MHz) with vector network analyzer measurements of distributed coplanar waveguide transmission lines (50 MHz 40 GHz), and determine the real and imaginary parts of the complex dielectric function vs. frequency by analyzing different geometry structures using simple models. In addition, we are able to perform these measurements at arbitrary temperatures in the range 20-300 K by using a variable temperature probe station. We apply these techniques to obtain the frequency dependent permittivity, loss tangent and tunability of a number of different voltage-tunable thin film materials at various temperatures of interest. We believe that such frequency-dependent measurements provide valuable new insights into the origin of losses in these materials and directly address the important issue of frequency dispersion of the dielectric properties.

SESSION DD3: MATERIALS AND MICROSTRUCTURE

Chairs: James S. Horwitz and Xiao-Dong Xiang Wednesday Afternoon, November 29, 2000 Room 206 (Hynes)

1:30 PM *DD3.1

STRUCTURAL AND DIELECTRIC PROPERTIES OF TUNABLE $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ FILMS, Q.X. Jia, B.H. Park, B.J. Gibbons, A.T. Findikoglu, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM; Y. Gim, Superconducting Device Group, Jet Propulsion Laboratory, Pasadena, CA; P. Lu, Dept. of MS&E, New Mexico Tech, Socorro, NM.

We have systematically studied nonlinear dielectric $Ba_{1-x}Sr_xTiO_3$ films deposited on LaAlO₃ and MgO substrates by pulsed laser deposition, where x is in the range of 0 to 1 at an interval of 0.1. The dielectric and structural properties of the films are strongly correlated with the chemical composition of the film, and the substrate temperature during deposition. The type of substrate also has influence on the dielectric properties of the films. By optimizing the chemical composition of the film, the type of substrate, and the processing conditions, we have achieved room-temperature dielectric tunability of 65% and figure of merit (tunability/loss) of 43 at an applied surface dc electric field of 80 kV/cm.

2:00 PM DD3.2

FERROELECTRIC Na_{0.5}K_{0.5}NbO₃ THIN FILMS ONTO SINGLE CRYSTAL OXIDE SUBSTRATES FOR TUNABLE MICROWAVE DEVICES. Choong-Rae Cho, Alex Grishin, Dept of Condensed Matter Physics, Royal Institute of Technology, Stockholm, SWEDEN; Johanna Andreasson, Dept of Materials and Manufacturing Engineering, Lulea University of Technology, Lulea, SWEDEN.

Single phase $\mathrm{Na_{0.5}K_{0.5}NbO_3}$ (NKN) thin films have been grown onto Al₂O₃(0112), LaAlO₃(001), and MgO(001) single crystal substrates using pulsed laser ablation of stoichiometric ceramic target. The microcrystalline properties of the films on these substrates were found to be quite different: highly c-axis oriented onto Al₂O₃, cube-on-cube epitaxial growth on LaAlO₃, and bi-axial textures on MgO. Furthermore, both [001] and [0kk] oriented textures in NKN films on MgO substrates showed in-plane relations with the substrate. The nature of pulsed laser deposited NKN film growth will be discussed in the framework of adatom interactions and film growth modes to interpret observed crystalline properties. All the samples on different substrates showed tunability higher than 50%, dielectric loss $\sim\,1\%$ at electric field ~ 200 kV/cm at frequency of 1 MHz. This work was supported by the Swedish Agency NUTEK.

2:15 PM <u>DD3.3</u>

MOCVD and SPUTTERED $Ba_xSr_{1-x}TiO_3$ (BST) THIN FILMS

FOR HIGH FREQUENCY TUNABLE DEVICES. $\underline{Jaemo\ Im},\ P.K.$ Baumann, O. Auciello, and S.K. Streiffer, Argonne National Laboratory, Materials Science Division, Argonne, IL; D.Y. Kauffman, and R.A. Erck, Argonne National Laboratory, Energy Technologies Division, Argonne, IL.

The high permittivity, high breakdown field, and voltage response of $(\mathrm{Ba}_x\mathrm{Sr}_{1-x})\mathrm{Ti}_{1y}\mathrm{O}_{3z}$ (BST) thin films make them attractive candidates for high frequency devices such as varactors, frequency triplers, and tunable phase shifters. In order to successfully realize BST for high frequency voltage tunable devices, it is critical to develop deposition technologies that can produce films with sufficient quality with respect to dielectric loss and tunability. In this paper, two such deposition methods, MOCVD and sputtering, are compared. We have grown phase pure, {001} fiber-textured BST thin films at 650°C on (111) oriented Pt films on high resistivity silicon substrates, using a large area vertical MOCVD system. Using this thermal MOCVD deposition technique, we obtained BST capacitors with dielectric losses as low as 0.003 and tunability higher than 2.5:1. On the other hand, we could achieve higher tunability (>4:1) but with higher losses (0.005) using the higher energy magnetron sputtering technique. High tunability of the sputtered BST capacitor results partly from the high breakdown field (~4 MV/cm), achieved by control of the BST-Pt interface composition and microstructure. Other approaches to improve high frequency device performance of the BST capacitors involve reducing conductor and substrate losses We will present the electrical properties of BST capacitors integrated with low loss substrates (quartz) and electrodes (Ir and Cu). Work supported by the U.S. Department of Energy, Office of Science and Office of Advanced Automotive Technologies under Contract W-31-109-ENG-38, and by DARPA-FAME Program.

 $2:\!30~\mathrm{PM}~\underline{^*\mathrm{DD3.4}}$ EFFECT OF LATTICE DISTORTION ON THE MICROWAVE DIELECTRIC PROPERTIES of (Ba,Sr)TiO₃ THIN FILMS. James S. Horwitz, Wonjeong Kim, Syed B. Qadri, Jeffrey M. Pond, Steven W. Kirchoefer, Huey-Daw Wu, Daniel M. Bubb and Douglas B. Chrisey, Naval Research Laboratory, Washington, DC.

Epitaxial $\mathrm{Ba_xSr_{1-x}TiO_3}$ (BST, x=0.4 to 0.7) films have been deposited onto (100) MgO and (100) LaAlO_3 (LAO) substrates by pulsed laser deposition. The dielectric constant and loss tangent have been measured at room temperature and microwave frequencies (1-20 GHz) as a function of electric field (< 200 kV/cm). The dielectric properties are strongly affected by the resulting stiochiometric and structural defects, eg, the ratio of Ba/Sr and oxygen vacancies. An analysis of the X-ray diffraction data for BST compositions (x < 0.5), which are expected to be cubic at room temperature, indicates that the films are tetragonally distorted. The magnitude of the distortion depends on the processing conditions and is strongly correlated with the microwave dielectric properties of the film. The tetragonal distortion is caused by the lattice and thermal mismatch between the film and substrate. Oxygen vacancies can be used to alter the nucleation and growth of the film and change the lattice parameter, and the subsequent distortion of the deposited film. Films deposited at low partial pressures of oxygen have an in-plane lattice parameter that is larger than the out of plane parameter resulting in a film that is in compressive stress. For high oxygen deposition pressures, the ratio is reversed and the film is in tensile stress. For all values of x, an oxygen deposition pressure can be found which results in the growth of a film with nearly equivalent in-plane and out-of-plane lattice parameters. For paraelectric compositions, strain-free films have a maximum microwave figure-of-merit (dielectric Q x % tuning). In addition, acceptor dopants such as W and Mn have been used reduce the free carrier concentration generated by the oxygen vacancies and further reduce the dielectric loss. A summary of the effect of procession conditions on film structure and properties as well as its impact on tunable device applications will be presented.

3:30 PM *DD3.5

MICROWAVE PROPERTIES OF THIN FILM FERROELECTRICS. R. Ramesh and S. Anlage, Materials Research Science and Engineering Center, University of Maryland, College Park, MD; S. Tidrow and E. Adler, Army Research Laboratory, Adelphi, MD; F van Keuls, R. Romanofsky and F. Miranda, NASA Lewis, Cleveland,

Over the past four years, we have been studying the interplay between thin film deposition parameters, film microstructure and dielectric properties of barium strontium titanate thin films for use in a variety of high frequency applications. Epitaxial BST films have been deposited on a variety of substrates including Si by pulsed laser deposition. The heterostructures have been characterized using a variety of conventional tools such as xray diffraction, transmission electron microscopy as well as novel probes such as scanning microwave microscopy. Dielectric measurements have been carried out at 1MHz as well as at microwave frequencies (11-19 GHz). We observe a strong decrease in the dielectric constant along with a higher loss tangent compared to the bulk. Theoretical models indicate that this could arise from one or all of the following: (i) substrate induced effects (such as clamping; (ii) formation of dislocations to relieve the lattice mismatch strain; (iii) point defects. In this presentation, we will discuss our results to date on these aspects.

This work is supported by the NSF-MRSEC and the ARL-MRCP.

4:00 PM <u>DD3.6</u> Abstract Withdrawn.

4:15 PM DD3.7

ELECTROPHORETIC DEPOSITION OF THICK FILMS OF BARIUM STRONTIUM TITANATE COMPOSITES FOR ELECTRONICALLY SCANNING ANTENNAS. Bonnie Gersten, Jennifer Synowcynski, Army Research Laboratory, Aberdeen Proving Ground, MD.

The composition of 0.45Ba_{0.55}Sr_{0.45}TiO₃ (BST)- 0.55 MgO bulk material was recently found to be a promising candidate as a low loss $(\tan\delta=0.007)$, highly tunable material for applications in electronically scanning antennas (ESA) at \geq 10 GHz. However, the baising fields required for phase shifting elements in ESA using the bulk materials are currently > 250V. Therefore, it is necessary to use high voltage circuitry and elements. However, the biasing fields can be reduced by developing thick films with similar properties. In this study, electrophoretic deposition (EPD) was used as a method for depositing thick films of BST composites on conducting substrates EPD thick film processing has the advantage of a low cost method that can be used to coat a conformal structure. Concentration and conductivity of the suspension, particle size of the powders, and bias field was studied to control the deposition thickness. The microstructure of the thick films prepared by EPD was compared with the bulk ceramics and other thick film processing techniques.

4:30 PM *DD3.8

COMPREHENSIVE SEARCH FOR FREQUENCY AGILE MATERIALS BY PARALLEL SYNTHESIS AND HIGH THROUGHPUT SCREENING. Xiao-Dong Xiang, Lawrence Berkeley National Laboratory, Berkeley, CA.

The parallel synthesis of materials chips and high throughput screening technique provided a vehicle to rapidly explore broad composition landscape of materials for superior frequency agile materials in microwave electronic applications. In this approach, "integrated materials chips" of large number of discrete compositions or continuously varying compositions designed to map the phase diagram of selected materials systems are fabricated by sequential thin film deposition of elemental precursors through "combinatorial masks" or linear shutters. Followed by proper annealing processes, thousands of distinct compounds or continuous binary and ternary phase-diagrams are formed, in either polycrystalline or more often epitaxial thin film format, on a small (e.g. inch 2) substrate. A scanning evanescent microwave probe is used to provide quantitative, non-destructive and high throughput mapping of dielectric constant, loss tangent and tunability of these materials chips. We have successfully used this approach to identify materials systems with much improved figure of merit. Mapping and identification of ferroelectric-paraelectric phase and morphotropic phase boundaries has provided new opportunities.

> SESSION DD4: POSTER SESSION Chairs: Brady J. Gibbons and Ichiro Takeuchi Wednesday Evening, November 29, 2000 8:00 PM Exhibition Hall D (Hynes)

DD4.1

MICROWAVE PROPERTIES AND RELAXATION IN POLYCRYSTALLINE Z-TYPE HEXAFERRITES. <u>D. Autissier</u>, P. Allegri, T. Taffary, CEA, Materials Dept, Monts, FRANCE.

Baryum hexaferrites are anisotropic iron oxides. High permeability values can be obtained at relatively high frequencies. In order to derive benefit from anisotropy, the grains of the ceramic have to be oriented. We have studied Z-type hexaferrites, which composition is Ba₃Co₂Fe₂₄O₄₁. Powders are synthesized using a classical ceramic method: stoichiometric amounts of basic components are ground and fired at high temperature (between 1150 and 1250°C in air or oxygen) to obtain the pure Z phase. The powders are then ground for 24 hours in order to reduce the particle size. The slurry is casted in a plaster matrix. This matrix is rotated between the poles of a stationary electromagnet. A field lower then 500 Oe is used for the orientation procedure. Samples are then sintered for different temperatures between 1200 and 1350°C in air or oxygen. If the materials present

high permittivity values, due to iron reduction, an annealing under oxygen could be executed. Investigations on its effects have been carried out. We present the results (magnetization, permeability, permittivity, orientation rate) obtained for the diverses elaboration conditions. We have studied the effect of iron and baryum excess, sintering temperature, atmosphere and dwell time on hyperfrequency magnetical and dielectrical properties, orientation rate, microstructure. In the case of oxygen sintering or annealing, the phase is pure, porosities are smaller than 5% and orientation rate (X-ray determination) is higher than 99%. Very high values of permeability (for this materials) have been obtained.

DD4.2

ENHANCED NONLINEARITY OF HETERO-EPITAXIAL Ba_{0.6}Sr_{0.4}TiO₃/MgO STRUCTURES. B.H. Park, Y. Fan, D.B. Jan, Q.X. Jia, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM; Y. Gim, Jet Propulsion Laboratory, Pasadena, CA; P. Lu, New Mexico Institute of Mining and Technology, Department of Materials Science and Engineering, Socorro, NM; X.Q. Pan, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI.

We have hetero-epitaxially deposited Ba_{0.6}Sr_{0.4}TiO₃ (BST) thin films on (001) MgO substrates using pulsed laser deposition. By optimizing the deposition temperature and adjusting the film thickness, we have successfully increased the nonlinearity and decreased the dielectric loss of BST films. Especially, a BST thin film grown at 750°C with a thickness of 1.1 mm had a tunability value of greater than 65% and a tunability/loss value of 43 at a surface electric field of 80 kV/cm at room temperature. X-ray diffraction (XRD) analysis showed that BST films were grown epitaxially at substrate temperatures between 650°C and 875°C. In spite of their similar epitaxial structures, tunability and dielectric loss of the BST films strongly depended on the deposition temperatures. XRD and transmission electron microscopy analyses confirmed that tunability and dielectric loss were closely related to the crystallinity of the epitaxial BST films. In addition, the variation of the BST film thickness resulted in a change of tunability as well as of the dielectric constant.

DD4.3

CONNECTION BETWEEN HIGH FREQUENCY ACOUSTIC ATTENUATION AND NEAR MILLIMETER RANGE DIELECTRIC LOSSES IN YTTRIUM-LUTECIUM GARNET SOLID SOLUTIONS. Boris M. Garin, Sergey N. Ivanov, Efim N. Khazanov, Ivan P. Nikitin, Vasiliy I. Polyakov Institute of Radioengineering & Electronics of Russian Academy of Sciences, Moscow, RUSSIA.

The connection between dielectric loss at near millimeter (NM) and neighbouring electromagnetic wavelengths range and the high frequency acoustic propagation properties in crystals follows from theory. Particularly low attenuation of high acoustic phonons are important criterion of low dielectric loss materials at that range. On the other hand, revealing and creation both of extremely low loss materials and materials with low acoustic wave (AW) attenuation play a key for various applications (high quality resonators, waveguide systems, most powerful gyrotron windows, delay lines, etc.) in the microwave technology. So the investigations of connection between mentioned dielectric and acoustic phenomena are very important. At the present work such investigations were done on the series of crystals of solid solutions of the Yttrium-Lutecium Aluminium Garnets (YLuAG) type: (Y_{1-c}Lu_c)₃ Al₅O₁₂ (c=0-1). Earlier it was found from measurements of AW attenuation in frequency range 1-10 GHz at T=78 K that in such YLuAG solutions the effective value of Grueniesen constant for longitudinal AW has a minimum at concentration c=0.25. At the present work the dielectric loss $tg\delta$ was measured at the wavelengths range 1-0.6 mm and room temperature. We used the quazioptical Teflon lens line. The source of NM electromagnetic waves was tunable Back Wave Oscillator, the receiver was cryogenic InSb bolometer. The value $tg\delta$ depends on Lu doping in Y-Lu garnet and its minimum was found at the same concentration c=0.25 as the minimum of AW attenuation (and Grueniesen constant minimum). The shapes of concentration dependencies of $tg\delta$ and AW attenuation are also qualitatively identical at whole concentration range. So the direct correlation between the dielectric loss at NM range and propagation parameters of high frequency AW in dielectric crystal is experimentally confirmed. Moreover, as it follows from comparison this results with theory, the observed dielectric losses at room temperature (tg $\delta \sim 10^{-3}$) is the intrinsic two-phonon lattice losses (due to the lattice anharmonicity) corresponding to ideal crystal and to the theoretical lower loss limit for these materials The work was supported by ISTC (grant No.1030) and Russian State Programme "Microwave Physics" (project 3.3).

DD4.4

CHARACTERISTICS OF MICROWAVE ABSORPTION OF Ni-Zn-FERRITE/SiC SEMI-CRYSTALLIZED COMPOSITES.

Tomonori Shibuya, Makoto Nakanishi, <u>Tatsuo Fujii</u>, Jun Takada, Dep. of Applied Chemistry, Okayama Univ., <u>Okayama</u>, JAPAN.

There is an increasing demand for highly efficient and wide-band microwave absorbers because of an increasing use of wireless communications in the microwave region. On this purpose we tried to prepare composite materials between the magnetic loss material (Ni-Zn-ferrite) and the dielectric loss material (SiC). Combination of two different materials with the different microwave response should make new characteristics. Polycarbosilane (PCS) was used as the precursor to prepare SiC. The heat treatment conditions before and after the mixing of PCS with Ni-Zn-ferrite could control the degree of crystallization of both materials. Ferrite/SiC mixtures were prepared by using two different ways. One is the conventional solid sate mixing of ferrite particles with PCS particles. Another is the suspension mixing of ferrite particle with dissolved PCS in ether. With increasing mixture ratio of PCS to ferrite, grain growth of ferrite particles was suppressed and the matching frequency of the microwave absorption was shifted to the higher frequency side. The sintered composites of the suspension mixture were much suppressed than those of the solid state mixture. And the matching frequency of the sintered composites of the suspension mixture was much affected. Heat treatment conditions also influenced the characteristics of the composite materials. Moderate heat treatment temperature for forming hemi-crystallized SiC between amorphous and well-crystallized SiC could properly influence the wide-band microwave absorption.

DD4.5

LOW TEMPERATURE GROWTH OF C-AXIS ORIENTED Y-TYPE HEXAGONAL FERRITES' FILMS BY POLYMERIZED COMPLEX METHOD. Tatsuo Fujii, Koutaro Komatsu, Makoto Nakanishi, Jun Takada, Dept. of Applied Chemistry, Okayama Univ., Okayama, JAPAN.

Y-type hexagonal ferrites, Ba₂Me₂Fe₁₂O₂₂ (Me₂-Y) where Me is divalent ions, are promising candidates for high-frequency microwave device applications. We successfully obtained well-crystallized Me2-Y (Me=Co, Ni, Zn) films at low temperature by polymerized complex method. The Me2-Y films with highly c-axis orientation were deposited on Ag substrates. The making procedure consisted of preparing coating solutions from Pechini-type process, depositing precursor films by a dip coating, and subsequent heat treatments ranging from 700 to 900°C for 1 hour. The films heat-treated at 700°C showed x-ray diffraction patterns of spinel oxides. After the heat treatment above 750°C formation of well-crystallized and c-axis oriented Me₂-Y films were observed, though a small amount of spinel oxides still contaminated them. The large hexagonal grain structures of Me2-Y crystals were developed with increasing the heat-treated temperature. Surface morphology of the crystallized films was not homogeneous at the moment. Magnetic properties of the $\mathrm{Me}_2 ext{-}\mathrm{Y}$ films were isotropic showing the narrow and sharp hysteresis curves.

DD4.6

FERROELECTRIC TUNING OF HTS SUPERCONDUCTING DEVICES. R. Ott, V. Goldrin, E. Hollmann, N. Klein, R. Wördenweber, Institut für Schicht- und Ionentechnik (ISI), Forschungszentrum Jülich, GERMANY.

The rapid development of mobile communication in the last few years has forced a strong demand for tunable high-Q resonators and filters for high frequencies in the GHz regime. The combination of HTS and (Ba,Sr)TiO₃ films for example provide the possibility of high quality and tuning due to the low microwave surface resistance of the HTS-material and the voltage dependent dieletric constant e of the ferroelectric layer, respectively. In this contribution we report on concepts for the integration of ferroelectric and HTS material in tunable resonator structures and the properties of sputter deposited SrTiO₃ films. SrTiO₃ films of different crystallinity, orientation and morphology were grown on sapphire and integrated into small planar Cu and YBa₂Cu₃O₇ capacities. The temperature and voltage dependence of the dielectric constant e, losses and and tunability n were determined at different frequencies. A correlation between the structural properties and conductivity on one hand and the dielectric properties e, and n on the other hand was observed. Optimised ferroelectric films proved to be suitable for tunable microwave devices, e.g. filters or phase shifters. The integration of the ferroelectric film into HTS structures was tested. In spite of the structural compatibility of both materials, problems connected to the oxygen stoichiometry of the HTS material were observed, which will be discussed.

DD4.7

AN APPARATUS FOR DETERMINATION OF MICROWAVE DIELECTRIC CONSTANT AND LOSS TANGENT AS FUNCTIONS OF TEMPERATURE AND FREQUENCY. S.C. Tidrow, A. Lee, D.D. Brickerd, D.M. Potrepka, A. Tauber, M.S. Patterson, E.D. Adler, B. Rod, U.S. Army Research Laboratory, AMSRL-SE-RE, Adelphi, MD.

An open confocal resonator has been used to measure the dielectric constant and loss tangent of perovskite oxide field-tunable materials; these measurements are reported as functions of temperature and frequency. The resonator can be used to make measurements in the frequency range from 10 to 100 GHz over a temperature range of -50°C to 100°C. In its present configuration, it can be used to provide measurements on dielectric constants ranging from 1 up to at least 300 and loss tangents from about 0.1 to 0.0001, depending upon frequency. Resonator theory and experimental configuration as well as experimental data are discussed.

Under contract with Geo-Centers, Inc. National Research Council Associate.

DD4.8

DIELECTRIC CHARACTERIZATION OF (Ba,Sr)TiO₃ FILMS AT 1-20 GHZ USING A SCANNING EVANESCENT MICROWAVE MICROSCOPE. <u>Ichiro Takeuchi</u>, University of Maryland, College Park, MD; Samuel E. Lofland, Rowan University, Glassboro, NJ.

Scanning evanescent microwave microscopy is a valuable technique in mapping out the microwave properties of voltage tunable ferroelectric/dielectric materials. The microscope consists of a high-Q co-axial resonator with a sharpened W tip attached to the center conductor protruding through an aperture at the bottom of the resonator. The interaction of the near-field electromagnetic radiation surrounding the tip with a sample placed underneath causes a shift in Q and the resonant frequency of the resonator from which real and imaginary parts of the dielectric constant of the material can be deduced. The spatial resolution of the microscope is determined by the tip radius. By monitoring the various TEM modes of the resonator, we can investigate the dielectric properties over a broad range of frequencies. We study (Ba,Sr)TiO₃ films made by various techniques under different conditions to investigate the correlation between their microstructure and the dielectric properties in the GHz range.

DD4.9

PROCESSING AND CHARACTERIZATION OF PURE AND DOPED ${\rm Ba_{1-x}Sr_xTiO_3}$ THIN FILMS FOR TUNABLE MICROWAVE APPLICATIONS. P.C. Joshi, M.W. Cole, E. Ngo, C.W. Hubbard, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, APG, MD.

Ba_{1-x}Sr_xTiO₃ thin films are being developed for high-density dynamic random access memory (DRAM) devices. The nonlinearity of its dielectric properties with respect to applied dc voltage make it attractive for tunable microwave devices such as filters, varactors, delay lines, and phase shifters. Properties of $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ are typically varied by changing the Ba/Sr ratio and/or doping. In this paper, we report on the effects of acceptor and donor doping on the microstructural and electrical properties of $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ thin films deposited by metalorganic solution deposition technique on platinum coated silicon substrates. The effects of doping on microstructure, leakage current, dielectric constant, and loss tangents have been analyzed. The structure of the films was analyzed by x-ray diffraction (XRD). The surface morphology of the films was examined by field emission scanning electron microscope (FESEM) and atomic force microscope (AFM) using tapping mode with amplitude modulation. The film/substrate interfacial characteristics were examined by RBS technique. The electrical measurements were conducted on metalinsulator-metal (MIM) capacitors using Pt as the top and bottom electrode. It was possible to significantly improve the leakage current characteristics and control the dielectric tunability by doping the $Ba_{1-x}Sr_xTiO_3$ thin films.

DD4.10

MAGNETIC LOSSES IN THICK BARIUM HEXAFERRITE FILM ON MgO (111). S.D. Yoon, C. Vittoria, Northeastern Univ, Dept of Electric and Computer Engineering, Boston, MA; S.A. Oliver, Northeastern Univ, Center for Electromagnetic Research, Boston, MA.

Thick barium hexaferrite (BaM) films are now being used for the fabrication of self-biased circulators operating at millimeter wavelengths. BaM films having thickness $<50\mu m$ were deposited onto 0.5mm MgO (111) substrates by pulsed laser ablation deposition (PLD). The BaM film properties were characterized by X-ray diffraction (XRD), vibrating sample magnetometry (VSM), and ferrimagnetic resonance (FMR) measurements over frequencies from 44 to 58GHz. The FMR linewidth for the $30\mu m$ films was $\sim 0.7 kOe$, while those of thinner $(3\mu\mathrm{m})$ films was near 0.45kOe. Portion of these films were annealed from 0.5 to 5 hours at 1000°C in order to improve the linewidth (ΔH). The narrowest ΔH of $\sim 40 \, \text{Oe}$ was found for a 3μ m film when annealed for 1 hour, while minimal improvement occured for the thicker films. However, increasing annealing time beyond 1 hour caused an increase in FMR ΔH . An examination of the effects of film stress on FMR ΔH was done by removing $300\mu m$ of MgO substrate from $30\mu m$ BaM film. The film showed a ΔH of $0.46 \mathrm{kOe}$ after the removal of substrate (compared to $\sim 0.7 \mathrm{kOe}$ with

substrate). However, the ΔH was further improved to 0.2kOe, when the same film was annealed for 1 hour. The averaged g value for all films was 1.973±0.017. The coercive fields for all the films decreased with annealing for the hysteresis loop obtained when the applied magnetic field was along the film easy axis. However, the coercive fields increased with annealing time for applied magnetic field transverse to the film easy axis. These results were attributed to strain at the interface between to the film easy axis. These results were attributed to strain at the interface between BaM layer and MgO substrate and the growth of small volumes with non c-axis textured material.

SESSION DD5: CHARACTERIZATION AND DEVICES

Chairs: Daniel E. Oates and Louise C. Sengupta Thursday Morning, November 30, 2000 Room 206 (Hynes)

8:30 AM *DD5.1

(Ba,Sr)TiO₃ CAPACITORS FOR TUNABLE HIGH FREQUENCY APPLICATIONS. J-P. Maria, F. Ayguavives, and A.I. Kingon, North Carolina State University, Department of MS&E, A. Tombak and A. Mortazawi, North Carolina State University, Department of Electrical and Computer Engineering, Raleigh, NC; G. Stauf, C. Ragaglia and J. Roeder, ATMI, Danbury, CT; M. Brand, Raytheon, Los Angeles, CA.

Provided suitably low losses, (when compared to traditional GaAs components), RF and microwave devices incorporating BST materials may provide reduced power dissipation, junction noise, operating voltage, and required area. Achieving low device losses however, is non-trivial; high quality BST and low resistance metallization are essential. We report on our recent efforts to produce high quality factor tunable BST capacitors defined by Pt top and bottom electrodes on 200 mm wafers by MOCVD. The primary goals include low loss tangent BST deposition, thick Pt metallization, device designs appropriate for the frequencies and power of interest, and an integration scheme facilitating fabrication. To date, BST films have been prepared with loss tangents of 0.003 between 100 Hz and 200 MHz. Permittivity measurements to 10 GHz reveal minimal dispersion, < 1%/decade, suggesting that loss tangents remain small to the GHz regime. At 100 MHz, tunabilities of 2:1 are commonly achieved. To simulate the performance under high power drive conditions, tunability measurements have been taken as a function of oscillator field. For a 0.1 μm to 0.5 μm BST film, the tunability is reduced by 25% when the oscillator field is increased to 250 kV/cm. Simulations indicating the importance of low resistance metallization in the context of total device losses will be presented. In addition, electrical results comparing devices incorporating 0.1 μm and 0.5 μm Pt metallization will be shown. In general, insertion losses of tunable band-pass filters can be halved when thick metallization layers are applied. Finally, a set of high frequency structures will be shown, with a discussion of the fabrication flow and the associated processing challenges.

9:00 AM DD5.2

FERROELECTRIC MATERIALS FOR Ka-BAND PHASE SHIFTERS. M.J. Muller, R.D. Brubaker, S.N. Stitzer, S.A. Gaglione, J. Talvacchio, T.M. Fertig and S.H. Talisa, Northrop Grumman Corporation, Baltimore, MD.

We will review our materials specifications for Ka-band ferroelectric phase shifters and compare them with measurements of materials available today.*Development of these specifications led us to use bulk ferroelectric materials rather than thin films and filled-waveguide configurations rather than microstrip or coplanar designs. Our specifications permit trade-offs to be made between tunability and dielectric loss, and between operating voltage and conductor losses. Based on their properties measured at lower frequencies, a number of candidate materials were selected for measurement of loss tangents and tunability at 35 GHz. Scaling of loss tangents to 35 GHz varied greatly among materials with similar properties at 10 GHz. We also evaluated materials for their time response in the range from 20 microseconds to 1 millisecond.

Supported by DARPA agreement MDA972-99-3-0005. *Materials provided by $\bar{\text{P}}$ aratek Microwave, Inc.

9:15 AM DD5.3

COHERENT AND INCOHERENT STRAIN IN SrTiO₃ AND $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ EPITAXIAL THIN FILMS. B.J. Gibbons, A.T. Findikoglu, D.W. Reagor and Q.X. Jia. Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

There has been significant interest in SrTiO₃ (STO) and $Ba_{1-x}Sr_xTiO_3$ (BST) epitaxial thin films for use in tunable microwave device applications. The majority of the research efforts on these materials have been towards maximizing the tunability of the material while maintaining a low dielectric loss. Unfortunately, in most cases it has been shown that it is not possible to do both. One method which has been shown to work is the use of a homoepitaxial buffer layer between the film and the substrate. This leads one to believe that the interfacial stress/strain state plays a critical role in determining the dielectric properties of the heterostructure. In this work, we have used the Warren-Averbach (WA) method to analyze the x-ray diffraction patterns of several films deposited via 90° off-axis rf-magnetron sputtering. This allows for the determination of the incoherent strains and crystallite sizes in these films. It is done via a deconvolution of the diffraction peak breadth for a family of peaks (e.g. 00l). Results indicate that for BST (60/40) films, the incoherent strain normal to the substrate is smaller on LAO compared to MgO for films 200 $ilde{A}$ - 3000 $ilde{A}$ thick. However, the opposite relationship holds for the coherent strains. The thickest films showed dielectric constants of 1200 and 1000 on MgO and LAO, respectively (at 1 MHz). All showed dielectric loss on the order of 0.007 with the room temperature tunability being 3 times greater for the films on MgO Correlations between the derived strains, material properties, deposition conditions, and dielectric properties will be discussed. This will allow for the determination of the critical variables in developing an optimized heterostructure.

9:30 AM <u>DD5.4</u>

THE EFFECT OF INTERNAL STRESS AND DEFECTS ON THE DIELECTRIC PROPERTIES OF (Ba,Sr)TiO₃ THIN FILMS FOR TUNABLE MICROWAVE APPLICATIONS. Hao Li, T. Tran, C.L. Canedy, S.P. Alpay, L. Salamanca-Riba and R. Ramesh, MS&E Center (MRSEC), Department of Materials and Nuclear Engineering, University of Maryland, College Park, MD; F. Van Keuls, R.R. Romanofsky and F.A. Miranda, NASA Glenn Research Center, Cleveland, OH.

Ferroelectric materials enjoy a large nonlinearity in their dielectric response, which give rise to an electric field dependent permittivity. This feature makes them particularly attractive for use as electrically tunable microwave components. However, it is still unclear how basic materials microstructure is correlated with dielectric losses and/or the degree of tunability. Our studies show that internal stress as well as defects in epitaxial $\mathrm{Ba_{1-x}Sr_xTiO_3}$ (BSTO) films play a very important role on their dielectric properties. The relationship between dielectric properties and tensile stress is well-matched to our theoretical calculations. In this study, we report the dielectric properties of BSTO films as a function of compressive stress. The dielectric properties at low frequency were measured using Au/Ti interdigitated electrodes and they are compared to the local and high frequency dielectric properties measured by novel scanning microwave microscopy. In addition, structural characterization was carried out using TEM, X-ray diffraction and AFM. The correlation between structure and electrical properties will be discussed. Furthermore, an assessment of the BSTO films at microwave frequencies was carried out by incorporating the films in an actual microwave phase shifter device based on a novel coupled microstripline phase shifter (CMPS) geometry.

This work was funded by NSF-MRSEC.

10:15 AM *DD5.5 MAGNETICALLY TUNABLE SUPERCONDUCTING FILTERS WITH FERROELECTRIC TRIMMING. $\underline{\text{D.E. Oates}}$, A.C. Anderson and G.F. Dionne, MIT, Lincoln Laboratory, Lexington, MA.

Tunable superconducting filters that employ the variable permeability of ferrite as the primary tuning means have been demonstrated at microwave frequencies. These filters show promise for implementing low-loss, rapidly tunable, narrowband devices. Tuning can be accomplished with relatively small magnetic fields, of order 100 Gauss or less, in compact low-energy structures. A three-pole $1\%\mbox{-bandwidth}$ filter with 10-GHz center frequency, 1-dB insertion loss, with greater than 10% tunability and approximately 10-μs tuning time has been fabricated to demonstrate the potential capabilities of this technology. For practical application, the hysteresis loop and the saturation magnetization of the ferrite must be optimized for cryogenic operation by altering the composition and processing from those used in the fabrication of conventional microwave ferrite materials. We will present the results of initial attempts towards optimization of the magnetic properties. In addition to the altered magnetic properties, high-Q materials must be employed because filters with more than three poles, or bandwidths less than 1%, and insertion loss less than 1 dB require resonator Q factors at low temperatures between 10^4 and 10⁵. These values have been demonstrated with high-purity single crystals, but presently the Qs of the conventional ferrites limit the performance of such filters. We will discuss the loss mechanisms, including the role of paramagnetic impurities and material microstructure, and show how to achieve the required Qs. Furthermore, filters with greater than three poles will require trimming of the individual resonator frequencies to maintain the

desired frequency response over the full tuning range. We will present the use of lightly coupled strontium titanate (STO) films to accomplish the trimming. By lightly coupling the STO to the resonators, the measured $K=2Q\Delta~f/f$ of 40 for the STO will not degrade the resonator Q, yet provide approximately 0.1% frequency trimming.

*This work was supported by DARPA.

10:45 AM $\underline{DD5.6}$

PARAELECTRIC COMPOUNDS BASED ON SUBSTITUTED BARIUM-TITANATE. A. Tauber, S.C. Tidrow, D.M. Potrepka, D.D. Brickerd, B. Rod, A. Lee, M.S. Patterson, E.D. Adler, U.S. Army Research Laboratory, AMSRL-SE-RE, Adelphi, MD.

Cubic compounds exhibiting paraelectric behavior have been identified in systems of solid solutions between BaTiO₃ and perovskites that are dielectrics. Over the composition ranges studied, many compounds are cubic and their lattice parameters obey Vegard's Law. Many compositions exhibit ferroelectric transition below room temperature. The variation of dielectric constant as functions of applied voltage and temperature are reported. In addition, microwave properties of some samples are reported. Under contract with Geo-Centers, Inc.

D.M. Potrepka is a National Research Council Associate.

11:00 AM DD5.7

BST BASED COMPOSITE THIN FILMS FOR TUNABLE MICROWAVE DEVICES WITH REDUCED CONTROL VOLTAGES. T.V. Rivkin, D.S. Ginley, C.M. Carlson, P.A. Parilla, J.D. Perkins, National Renewable Energy Laboratory, Golden, CO; L.C. Sengupta, L. Chiu, X. Zhang, S. He, W. Chang, Paratek Microwave, Inc., Columbia, MD.

Ba Sr₁₋ TiO₃ (BST) based composite thick films produce excellent performance tunable microwave devices. Coplanar phase shifters with a high figure of merit (120 deg/dB at 30 GHz) were fabricated using such composite films. A major limitation of the existing technology is the high bias voltages (~500 Volts) required to control the composite tunable elements. Reducing the control voltages down to 20 V (semiconductor industry standard) would require a reduced ($\sim 4 \mu m$) electrode spacing. However, the large surface roughness ($\sim 2~\mu m$) of these composite thick films does not allow fabrication of such fine electrode features in the coplanar configuration. To overcome these difficulties, work has begun to develop thin composite films with both the superior microwave tuning properties of the thick film composites and a smooth surface texture. These latter films, grown by Pulsed Laser Deposition, are substantially smoother ($\sim 300 \text{ Å vs. } 2 \text{ } \mu\text{m}$). Furthermore, they demonstrate a tunability similar to that of the thick films ($\sim 30\%$ at $5 \text{V}/\mu\text{m}$). Progress in the development of the thin-film tuning elements with closely spaced (less than 4 $\mu \mathrm{m})$ top electrodes will be reported. Microwave performance of such tuning elements in the 10 - 30 GHz range will be characterized. Trilayer devices with a composite thin film sandwiched between electrode layers will also be evaluated as a means to overcome the resolution limits of the photolithography process.

11:15 AM *DD5.8

TUNABLE DIELECTRIC MATERIALS FOR USE IN WIRELESS PRODUCTS. Louise C. Sengupta, Paratek Microwave, Inc., Columbia, MD.

Bulk, thick and thin film composite tunable dielectric materials have been fabricated. These multiphase composites show low insertion loss and high tunability. The dielectric constant of the materials can be tailored to a particular application. The most recent 24 GHz results of our tunable dielectric composites indicate that a Figure of Merit, FOM, defined as the insertion loss / 360 degrees of phase shift, of 1.8 dB/360 degrees can be achieved. Also, composite thin films have a Q (1/loss tangent) of over 200, measured at 8 GHz, with a tunability (%) of 20% at an electric field strength of 27 V/micron. Additionally, these thin films were deposited on low cost alumina substrate. Tunable filters and phase shifters have been fabricated using these materials at various frequency ranges. These devices have low insertion loss, fast switching speeds, and high RF power handling capability. High frequency data for both the materials and the devices will be presented.

11:45 AM DD5.9

TUNABILITY IN $\mathrm{Ba_{1-x}Sr_xTiO_3}$ -BASED FERROELECTRICS. D.M. Potrepka and S.C. Tidrow, US Army Research Lab, Sensors and Electron Devices Directorate, Adelphi, MD; A. Tauber, Geocenters, Inc., MA.

Ferroelectrics are presently of interest for RF-tunable devices such as phase shifters and true time delay for electronic scanning antennas. For low cost, low power consumption, and to improve device performance, materials must have high tunability, low dielectric

constant, and low loss tangent. Using existing predictive techniques, compositions of Ba_{1-} Sr TiO3-based ferroelectrics with charge-compensated substitutions for Ti^{4+} were synthesized. Results of capacitance measurements are used to obtain dielectric constant and tunability in the paraelectric (T>T) regime. The relevance to device requirements will be discussed. Results for substituted samples are compared to those for (unsubstituted) Ba_{1-} Sr TiO3. Discussion of the impact of the results on predictive techniques for tunability will be addressed.

DMP acknowledges support from National Research Council Associateship Program.

SESSION DD6/CC14: JOINT SESSION HIGH-FREQUENCY APPLICATIONS OF FERROELECTRICS

Chairs: Quanxi Jia and Jon-Paul Maria Thursday Afternoon, November 30, 2000 Room 312 (Hynes)

1:30 PM *DD6.1/CC14.1

MICROSTRUCTURE AND CHEMISTRY OF NON-STOICHIOMETRIC (Ba,Sr)TiO₃ THIN FILMS. <u>Igor Levin</u>, NIST, Gaithersburg, MD; Richard Leapman, NIH, Bethesda, MD; Debra Kaiser, NIST, Gaithersburg, MD.

The microstructure and chemistry of $(Ba,Sr)TiO_3$ thin films deposited on $Pt/SiO_2/Si$ substrates by metalorganic chemical vapor deposition were studied using high-resolution transmission electron microscopy and quantitative spectrum imaging in electron energy loss spectroscopy. The grain boundaries in all films with overall Ti content ranging from 50.7% to 53.4% exhibit a significant increase in Ti/Ba ratio as compared to the grain interiors. The results suggest that the deviations of Ti/(Ba Sr) ratio from the stoichiometric value of unity are accommodated by the creation of Ba/Sr vacancies which segregate to the grain boundary regions. The films with Ti contents equal to or greater than 52% additionally contained an amorphous Ti-rich phase at some grain boundaries and multiple grain junctions; the amount of this phase increases with increasing overall Ti content. The analysis indicates that the amorphous phase can only partially account for the significant drop in dielectric permittivity accompanying increases in the Ti/(Ba Sr) ratio.

2:00 PM DD6.2/CC14.2

MICROSTRUCTURAL AND ELECTRICAL PROPERTIES OF La DOPED ${\rm Ba_{1-}}_x{\rm Sr_xTiO_3}$ THIN FILMS FOR TUNABLE DEVICE APPLICATIONS. M.W. Cole, P.C. Joshi, E. Ngo, C.W. Hubbard, R.L. Pfeffer, M.H. Ervin, M.C. Wood, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, APG, MD.

 $Ba_{1-x}Sr_xTiO_3$ (BST) is a promising ferroelectric material for tunable microwave device applications such as electronically tunable mixers. delay lines, filters and phase shifters. Pure BST in thin film form offers tunabilities upward of 50% at bias voltages of less than $10~\mathrm{V},$ which is compatible with the voltage requirements of present semiconductor based systems. Unfortunately, the tradeoff to high tunabilities are high loss tangents, that is, tan $\delta > 0.01$. It is well documented that small concentrations of dopants can dramatically modify the properties of ferroelectric materials such as BST. In this work we investigated the materials and dielectric properties of $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ solid solution thin films doped with La. The films were fabricated via metalorganic solution decomposition (MOSD) technique. The 0 to 20 mol% La doped films were characterized for materials, dielectric, and insulating properties. Specifically, x-ray diffraction (XRD) was used to assess film crystallinity, phase formation, and film orientation. Atomic force microscopy (AFM) and field emission scanning electron microscopy (FESEM) were employed to access surface morphology. Cross-sectional transmission electron microscopy (X-TEM), combined with energy dispersive spectroscopy (EDS) analysis, was used to detail the film microstructure and film-substrate interfacial properties. RBS was employed to access film stoichimetry. The electrical measurements were conducted on films in the MIM capacitor configuration. Our results show that La doping of BST results in a reduction of the film dielectric constant, loss tangent, and leakage current. The dielectric and insulating properties will be discussed and correlated to film microstructure, crystal structure, and the quality of the electrode-film interface in order to determine the trade-offs between material quality and potential device performance.

2:15 PM DD6.3/CC14.3

LINKING THE DIELECTRIC RESPONSE OF CERAMIC AND THIN FILM ${\rm Ba}_x{\rm Sr}_{1-x}{\rm TiO}_3$: THE EFFECTS OF STRAIN. Chris M. Carlson, Dept of Physics, Univ of Colorado, Boulder, CO; Philip A. Parilla, Tanya V. Rivkin, John D. Perkins and David S. Ginley, National Renewable Energy Laboratory, Golden, CO.

Ba Sr₁₋ TiO₃ (BST) has long been studied for its potential in a wide array of applications, including tunable microwave and RF devices as well as high-density memories (DRAM). A factor that has limited the performance of devices based on thin film BST and other materials is the differences in dielectric response between bulk ceramic materials and thin films. These differences are typically ascribed, in part, to the presence of residual strain in the film-substrate heterostructures. Here we present the correlation of residual strain, as measured by x-ray diffraction, with dielectric response in epitaxial BST (x=0.4) films grown on MgO substrates by pulsed laser deposition. Using a theory recently developed by Pertsev, et. al., we model the temperature, electric field, and strain dependence of the dielectric constant for a series of BST films with varying biaxial and hydrostatic strain. Both the biaxial and hydrostatic strain components produce linear shifts in the transition temperature and, therefore, the entire temperature dependence. A second effect of the hydrostatic strain is the suppression of the overall dielectric response due to an electric field that is internally generated by growth-induced defects. By means of these effects, the dielectric response of the entire series of BST films can be seen as strain-induced modifications of the bulk response. That is, our modeling shows strain to link the dielectric response of these films with that of the bulk material.

2:30 PM DD6.4/CC14.4

STRESS EFFECTS ON DIELECTRIC PROPERTIES OF EPITAXIAL Ba $_{0.6}$ Sr $_{0.4}$ TiO $_{3}$ THIN FILMS. B.H. Park, E.J. Peterson, Q.X. Jia, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM; J. Lee, Sung Kyun Kwan University, Department of Materials Engineering, Suwon, KOREA; X.X. Xi, The Pennsylvania State University, Department of Physics, University Park, PA.

Dielectric properties of $Ba_{1-x}Sr_xTiO_3$ thin films are strongly dependent on their compositions, microstructures, and stress states. Recently, many researchers have tried to investigate stress effects on the dielectric properties of $Ba_{1-x}Sr_xTiO_3$ thin films. Basic approaches to change the stress include the variation of substrates or variation of deposition conditions, such as oxygen pressure. Here we report our approach to manipulate the stress in the film by inserting a strain layer between the substrate and the main body of the film. In detail, we deposited epitaxial $\mathrm{Ba_{0.6}Sr_{0.4}TiO_3(BST)}$ thin films on ${
m MgO(001)}$ substrates by inserting a very thin ${
m Ba_{1-x}Sr_xTiO_3}$ (x = 0.1 - 0.7) interlayer to manipulate the stress of the BST films. Since the only difference of those epitaxial BST films is the lattice constant of the buffer layers, we are very successful in controlling the stress of the BST films. We measured the dielectric properties of those films using coplanar capacitor structures at a 1 MHz frequency. BST films under small tensile stress showed larger dielectric constants and tunability values than those without stress as well as those under compressive

2:45 PM DD6.5/CC14.5

 $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\overline{\mathrm{TiO}_3}$ THIN FILM CAPACITORS WITH COPPER ELECTRODES. <u>Jaemo Im</u>, O. Auciello, Materials Science Division, Argonne National Laboratory, Argonne, IL; A.R. Krauss, Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne II.

 $\mathrm{Ba}_{1-x}\mathrm{Sr}_x\mathrm{TiO}_3$ (BST) films are being intensively investigated for application as capacitor materials for Gbit DRAMs and as high frequency tunable devices. Pt is typically used as an electrode material for BST capacitors, because of its oxygen resistance. In the past few years, Cu has been investigated as an interconnect material for deep sub-micron integrated circuits (IC) due to its low resistivity and high electromigration resistance. Using Cu to replace the conventional Pt as electrodes for BST thin film based capacitors will lead to the low R-C delay for advanced ICs and to reduce conductor losses for high frequency devices. A key issue for the integration of Cu into BST thin film based capacitors is to prevent the oxidation of Cu electrodes at the interface during the BST deposition process. Our approach for this issue is to deposit initial thin BST layer at room temperature with conducting barriers between BST and Cu electrodes. Initial characterization of the Cu/BST/Cu capacitors grown on silicon substrates, using magnetron sputter-deposition, show very low losses (tan $\delta\sim0.002,\,\rm Q{>}500)$ with 25% tunability, measured at room temperature and 1 MHz. The effect of barrier electrode layer thickness and BST deposition temperatures, on the electrical properties of BST thin film capacitors will be discussed.

3:30 PM *DD6.6/CC14.6

MECHANISMS OF LOSS AND TUNABILITY OF FERRO-ELECTRIC THIN FILMS PROBED BY LATTICE DYNAMICAL STUDIES. X.X. Xi, A.A. Sirenko, A.M. Clark, Weidong Si, The Pennsylvania State University, Dept of Physics, University Park, PA

Lattice dynamics is of central importance for the ferroelectric as well as dielectric properties of ferroelectric materials. For tunable devices

using ferroelectric thin films, lattice dynamical studies provide important insights into the mechanisms of dielectric nonlinearity and losses. Through Raman scattering, with and without bias electric field, and infrared ellipsometry studies in SrTiO₃ films grown by pulsed laser deposition, we find that the soft mode in the films is harder than that in bulk crystals. This observation is in agreement with the Lyddane-Sachs-Teller formalism to explain the low dielectric constant in the films. The existence of local polar regions is proposed as a critical factor determining the dielectric properties of ferroelectric thin films such as tunability and loss.

4:00 PM DD6.7/CC14.7

ELECTRIC-FIELD-INDUC ED RAMAN SCATTERING OF SrTiO₃ AND Ba_{1-x}Sr_xTiO₃ THIN FILMS. A.A. Sirenko, I.A. Akimov, A.M. Clark, K. Chen, X.X. Xi, Department of Physics, Pennsylvania State University. University Park. PA.

We have performed Raman scattering experiments on SrTiO₃ (STO) and $\mathrm{Ba_{1-x}S_{1-x}S_{1}}$ (BST) thin films grown by pulsed laser deposition. Electric-field-induced Raman scattering was studied using indium-tin oxide(ITO)/SrO/SrRuO₃ and ITO/BST/SrRuO₃ structures in a parallel plate capacitor configuration. Temperature dependence of the phonon frequencies reveals a soft-mode in films higher in frequency than found in bulk crystals. This effect is explained by the existence of local polar regions induced by oxygen vacancies in the thin films. The local polarization due to these defects is the basis for the difference in soft mode frequencies in films and bulk crystals. Electric field measurements demonstrate hardening of the soft mode under applied electric fields, which is consistent with tuning of the static dielectric constant described by the Lyddane-Sachs-Teller (LST) relationship.

4:15 PM DD6.8/CC14.8

PSEUDO-SPIN FLOP TRANSITION IN OXYGEN-DEPRIVED BST THIN FILMS. <u>Charles Hubert</u>, Jeremy Levy, Dept of Physics and Astronomy, University of Pittsburgh, Pittsburgh, PA; Chris M. Carlson, Department of Physics, University of Colorado, Boulder, CO; Philip A. Parilla, Tanya V. Rivkin, John D. Perkins, David S. Ginley, National Renewable Energy Laboratory, Golden, CO.

Time-resolved confocal scanning optical microscopy is used to probe the local microwave dynamics of ferroelectric nanoregions in Oxygen-deprived $Ba_{0,4}Sr_{0,6}TiO_3/M\,gO$ thin films. The films are grown using pulsed laser deposition in an O_2 pressure ranging between 40 mT-250 mT. The biaxial strain is observed through x-ray measurements to cross from in-plane to out-of-plane at a growth O_2 pressure of approximately 85 mT. Both the in-plane and out-of-plane components of the ferroelectric polarization response at microwave frequencies are monitored as a function of a static electric field. Significant dielectric dispersion is observed in the out-of-plane ferroelectric response for the 85 mT sample, indicating the "softness" of this mode. Application of an in-plane field quenches the out-of-plane response. We attribute this behavior to a pseudo-spin flop transition in which the out-of-plane polarization (regarded as a pseudo-spin degree of freedom) reorients due to the externally applied electric field.

This work is supported by DOE contract no. DE-AC36-99G010337 (NREL) and by ONR contract N00173-98-1-G011 (U. Pittsburgh).

4:30 PM DD6.9/CC14.9

EXPERIMENTAL AND THEORETICAL INVESTIGATION INTO THE DIELECTRIC BEHAVIOUR OF FERROELECTRIC THIN FILM SUPERLATTICES. Marty Gregg, Deirdre O'Neill, Gustau Catalan and Robert Bowman, Queens University Belfast, Department of Pure and Applied Physics, Belfast, Northern Ireland, UNITED KINGDOM.

In an attempt to reproduce the physics and dielectric properties of relaxor electroceramics, pulsed laser deposition was used to fabricate thin film capacitor structures in which the dielectric layer is composed of a superlattice of $Ba_{0.8}Sr_{0.2}TiO_3$ and $Ba_{0.2}Sr_{0.8}TiO_3$. The properties of the capacitors were investigated as a function of superlattice periodicity. The dielectric constant was enhanced at stacking periodicities of several unit cells, consistent with relaxors. Enhancement was also associated with relaxation phenomena. However, such properties were found to be associated with relatively high dielectric loss. Analysis of the imaginary permittivity showed that fine scale superlattices conform to Maxwell-Wagner behaviour, indicating that 'relaxor-like' features may well be an artifact of increased carrier mobility. Comparison of data with that already published on superlattices suggests that previous claims of dielectric enhancement in superlattices may also be attributed to Maxwell-Wagner behaviour. In addition, a novel temperature-dependent transport model has been developed, which shows that all dielectric features of superlattice structures observed experimentally to date can be reproduced by Maxwell-Wagner formalism.

4:45 PM DD6.10/CC14.10

EFFECT OF THE LASER ENERGY AND NUMBER OF LASER PULSES ON THE MICROSTRUCTURE, COMPOSITION AND PROPERTIES OF BARIUM STRONTIUM TITANATE THIN FILMS SYNTHESIZED BY PULSED LASER DEPOSITION. C.G. Fountzoulas, J.D. Demaree, Army Research Laboratory, Weapons and Materials Research Directorate, Army Research Laboratory, APG, MD.

Thin films of novel barium strontium titanate (BSTO), deposited by the pulsed laser deposition (PLD) technique exhibit excellent electronic properties including tunable dielectric constants and low electronic loss. The microstructure of the film influences the electronic, and mechanical properties (internal stresses and adhesion), important factors affecting the mechanical integrity and reliability of a device made of these thin films, which in turn influences the performance of the film. The BSTO thin films were synthesized at ambient temperature and 30 mT oxygen partial pressure, with 300, 400 and 500 mJ laser energy at 5, 10 and 20 pulses per second on silicon wafer substrates. All films were subsequently post-annealed at 750°C in an continuous oxygen stream. The thickness of the films increased with increasing laser energy and increasing pulse number. In particular, the thickness of the films synthesized at the same laser energy increased linearly with increasing pulse rate, while the thickness of the films synthesized at the same laser pulse rate increased non-linearly with increasing laser energy. The crystallinity and lattice constant variation of all films were studied with the aid of x-ray diffraction analysis. All post annealed films were crystalline. The microstructure of the films and particulate density were examined with the aid of optical microscopy, scanning electron microscopy (SEM) and FT-Raman spectroscopy. The film stoichiometry, and in particular the oxygen composition prior and after annealing, were studied with the aid of a Rutherford backscattering technique. The nanohardness, modulus of elasticity, cohesion and adhesion and wear properties of the films were studied with the aid of a nanohardness indenter and a ball-on-disk tribometer. The electronic, mechanical, physical properties and an initial microstructural zone model of these films will be discussed as a function of the laser energy and pulse rate. These results will be combined with the results of our previous work on the effect of substrate temperature and oxygen partial pressure on the microstructure and properties of the BSTO films.