

# SYMPOSIUM H

## Materials Issues for Tunable RF and Microwave Devices III

April 2 – 3, 2002

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as Volume 720  
of the Materials Research Society  
Symposium Proceedings Series

\* Invited paper

## SESSION H1: BST FILMS AND APPLICATIONS I

Chairs: James S. Horwitz and Xiaoxing Xi

Tuesday Morning, April 2, 2002

Salon 3/4 (Marriott)

### 8:00 AM \*H1.1

WILL FAME BRING FORTUNE? AN UPDATE OF THE DARPA FREQUENCY AGILE MATERIALS FOR ELECTRONICS PROJECT. Stuart Wolf, DARPA/DSO, Arlington VA and the Naval Research Laboratory, Washington, DC.

DARPA FAME has had several projects to improve the properties (lower loss, higher tuneability) of paraelectric and ferrite frequency agile materials. The materials state of the art has improved to the point that several important communication and remote sensing applications have become very viable using these materials. I will describe the results of this program to date and indicate what has been achieved, where the applications are and how soon we may see both DoD and commercial products.

### 8:30 AM \*H1.2

SCIENCE AND TECHNOLOGY OF BARIUM STRONTIUM TITANATE THIN FILMS AND THEIR INTEGRATION INTO HIGH FREQUENCY TUNABLE DEVICES. O. Auciello, S. Saha, D.Y. Kaufman, S.K. Streiffer, J. Im, P. Baumann, R.A. Erck, Argonne National Laboratory, R.A. York, B. Acikel, T. Taylor, P. Hansen, J.S. Speck, University of California Santa Barbara, Santa Barbara, CA.

We have performed extensive work in the last three years on the synthesis and characterization ( $\text{Ba}_x\text{Sr}_{1-x}\text{Ti}_{1+y}\text{O}_{3+z}$ ) (BST) films and their integration into capacitors for high frequency tunable devices. High performance / high frequency voltage tunable devices require the synthesis of BST film based-capacitors with high tunability as well as low dielectric, conductor and substrate losses. A review of our work will be presented including a critical comparison between the MOCVD and sputter-deposition methods to produce device-compatible BST films and integration with different electrode layers (e.g., Pt and Ir) and substrates (e.g., Si and glass) to produce BST capacitors with high tunability and low losses and leakage. BST thin-films were developed with properties that are especially attractive for application to high-frequency devices, including: (a) high dielectric constant (200-300): useful for small-area bypass capacitors and MEMS switches, (b) up to 4:1 variation in permittivity or capacitance as a function of applied voltage, useful for tunable RF circuits such as phase-shifters, filters, and (VCOs), (c) fast polarization response permitting rapid tuning and frequency conversion devices, and (d) high breakdown field ( $>2 \times 10^6$  V/cm) allowing for large bipolar voltage swings and hence good power handling. These properties suggest that BST-based voltage-variable capacitors (varactors) can provide a compelling alternative to semiconductor-based varactors. Very low cost and high-performance microwave and millimeter-wave varactor circuits can be fabricated using modern high-volume thin film deposition techniques such as MOCVD and magnetron sputtering in conjunction with low cost substrates such as glass. Numerous challenges remain, including identifying microwave loss mechanisms and methods for controlling loss; developing reliable and compatible electrodes and fabrication processes; improving device reliability and lifetime. A review past and projected work will be presented. \*Work supported by the DOE, Office of Science and Office of Advanced Automotive Technologies under Contract W-31-109-ENG-38, and by DARPA-FAME Program.

### 9:00 AM \*H1.3

TUNABLE  $\text{BaTiO}_3$ - $\text{SrTiO}_3$  AND  $\text{PbTiO}_3$ - $\text{SrTiO}_3$  DIELECTRIC FILMS BY MOCVD: NANOSTRUCTURE EVOLUTION AND PROPERTIES. Sandwip K. Dey, Arizona State University, Department of Chemical and Materials Engineering, & Department of Electrical Engineering, Tempe, AZ.

High Q and voltage tunable dielectric thin films are required for components such as a low loss, compact phase shifters for T/R modules used in phased array radars, or wide-band, low loss tunable filters for receivers. To accomplish this, a reliable vapor deposition process is necessary. A MOCVD process, for the deposition of Ba-Sr and Pb-Sr based perovskite oxide thin films on Si and Sapphire substrates, is developed through the understanding of nanostructure evolution and interfacial phenomenon. For guidance in carrying out thermal MOCVD experiments, thermodynamic phase stability diagrams were calculated for the Ba/Pb-Sr-Ti-O-C-Ar-H systems. The Sr/Pb/Ba(thd)<sub>2</sub> and Ti(t-BuO)<sub>2</sub>(thd)<sub>2</sub> precursors were dissolved in THF solvent, and Ar was used as the carrier gas to deliver the vaporized precursors into the reactor, while oxygen was used as the reactant gas. The SrTiO<sub>3</sub>, BaTiO<sub>3</sub>-SrTiO<sub>3</sub>, and PbTiO<sub>3</sub>-SrTiO<sub>3</sub> films (15-100 nm) were deposited on metal-passivated Si and Sapphire substrates by MOCVD at rates of 8-22 nm/min. The nanostructure evolution of polycrystalline and epitaxial films were determined using RBS, FESEM, XRD, HRTEM, and Raman Spectroscopy. This paper

will discuss the mechanism of nanostructure evolution, and its effect on the temperature, frequency, and voltage dependence of the dielectric properties.

### 9:30 AM \*H1.4

HIGH FREQUENCY VARACTORS USING THIN-FILM BST: DEVICE APPLICATIONS AND MATERIALS CHALLENGES. Robert York, Baki Acikel, Department of Electrical and Computer Engineering, University of California, Santa Barbara, CA; Troy Taylor, Pete Hansen, James Speck, Materials Department, University of California, Santa Barbara, CA; Chris Elsass, Agile Materials and Technologies Inc., Goleta, CA.

BST thin-films have been developed with properties that are especially attractive for high-frequency applications, including: 1) High dielectric constant (200-300): useful for small-area bypass capacitors and MEMS switches; 2) Field-dependent permittivity: as much as 4:1 variation in permittivity or capacitance as a function of applied voltage, useful for tunable RF circuits such as phase-shifters, filters, and (VCOs); 3) "Fast" polarization response: allows for rapid tuning and frequency conversion devices; 4) High breakdown field: typically  $>1\text{MV/cm}$ , allowing for large bipolar voltage swings and hence good power handling. These properties suggest that BST-based voltage-variable capacitors (varactors) can provide a compelling alternative to semiconductor-based varactors. These find use in numerous modern high-frequency circuits and applications. An important feature of the high dielectric constant BST thin film technology is the wide variety of substrate materials available for thin-film deposition. Very low cost and high-performance microwave and millimeter-wave varactor circuits can be fabricated using modern high-volume thin film deposition techniques such as magnetron sputtering in conjunction with low cost insulating substrates. At the present time, this potential is largely unrealized commercially, but rapid progress has been made both in thin film technology and device architecture. Numerous challenges remain, including identifying microwave loss mechanisms (extrinsic and intrinsic) and methods for controlling loss; developing reliable and compatible electrodes and fabrication processes for high volume circuit integration; improving device reliability or lifetime. In this talk, we will discuss progress in the areas described above, as well as numerous potential microwave applications. A focal point of the work at UCSB has been the development of low-loss phase shifters for microwave and millimeter-wave applications, and we will report on devices with  $>90$  deg/dB figures-of-merit.

### 10:00 AM H1.5

DIRECT WRITE PROCESSING FOR TUNABLE MICROWAVE DEVICES. Tanya Rivkin, Calvin Curtis, Alex Miedaner, John Perkins, Jeff Alleman and David Ginley, National Renewable Energy Laboratory, Golden, CO.

We report on the application of ink jet printing techniques to high frequency devices based on barium strontium titanate (BST) and the associated metalizations. A key issue in developing frequency agile circuitry is the ability to develop low cost production techniques and still maintain high quality thin film devices. Ink jet printing of metal organic and nanoparticulate precursors potentially offers the ability to deposit high quality materials with spatial resolution. Coupled with direct write metalizations this approach can potentially improve performance while reducing cost. We report on the use of a variety of MOD precursors with and without nanoparticle additions to directly form high quality BST. Composite BST/MgO thin films with 60% percent tuning and  $0.007 \tan \delta$  at 2 GHz were deposited using Metal Organic Decomposition (MOD) by spin coating on single crystal MgO substrates. Films with approximately 1 mol% of MgO had a better tuning/loss ratio than 10 mol% MgO substituted or pure BST films. BST films deposited by ink-jet printing of metalorganic precursors had structural properties similar to those of the spin-coated films. Both printed and spin-coated films had the desired smooth granular surface required for high electrode resolution. Metalizations have been direct written with line resolutions below  $100 \mu\text{m}$  and conductivity near that of vacuum deposited materials. We will discuss the high frequency properties of the direct written materials and devices.

### 10:45 AM \*H1.6

HIGH FREQUENCY APPLICATIONS OF LOW LOSS, TUNABLE BARIUM STRONTIUM TITANATE THIN FILMS. Gregory T. Stauf, Craig Ragaglia, Jeffrey F. Roeder, ATMI, Danbury, CT; Jon-Paul Maria, Jin Zhang, Angus Kingon, North Carolina State University, EGRC, Raleigh, NC; Amir Mortazawi, Ali Tombak, University of Michigan, EECS Building, Ann Arbor, MI; Tito Agyuavives, formerly NCSU, now at Coherent Inc., Hoboken, NJ; Michael Brand, Raytheon Systems Company, El Segundo, CA.

Thin film barium strontium titanate (BST) shows great promise for voltage tunable dielectric devices or "varactors" for use at RF and microwave frequencies. An MOCVD process is described for

production of BST that gives very low losses ( $\tan \delta$  as low as 0.002-0.004) in thin film materials, while retaining large ( $\Delta C/C > 50\%$ ) tunabilities at low operation voltages. Applications of these tunable layers to individual tunable capacitors, as well as in circuits such as tunable filters will be discussed. Emphasis will be placed on the importance of electrodes in determining total device loss at high frequencies, and solutions developed for integration of BST with thick electrodes to make very small parallel plate capacitors for high frequency use. Successful combination of these processes have allowed production of devices with dielectric Q factors, the inverse of loss or  $\tan \delta$ , over 250 at 100 MHz ( $\tan \delta \sim 0.004$  as measured and modeled by S-parameters) and as high as 100 at 10 GHz ( $\tan \delta \sim 0.01$ ). Overall device Q factors were over 50 at 30 MHz, comparable to or better than mature semiconductor varactor technology for similar large capacitance values. Low (<10%) dispersion of dielectric constant with frequency was seen up to 10 GHz. Tunable low pass and bandpass filters based on these varactors were also designed and fabricated. Tunabilities of 50% and low insertion losses ( $\sim 3$  dB, with BST accounting for less than half this loss) at VHF frequencies were achieved. The effects of film thickness, growth temperature, and stoichiometry on electrical properties at low (10 kHz) and high (100-10,000 MHz) frequencies will be discussed, as well as issues relating to temperature sensitivity, repeatability and manufacturability for these devices.

#### 11:15 AM \*H1.7

EPITAXIAL GROWTH AND INTERFACE STRUCTURES OF PEROVSKITE (Ba,Sr)TiO<sub>3</sub> THIN FILMS. C.L. Chen, Y. Lin, T. Garrett, The Texas Center for Superconductivity and Department of Physics, University of Houston, Houston, TX; J.C. Jiang, E.I. Meletis, Mechanical Engineering Department, Louisiana State University, Baton Rouge, LA; F.A. Miranda, Glenn Research Center, Communications Technology Division, Cleveland, OH.

Ferroelectric (Ba,Sr)TiO<sub>3</sub> and Nb doped (Ba,Sr)TiO<sub>3</sub> thin films have been epitaxially grown on (001) MgO and (001) LaAlO<sub>3</sub> using pulsed laser ablation. Microwave phase shifters were developed from the as-grown films with excellent room temperature dielectric properties of high dielectric constant, low loss tangent, and large tunability. The microwave property measurements indicated that the room temperature coupled microwave phase shifter has achieved a phase shift over 275° at 23.675 GHz and a figure of Merit of near 70°/dB. X-ray diffraction and pole figure reveal that the as-grown films are c-axis oriented and have good single crystal quality. The RBS studies suggest the films have excellent epitaxial behavior with a minimum yields of about 3%. Cross-sectional and plan-view transmission electron microscopy studies confirm that the as-grown films are excellent single crystalline and have sharp interface. For the films grown on (001) MgO, the thinner films (thickness less than 100 nm) have a flat surface but the thicker one rough (rms roughness  $\sim 15$  nm). The BSTO films exhibit a single crystal cubic structure with an interface relationship of (001)<sub>BST</sub> || (001)<sub>SUB</sub> and  $\langle 100 \rangle_{BST} \parallel \langle 100 \rangle_{SUB}$  with respect to the substrates of MgO or LaAlO<sub>3</sub>. The lattice mismatch between (Ba,Sr)TiO<sub>3</sub> and MgO was completely released at the interface by forming equally spaced misfit dislocations, which can be observed from the high-resolution transmission electron microscopy. On the other hand, the conservative and non-conservative antiphase boundaries can form in the thin films due to the "surface steps and terraces induced antiphase domain formation mechanisms". Thus, Artificial Domain Structured Optoelectronic Modulators is proposed from this epitaxial model. Details will be presented in the talk.

#### 11:45 AM H1.8

THE TEMPERATURE DEPENDENCE OF BST CAPACITORS FOR TUNABLE HIGH FREQUENCY APPLICATIONS. A.I. Kingon, C.B. Parker, F. Agyavives, J-P. Maria, North Carolina State University, Materials Science and Engineering, Raleigh, NC; G. Stauf, C. Ragaglia, J. Roeder, ATMI, Danbury, CT.

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. The temperature dependence of these components is, however, an issue of concern. The strongly temperature dependent properties of bulk ferroelectric materials, especially near the transition temperature are well known. In thin films these transitions become diffuse, and the temperature dependence is reduced - this behavior is commonly referred to as crystal size effects. We report on recent investigations of BST thin films deposited by MOCVD, specifically targeting the temperature dependence of the permittivity, the loss tangent, and the tunability. All measurements are taken as a function of film thickness and applied voltage over a thickness range spanning 15 nm to 600 nm. In this manner, a complete set of data has been generated to describe the dielectric function of BST. This data has been used to determine the optimal figure of merit for MOCVD BST. In general, the magnitude of the temperature dependence can be reduced from

approximately 50% to 15% by reducing film thickness from 600 nm to 15 nm. This reduction comes at the similar expense in reduced total tunability. In addition, thinner films exhibit lower loss tangent values and can be attributed to the proximity in temperature of the ferroelectric transition.

## SESSION H2: BST FILMS AND APPLICATIONS II

Chairs: David S. Ginley and Robert A. York

Tuesday Afternoon, April 2, 2002

Salon 3/4 (Marriott)

### 1:30 PM \*H2.1

INVESTIGATIONS ON SOL-GEL DERIVED TRANSITION METAL DOPED BST THIN FILMS FOR PHASE SHIFTER APPLICATIONS. R.S. Katiyar, M. Jain, S.B. Majumder, Department of Physics, University of Puerto Rico, San Juan, PR; R.R. Romanofsky, F.W. Van Keuls, and F.A. Miranda, National Aeronautics and Space Administration, Glenn Research Center, Cleveland, OH.

Highly (100) oriented manganese (Mn) doped Ba<sub>0.5</sub>Sr<sub>0.5</sub>TiO<sub>3</sub> (BST) thin films were prepared on (100) LaAlO<sub>3</sub> substrates by chemical solution deposition technique. The degree of texturing and quality of the in-plane epitaxy, characterized by X-ray pole figure analysis and RBS channeling measurements, were found to be improved with Mn doping of up to 3 at%. The broad nature of the dielectric maximum and its shift towards the higher temperature in thin films as compared to the bulk counterpart due to the finer grain size and/or unrelieved strain of the annealed films. The dc field dependent dielectric constant and loss tangent (measured at 1 MHz) was fitted to the phenomenological equations based on Devonshire's theory that enable us to calculate the order parameter (a) of the anharmonic interaction of the B site cations and correlate it with the dielectric figure of merit (K factor, defined by the ratio of tunability and dielectric loss tangent). The resistive dielectric loss component remains low for the undoped BST film, whereas with the increase in Mn content it dominates the capacitive loss component. We have fabricated eight element coupled microstrip phase shifters and characterized them in terms of their degree of phase shift and insertion loss characteristics measured at 14.5 GHz. The phase shift increases from 239° to 337° with 0 to 3 at% Mn doping. However, the insertion loss also increases (5.4 to 9.9 dB) with the increase in dopant concentration so that the effective  $\kappa$  factor (defined as phase shift/insertion loss) does not improve significantly and remains in the range of 33 - 44°/dB. The observed dielectric characteristics are correlated with the epitaxial quality and surface morphologies of the Mn doped BST thin films. These and other transition metal doped dielectric data of textured BST thin films will be presented.

### 2:00 PM \*H2.2

NOVEL TUNABLE THIN FILM FOR HIGH QUALITY TUNABLE MICROWAVE DEVICES. G. Lin, R. Fu, S. He, J. Sun, X. Zhang and L. Sengupta, Paratek, Columbia, MD.

A new process has been developed in Paratek Microwave Inc. to formulate stable tunable thin film material. Varactors, with a co-planar interdigitated electrode (IDE) structure, were fabricated by using the new material. Varactor Q as high as 100 at 2GHz was observed with tunability of 46% at 100V. The lifetime tests indicated that this material is very stable under continuous 100 V dc bias both at the room temperature and in 70°C environment. Thus, this novel tunable thin film material opens a new avenue to develop high quality tunable microwave devices. A tunable IF filter has also been built using this novel material.

### 2:30 PM H2.3

A NEW FERROELECTRIC VARACTOR FROM WATER BASED INORGANIC PRECURSORS. T. Kirk Dougherty, John Drab, Mike Brand, Kathy Kehle, Raytheon, El Segundo, CA.

Solution deposition processes for the production of multi-element metal oxide thin films continue with great interest and varied success. Solution deposition via either sol-gel or MOD (Metal Organic Decomposition) methods are of interest due to the ability to produce a wide variety of compositional products at low capital investment cost. This paper will review the materials and process challenges and related issues to production of consistent high quality metal oxide films via the MOD process. The solid solution Ba<sub>x</sub>Sr<sub>1-x</sub>TiO<sub>3</sub> will be used as an example. Several new water stable and water soluble ceramic precursors used to make the thin films are described. These new ceramic precursor materials offer a more environmentally friendly process and more importantly yield improved thin films as compared to the prior art. Details of the RF electrical properties of capacitors made from the new water based precursors are shown. Devices made from the new thin film materials including a high power, high Q varactor will be described. The new varactor is built from a design

specific to the application as a high power RF modulator and offers a tuning capacitance range of over 5 to 1 using less than a 20 volt controlled voltage. Results of tuning, Q and intermodulation effects of this device in a high power RF circuit will be shown. The device offers a low cost and simpler replacement to semiconductor based varactor diodes.

#### 2:45 PM H2.4

MICROSTRUCTURAL AND ELECTRICAL CHARACTERIZATION OF BARIUM STRONTIUM TITANATE-BASED SOLID SOLUTION THIN FILMS DEPOSITED ON CERAMIC SUBSTRATES BY PULSED LASER DEPOSITION.

Costas G. Fountzoulas<sup>a</sup>, Daniel M. Potrepka<sup>b</sup>, and Steven C.

Tidrow<sup>b</sup>; <sup>a</sup> Weapons Materials Research Directorate, Army Research Laboratory, Aberdeen Proving Ground, MD; <sup>b</sup> Sensors and Electron Devices Directorate, Army Research Laboratory, Adelphi, MD.

Thin films, from novel B-site substituted barium strontium titanate (BSTO) bulk targets, have been deposited using the pulsed laser deposition (PLD) technique. The measured electrical properties of these thin films will be compared with the electrical properties of the bulk materials. The dielectric constant of the BSTO ferroelectrics can be changed by applying an electric field. Variable dielectric constant results in a change in phase velocity in the device allowing it to be tuned in real time for a particular application. The microstructure of the film influences the electronic properties which in turn influence the performance of the film.  $Ba_{0.6}Sr_{0.4}Ti_{1-y}(A^{3+})_y(B^{5+})_yO_3$  thin films were synthesized at ambient temperature and 30 mT oxygen partial pressure, at 500 mJ laser energy and 10 pulses per second on  $LaAlO_3$  (100) substrates, previously coated with  $LaSrCoO$  conductive buffer layer, using the pulsed laser deposition technique. The characteristics of the post-annealed thin films studied using x-ray diffraction, SEM, Rutherford backscattering, and capacitance measurements will be discussed in detail.

#### 3:30 PM H2.5

OFF AXIS GROWTH OF STRONTIUM TITANATE FILMS WITH HIGH DIELECTRIC TUNING AND LOW LOSS. Charles T. Rogers, Satreerat Kampangkeaw, Department of Physics, University of Colorado at Boulder, Boulder, CO.

The development of low-loss ferroelectric materials is useful for applications such as tunable band-pass filters, voltage tunable filters, phase shifters, and frequency agile antenna at RF and microwave frequencies. Strontium titanate (STO) is a well characterized material. We have studied nonlinear dielectric properties of thin film strontium titanate grown on neodymium gallate(NGO) and lanthanum aluminate(LAO) substrates using off-axis pulsed laser deposition. Laser ablation of a STO target is used to direct the material onto the substrates at 820C under 600 mTorr of oxygen background. We measured the film dielectric constant and loss tangent in the 10kHz to 1 MHz frequency range from room temperature down to 4K. We also obtained a figure of merit from the relative variation of the dielectric constant divided by the loss tangent in the presence of a DC electric field up to  $\pm 5V/\mu m$ . The resulting films significant variations of dielectric properties with the position of the substrates respect to the axis of the plume during the growth. STO films on LAO substrates show low loss and a dielectric constant close to that of bulk STO in regions near the center and the edges of the plume. On the other hand, STO on NGO shows this effect only on the films grown far from the plume axis. Careful mapping of the plume crosssection allowed us to improve the quality and reproducibility of the dielectric films yielding dielectric constant, loss tangent, and figure of merit at 35K and 1MHz close to bulk values. The effect of the film thickness on the dielectric properties is in progress.

#### 3:45 PM H2.6

MICROWAVE DIELECTRIC PROPERTIES of  $(Ba,Sr)TiO_3$  THIN FILMS. James S. Horwitz, N. Navi, Huey-Daw Wu, Syed B. Qadri, W. Kim, Steven W. Kirchoefer, D.M. Bubb and Douglas B. Chrisey, Naval Research Laboratory, Washington, DC.

Epitaxial and polycrystalline  $Ba_xSr_{1-x}TiO_3$  films have been deposited onto single crystal and polycrystalline substrates by pulsed laser deposition. The microwave dielectric properties have been measured using interdigitated and gap capacitors at room temperature and microwave frequencies (1-20 GHz) as a function of electric field ( $\leq 200$  kV/cm). Resonant structures have also been used to characterize microwave dielectric properties at  $\sim 2$  GHz. The room temperature dielectric constant, change in dielectric constant under a DC bias and the dielectric loss are strongly affected by film composition (the ratio of Ba/Sr and oxygen vacancies) and film strain. Oxygen vacancies can be used to alter the nucleation and growth of the film and minimize the strain. For all values of x, an oxygen deposition pressure can be found which results in the growth of a strain free, but oxygen deficient film. For film compositions that are paraelectric at room temperature, the minimum strain condition

yields a maximum microwave figure of merit ( $K = \text{dielectric } Q \times \% \text{ tuning}$ ). For films that are ferroelectric at room temperature, the minimum strain condition corresponds to a minimum in the microwave figure of merit. Films have been post-annealed in oxygen. The post-deposition anneal fills oxygen vacancies and causes the lattice parameter to decrease. Acceptors such as W and Mn have been used reduce the free carrier concentration generated by the oxygen vacancies and further reduce the dielectric loss.

#### 4:00 PM H2.7

THE IMPACT OF THERMAL STRAIN ON THE DIELECTRIC CONSTANT OF BARIUM STRONTIUM TITANATE THIN FILMS.

T.R. Taylor, P.J. Hansen, B. Acikel, N. Pervez, R.A. York, and J.S. Speck, University of California Santa Barbara, Santa Barbara, CA; S.K. Streiffer, Argonne National Labs, Argonne, IL.

Barium strontium titanate (BST) thin films were deposited by sputtering on Pt/SiO<sub>2</sub> structures using five different host substrates: magnesium oxide, strontium titanate, sapphire, silicon, and vycor glass. These substrates were chosen to provide a systematic change in thermal strain while maintaining the same film microstructure. PECVD was used to deposit 60 nm of SiO<sub>2</sub> on all the samples simultaneously. The thin SiO<sub>2</sub> layer provided each sample with a similar growth surface for the platinum electrode and subsequent oxide film. The BST film (100 nm) and platinum electrode (100 nm) were deposited concurrently and in-situ to avoid variation between samples. A thin Zr layer (2.5 nm) was used as an adhesion layer for platinum and SiO<sub>2</sub>. All films have a weakly textured microstructure. Temperature dependent dielectric measurements from 100-500 K determined that decreasing thermal expansion coefficient of the host substrate (i.e. larger tensile thermal strain) reduces the film dielectric permittivity in agreement with Shaw et al. (Appl. Phys. Lett. 75, 2129 (1999)). The experimentally determined Curie-Weiss temperature (constant\*10e5) were 8.3K(0.989), -51K(1), -67.7K(1.13), -73.7K(1.11), and -156K (1.13) for magnesium oxide, strontium titanate, sapphire, silicon, and vycor glass respectively. The Curie Weiss temperature decreases with increasing tensile thermal strain and the Curie-Weiss constant increases with tensile strain as predicted by Pertsev et al. (J. Appl. Phys. 85, 1698 (1999)).

#### SESSION H3: POSTER SESSION

Chair: Steven C. Tidrow

Tuesday Evening, April 2, 2002

8:00 PM

Salon 1-7 (Marriott)

#### H3.1

CHARACTERISTICS ANALYSIS OF SAW FILTER USING

$Al_{0.36}Ga_{0.64}N$  THIN FILM. Sun-Ki Kim, Min-Jung Park, Cheol-Yeong Jang, Hyun-Chul Choi, Jung-Hee Lee, Yong-Hyun Lee, Kyungpook National University, School of Electronic & Electrical Engineering, KOREA.

Acoustic wave devices are necessary for contemporary mobile communication system. AlN, GaN, and their alloys are important piezoelectric semiconductors which are suitable for SAW (Surface Acoustic Wave) applications as well as blue/green light emitters and high power/high temperature transistors. SAW velocities of AlN and GaN are 5700 m/s and 4800 m/s, respectively. Theoretical SAW velocity of  $Al_xGa_{1-x}N$  is expected to be from 4800 m/s to 5700 m/s varying with  $x=0$  to  $x=1$ , which indicates that the operating frequency of the SAW can be controllable by simply changing Al-mole fraction.  $Al_xGa_{1-x}N$  sample used in this work was epitaxially grown on sapphire by MOCVD. Al-mole fraction of  $x=0.36$  was measured by RBS. Characteristic parameters of the fabricated SAW filter were measured by HP 8753C network analyzer. SAW velocity of 5420 m/s and TCF (temperature coefficient of frequency) of -51.20 ppm/° were measured, when kh value was 0.078, and temperature was ranged between -30° and 60°, where k is wave number and h is thickness of GaN thin film. Electro-mechanical coupling coefficient was ranged from 1.26% to 2.22%. The fabricated SAW filter had good device performance with insertion loss of -33.853 dB and side lobe attenuation of 20 dB. SAW filter using  $Al_xGa_{1-x}N$ /sapphire structure was, for the first time, fabricated and demonstrated a good device performance. This is very important in fact that the SAW velocity and the related operating frequency can be controllable by simply changing Al-mole fraction. This work was supported by grant No. 2000-2-30200-006-2 from the Basic Research Program of the Korea Science & Engineering Foundation.

#### H3.2

MICROWAVE DIELECTRIC SPECTROSCOPY OF

FERROELECTRIC THIN FILMS. Beomjin Kim, Minki Jeong, Sunggi Baik, Pohang University of Science and Technology, Dept of Materials Science and Engineering, Pohang, KOREA; Victor

Kazmirenko, Yuriy Poplavko, National Technical University of Ukraine, Dept of Microelectronics, Kiev, UKRAINE.

We devised a measurement method of microwave dielectric constants of dielectric thin films without applying electrodes. The method uses a rectangular waveguide in which the dielectric thin films prepared on a substrate are filled vertically at the center. The frequency dependence of S-parameter measured by network analyzer enables us to calculate the dielectric constant and loss factor of the films at the microwave region through simulation. We prepared  $Ba_{1-x}Sr_xTiO_3$  thin films on MgO (001) single crystal substrate by pulsed laser deposition (PLD), and determined their dielectric constants and loss factors as a function of its composition (x) at  $\sim 10$ GHz using this method.

**H3.3**  
SYNTHESIS OF NANOSTRUCTURED MAGNETIC MIXED-OXIDE FERRITE POWDERS BY USING A NOVEL CHEMICAL METHOD. N.N. Ghosh, Birla Institute of Technology and Science, Chemistry Group, Pilani, Rajasthan, INDIA.

Research in the field of nanostructured ceramic powders have gained immense importance because of their potential application in many areas of technology. Technologically, fine-particle ferrites have been of interest due to their wide spectrum of applications as inductor cores in RF systems, circulators, permanent magnets, advanced memories, sintered electrodes, microwave devices, anti-detection (stealth) technology applications, catalysts etc. The surface properties and the microstructures of such powders, which control most of the parameters required for any particular application, often depend on the method of their preparation. In the conventional ceramic method for the preparation of ferrites long heating schedules and high temperatures, sinter the final product and result in the loss of the fine particle nature of the powders. This method is thus affected by a poor control of the particle size, morphology and microscopic homogeneity. In the present investigation, an attempt has been made to establish a new chemical route for synthesis of the nanostructured mixed oxide ferrite powders. By using this chemical method a variety of ferrite powders having spinel structure and doped with Co, Ni, Mn, Zn etc has been prepared. In this method nitrate salts of the different metals were used as starting materials. The aqueous solutions of the metal nitrates were mixed according to the molar ration of the compositions. Then the mixtures were mixed with an aqueous solution of water soluble polymer. This mixture after drying yield a brown powders. These powders were then calcined at different temperatures ranging from 6000C to 9000C. Nanostructured powders were obtained from the thermal decomposition of the brown powders. The powders, prepared by calcinations at different temperatures, were characterized by using X-Ray diffraction analysis, IR spectroscopy, TGA/ DTA, and TEM. It was observed that the average particle size of the powders are in nanometer scale with a narrow size distribution. The average particle size of the powders was increased with the increase of calcinations temperature. This chemical method has proved to provide a convenient process for the preparation of nanostructured ceramic powders at comparatively low temperatures and offers the potential of being a simple and cost-effective route.

**H3.4**  
THE EFFECTS OF PLASMA INDUCED DAMAGE ON THE CHANNEL LAYERS OF ION IMPLANTED GaAs MESFETS DURING REACTIVE ION ETCHING (RIE) AND PLASMA ASHING PROCESSES. Hokyun Ahn, Jaekyoung Mun, Heacheon Kim, Electronics and Telecommunications Research Institute (ETRI), Advanced Micro-Electronics Research Laboratory, Taejon, KOREA.

The gate length of GaAs MESFETs is required to be shorter for their higher microwave frequency applications. The side-wall process using silicon nitride is the one of the useful processes to fabricate short gate length GaAs MESFETs. The side-wall process consists of deposition and anisotropic etching of silicon nitride and delivers plasma induced damages on the channel layers of the devices. In this study, the effects of plasma induced damage on the channel layers of ion implanted GaAs MESFETs during reactive ion etching and plasma ashing processes have been investigated. The plasma induced damage was characterized by sheet resistance measurement, X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES) of different etched surfaces compared with a chemically wet-etched reference surface. Also the effect of the plasma induced damage on the device performance was investigated. As a result, plasma ashing can deteriorate the plasma induced damage by RIE.

**H3.5**  
RF SPUTTERED BZN THIN FILMS FOR VOLTAGE TUNABLE DIELECTRIC DEVICE APPLICATIONS. Young Pyo Hong, Seok Ha, Ha Yong Lee, Young Cheol Lee, and Kyung Hyun Ko, Department of Material Science and Engineering, Ajou University, Suwon, KOREA; Dong-Wan Kim, Hee Bum Hong, and Kug Sun Hong, School of Materials Science and Engineering, College of

Engineering, Seoul National University, Seoul, KOREA.

Bismuth zinc niobate (BZN) thin films have potential applications for tunable RF and microwave devices due to a relatively high dielectric constant, low dielectric loss, an adjustable temperature coefficient of resonant frequency, and electric field dielectric constant tunability. In this study, tunability of BZN films deposited by a reactive RF magnetron sputtering was investigated. The composition of  $(Bi_{1.5}Zn_{0.5})(Zn_{0.5}Nb_{1.5})O_7$  cubic pyrochlore was fabricated in to 2inch disk for target. For tunability measurements, films were process in to standard capacitor type MIM structure on the Si employing Ag(top) and Pt(bottom) electrodes. As-deposited films were amorphous and annealed at 600~800°C for 3 hours in air to be crystallized as a cubic pyrochlore. With increase of annealing temperature, tunability increased. Furthermore, it was found that oxygen partial pressure during sputtering was the critical factor on the loss tangent and tunability. While the loss tangent continuously decreased with increase of oxygen partial pressure, tunability increase up to 10% of oxygen partial pressure and decreased again. With optimized sputtering conditions of 10% oxygen partial pressure and 800°C of annealing temperature, cubic BZN thin films with a dielectric constant of  $\sim 150$ , loss tangent  $< 0.003$ , and dielectric tunability of 10% at 800kV/cm was obtained.

**H3.6**  
Ag(Ta,Nb)O<sub>3</sub> FOR TUNABLE HIGH FREQUENCY DEVICES. Frédéric Zimmermann, Wolfgang Menesklou, Ellen Ivers-Tiffée, Institut für Werkstoffe der Elektrotechnik, Universität Karlsruhe (TH), GERMANY.

Measurements of permittivity and dielectric losses have shown that Ag(Ta,Nb)O<sub>3</sub> (ATN) is a promising candidate for application in tunable high frequency devices. AgTa<sub>0.05</sub>Nb<sub>0.95</sub>O<sub>3</sub> as a bulk ceramic shows relatively low permittivity of  $\epsilon = 270$  and tunability ( $\tau = (\epsilon(0) - \epsilon(E_{max})) / \epsilon(0)$ ) of 15% for an applied field of 1 kV/mm. AgTa<sub>x</sub>Nb<sub>1-x</sub>O<sub>3</sub> powders with x=0.4; 0.5; 0.6; 0.9; 0.95 have been prepared using a mixed oxide technique. Permittivity, losses and tunability of ATN bulk ceramic and screen printed thick films on Al<sub>2</sub>O<sub>3</sub> substrates have been characterized at low frequency in a temperature range from -150°C to 120°C. ATN thick films have also been characterized at 10 GHz, in a temperature range from -40°C to 120°C by measuring coplanar waveguide resonators.

**H3.7**  
STUDIES OF THIN FILM FERROELECTRICS WITH CHARGE-COMPENSATED SUBSTITUTIONS IN BST. Daniel Potrepka, Steven Tidrow, Arthur Tauber, Kevin Kirchner, Bernard Rod, SEDD, U.S. Army Research Lab, Adelphi, MD; Daniel Bubb, Seton Hall Univ, South Orange, NJ; James Horwitz, Naval Research Lab, Washington, DC.

Thin films were prepared from bulk targets by PLD techniques. The targets were composed of Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> with charge-compensated substitutions for Ti<sup>4+</sup>. Results of the dielectric characterization measurements will be presented and compared to the results of similar measurements in bulk materials with the same composition.

**H3.8**  
MICROWAVE PHASE SHIFTERS USING FERROELECTRIC (BaSr)TiO<sub>3</sub>. Won-Jeong Kim, Eun-Kyoung Kim, Seung-Eon Moon, Su-Jae Lee, Kwang-Yong Kang, and Seok-Kil Han, Electronics and Telecommunications Research Institute, Taejon, KOREA.

The ferroelectric Ba<sub>1-x</sub>Sr<sub>x</sub>TiO<sub>3</sub> films were prepared on (001) MgO single crystals by pulsed laser deposition. Coplanar waveguide (CPW) type phase shifters controlled by external dc bias field were fabricated on Ba<sub>1-x</sub>Sr<sub>x</sub>TiO<sub>3</sub> films using a 2000 nm thick metal layer to reduce metal loss. Microwave properties of the CPW phase shifter were measured using a HP 8510C vector network analyzer from 0.045-20 GHz. The fabricated CPW phase shifters exhibited differential phase angle of 60 - 250° at 10 GHz with a dc bias field of less than 40 kV/cm between center and ground conductors. By controlling film growth condition, phase shifters exhibited a large differential phase shift and a low insertion loss at the same time. Furthermore, a stable differential phase angle (within 3.5°) was observed while changing the power of incident microwave from -10 to +30 dBm. These results demonstrate the possibility of ferroelectric tunable devices on a high power wireless telecommunication using the nonlinear response of ferroelectrics. In this presentation, detailed microwave properties of the ferroelectric CPW will be presented.

**H3.9**  
ORIENTATION DEPENDENT MICROWAVE PROPERTIES OF FERROELECTRIC (Ba<sub>1-x</sub>Sr<sub>x</sub>)TiO<sub>3</sub> THIN FILMS. Seung Eon Moon, Eun-Kyoung Kim, Su-Jae Lee, Kwang-Yong Kang, and Won-Jeong Kim, Basic Research Department, Electronics and Telecommunications Research Institute, Taejon, KOREA.

To study the effect of the orientation of ferroelectric  $\text{Ba}_{1-x}\text{Sr}_x\text{TiO}_3$  (BST) films on the microwave dielectric properties, the surface orientations of the ferroelectric BST films were engineered by controlling the growth condition of pulsed laser deposition with MgO single crystals with different surface normal directions, such as (100) and (110). Structural properties and surface morphologies of BST films were investigated using a 4-circle X-ray diffractometer and a scanning electron microscope, respectively. The microwave properties of orientation engineered BST films were investigated using coplanar waveguide transmission lines which were fabricated on BST films using a thick metal layer by photolithography and etching process. Phase shifters fabricated on (110) BST/MgO exhibited larger differential phase angles and figure of merit at 10 GHz than those fabricated on (100) BST/MgO. ( $\Delta\phi = 94^\circ$  and  $63^\circ$  for phase shifters on (100) and (110) BST/MgO, respectively.) In this paper, details of physical and electrical properties along the crystallographic orientation of BST films will be discussed.

### H3.10

MICROWAVE MEASUREMENTS OF DIELECTRIC CONSTANT AND LOSS TANGENT OF TUNABLE MICROWAVE MATERIALS AS FUNCTIONS OF TEMPERATURE AND FREQUENCY USING AN OPEN CONFOCAL RESONATOR. S.C. Tidrow, F.J. Crowne, D.M. Potrepka, A. Tauber, E.D. Adler, A. Lee, B. Rod, M.S. Patterson, U.S. Army Research Laboratory, Adelphi, MD.

The dielectric constant and loss tangent of several tunable microwave materials have been measured and are reported for the temperature range from  $-50$  to  $100^\circ\text{C}$  and frequency range from 15 to 50 GHz. The apparatus used for making the measurements is an open confocal resonator that uses well-established Gaussian beam theory and can be used to make measurements in the 10 to 100 GHz range. In order to evaluate microwave materials to meet military specifications, the resonator has been placed in an environmental chamber to cycle the sample temperature from  $-50$  to  $100^\circ\text{C}$ . In the present configuration, the resonator is capable of providing measurements on dielectric constants ranging from about 1 up to at least 300 and loss tangents ranging from about 0.1 down to as low as  $1 \times 10^{-5}$ . In addition to providing dielectric constants and loss tangents of both tunable and non-tunable microwave materials, some of the particulars of the resonator theory and resonator configuration are presented.

### H3.11

SUB  $0.1\mu\text{m}$  ASYMMETRIC GAMMA-GATE PHEMT PROCESS USING ELECTRON BEAM LITHOGRAPHY. W.S. Sul, D.H. Shin, J.K. Rhee, Dongguk Univ., Millimeter-wave Innovation Technology Research Center, Seoul, SOUTH KOREA.

We studied the fabrication of GaAs-based pseudomorphic high electron mobility transistors (PHEMT's) for the purpose of millimeter-wave applications. To fabricate the high performance GaAs-based PHEMT's, we performed the simulation to analyze the designed epitaxial-structures. Each unit processes, such as  $0.1\mu\text{m}$  Gamma-gate lithography, silicon nitride passivation and air-bridge process were developed to achieve high performance device characteristics. The DC characteristics of the PHEMT's were measured at a  $70\mu\text{m}$  unit gate width of 2 gate fingers, and showed a good pinch-off property ( $V_P = -1.75\text{ V}$ ) and a drain-source saturation current density ( $I_{\text{dss}}$ ) of  $373.53\text{ mA/mm}$ . Maximum extrinsic transconductance (gm) was  $522.4\text{ mS/mm}$  at  $V_{\text{gs}} = -0.3\text{ V}$ ,  $V_{\text{ds}} = 1.5\text{ V}$ , and  $I_{\text{ds}} = 0.5\text{ I}_{\text{dss}}$ . The RF measurements were performed in the frequency range of 1.0–50 GHz. For this measurement, the drain and gate voltage were 1.5 V and -0.3 V, respectively. At 50 GHz, 9.2 dB of maximum stable gain (MSG) and 4.2 dB of S21 gain were obtained, respectively. A current gain cut-off frequency ( $f_T$ ) of 106.4 GHz and a maximum frequency of oscillation ( $f_{\text{max}}$ ) of 180 GHz were achieved from the fabricated PHEMT's of  $0.1\mu\text{m}$  gate length.

### H3.12

ELECTRICALLY TUNABLE MICROWAVE PHASE SHIFTER WITH AIR-DIELECTRIC SANDWICH STRUCTURE. Minki Jeong, Beomjin Kim, Sunggi Baik, Pohang University of Science and Technology, Dept of Materials Science and Engineering, Pohang, KOREA; Victor Kazmirenko, Yuriy Poplavko, Dept of Microelectronics, Kiev, UKRAINE.

We propose an air-dielectric sandwich structure as a new concept of tunable phase shifter. The structure consists of very thin air layer and relatively thick dielectric material inside a waveguide. Electro-mechanical actuator attached to the dielectric material controls the thickness of air gap. Variation in the thickness ratio between air gap and dielectric plate leads to the change in the effective dielectric constant of the structure. For instance, if we use  $\text{BaTi}_4\text{O}_9$  ( $\epsilon = 37$ ) as the dielectrics, the variation in the thickness of air gap by only several tens of micrometers significantly changes the effective dielectric constant, which induces phase shift for the microwave transmitting

through the air-dielectric sandwich. The major advantage of the proposed phase shifter is that low dielectric loss could be realized by proper selection of dielectric materials. The response time would be determined by the selection of the electromechanical actuator.

### H3.13

DEPOSITION OF POLYCRYSTALLINE ZnO FILMS BY TWO-STEP METHOD AND CHARACTERIZATION OF THERMAL ANNEALING EFFECTS. Jin-Bock Lee, Myung-Ho Lee, Hye-Jung Lee, Jin-Seok Park, Hanyang Univ, Dept of Electrical Engineering, Ansan, SOUTH KOREA.

With the rapid progress of communication technology, there has been an increasing interest in developing thin film band-pass filters, including surface acoustic wave (SAW) filters and film bulk acoustic resonators (FBARs). These devices require the thin film to have c-axis oriented growth nature, excellent crystallinity, and high electrical resistivity. Polycrystalline ZnO has been considered as one of promising materials for such device applications. The sputtering has widely been used to obtain the c-axis oriented ZnO film. Various methods, such as doping with impurities (Li, Cu, etc.) and addition of oxygen during deposition, have been used to increase the resistivity. However, it has not been shown with these methods that both the c-axis growth nature and the resistivity of ZnO can be enhanced together. Furthermore, the sputtered ZnO film may include considerable grain boundaries, as a result it can degrade its crystalline property. In this paper we propose a novel deposition method enabling to achieve a high-quality ZnO film and present experimental results regarding to the thermal annealing effects. ZnO films were deposited on the  $\text{SiO}_2/\text{Si}$  substrate by RF magnetron sputtering. The method that we propose consists of a 1<sup>st</sup>-step deposition at 100 W without adding oxygen and the 2<sup>nd</sup>-step deposition at 100 W (or higher) with adding the oxygen ( $\text{O}_2/(\text{Ar}+\text{O}_2) = 10\sim 50\%$ ). Texture coefficient (TC) for the (002)-peak and crystallite size of deposited ZnO films were evaluated from the XRD patterns. The I-V characteristic was measured to estimate the film resistivity. Raman spectra were monitored in all experiments to identify the wurtzite phase of the ZnO lattice. By using the two-step method, a strongly c-axis oriented (TC  $\sim 100\%$ ) and highly resistive ( $>10^7\ \Omega\text{cm}$ ) ZnO film could be successfully deposited. It was also observed that the crystallite size of as-deposited film was noticeably increased after thermal annealing.

### H3.14

ON THE GHz FREQUENCY RESPONSE IN NANOCRYSTALLINE FeXN ULTRA-SOFTSOFT MAGNETIC FILMS. N.G. Chechenin, C.B. Craus, A.R. Chezan, D.O. Boerma, and L. Niesen, Materials Science Centre, University of Groningen, Groningen, THE NETHERLANDS.

Nanocrystalline FeXN films have been proved to have excellent ultrasoft magnetic properties with a high magnetic susceptibility in the frequency region above 1 GHz. The influence of the grain size, D, on the dc-magnetism has been analyzed in Hoffmann papers ([1] and ref. therein). The high-frequency response in a nanocrystalline film, in spite of the importance, has not been analyzed theoretically. Here we make an attempt to contribute to this item, based on the observations in our ultra-soft magnetic FeZrN films. A grain size much smaller than the size of the coupling volume is necessary to average out the magnetocrystalline anisotropy. The residual magnetocrystalline anisotropy, proportional to  $D^{3/2}$ , causes a stray field predominantly oriented along the easy axis and an angular spread of the magnetization, which is observed as a ripple in Lorentz TEM. The r.m.s. magnitude of the angular spread  $\langle \phi^2 \rangle > 1/2$ , which is a measure of the stray field, was obtained using LTEM and an advanced analysis of the LTEM ripples for the films deposited and post-treated under various conditions. The theoretical description of the influence of the micromagnetic ripple on the high-frequency response is based on the approach developed by Clogston [2]. We conclude that, under certain conditions, magnetic inhomogeneities due to stray fields can have a major contribution to the broadening of the ferromagnetic resonance, restricting the high frequency range of soft magnetic films. [1] H. Hoffmann, Thin Solid Films, 58 (1979) 223. [2] A.M. Clogston, J. Appl. Phys., 29 (1958) 334.

#### SESSION H4: BST FILM CHARACTERIZATION

Chair: Chonglin Chen

Wednesday Morning, April 3, 2002

Salon 3/4 (Marriott)

#### 8:00 AM \*H4.1

A STATISTICAL ANALYSIS OF LASER ABLATED BSTO/LaO FILMS FOR MICROWAVE APPLICATIONS. R.R. Romanofsky, S.A. Alterovitz, C.M. Mueller, F.W. Van Keuls, NASA Glenn Research Center, Cleveland, OH; J. Kim, K.S. Harshavardhan, Neocera, Inc., Beltsville, MD.

The NASA Glenn Research Center is constructing a 500 element scanning phased array antenna using  $Ba_xSr_{1-x}TiO_3$ -based phase shifters. An ultimate goal is to produce phase shifters at 19 GHz with 3 dB insertion loss and at least 360 degrees phase shift with 3 percent bandwidth. It is well known that there is a direct relationship between dielectric tuning and loss due to the Kramer-Kronig relationship and that film crystallinity and strain, affected by the substrate template, play an important role.  $Ba_{0.50}Sr_{0.50}TiO_3$  films, nominally 400 nm thick, were deposited on 50 0.25 mm thick, 5 cm diameter  $LaAlO_3$  wafers. Although previous results suggested that Mn-doped films on MgO were intrinsically superior in terms of phase shift per unit loss, for this application phase shift per unit length was more important. The composition was selected as a compromise between tuning and loss for room temperature operation (e.g. crystallinity progressively degrades for Ba concentrations in excess of 30 percent). As a prelude to fabricating the array, it was necessary to process, screen, and inventory a large number of samples. Variable angle ellipsometry was used to characterize refractive index and film thickness across each wafer. Microstructural properties of the thin films were characterized using high resolution X-ray diffractometry. Finally, prototype phase shifters were patterned on each wafer and RF probed to measure insertion loss and phase shift as a function of dc bias voltage. The relationship among film quality and uniformity and performance is analyzed. This work presents the first statistically relevant study of film quality and microwave performance and represents a milestone towards commercialization of thin ferroelectric films for microwave applications.

#### 8:30 AM \*H4.2

#### LARGE DIELECTRIC RELAXATION IN BARIUM STRONTIUM TITANATE THIN FILMS AT MICROWAVE FREQUENCIES.

James C. Booth and R.H. Ono, National Institute of Standards and Technology, Boulder, CO; Ichiro Takeuchi and Kao-Shuo Chang, Department of Materials Science and Engineering and Center for Superconductivity Research, Department of Physics, University of Maryland, College Park, MD.

Ferroelectric thin films such as  $Ba_{0.3}Sr_{0.7}TiO_3$  hold promise for electronically tunable microwave devices. However, the losses in such films are unacceptably high, while the origins of the losses are poorly understood. We report evidence that a major contribution to the enhanced losses in these materials at microwave frequencies comes from dielectric relaxation effects. We directly observe dielectric relaxation in the frequency dependence of the measured complex permittivity in  $Ba_{0.3}Sr_{0.7}TiO_3$  thin films over a wide temperature range. By fitting both the real and imaginary parts of the measured permittivity to the Cole-Cole function over the frequency range 0.05 to 40 GHz, we are able to directly obtain values for the relaxation time for this material over the temperature range from 55 to 295K. The relaxation time extracted from such fits obtains a maximum value of 0.9 ps near the Curie temperature  $T_c$ , and decreases with increasing reduced temperature  $|T-T_c|$  or bias voltage. We relate the relaxation time to the low-frequency permittivity by the Lyddane-Sachs-Teller relation, indicating that soft mode dynamics control the microwave frequency electro-dynamics in this material system.

#### 9:00 AM H4.3

#### CORRELATION OF MICROWAVE DIELECTRIC PROPERTIES AND MICROSTRUCTURE OF UNPATTERNED FERROELECTRIC THIN FILMS.

R.G. Geyer, National Institute of Standards and Technology, Boulder, CO; M. Cole and E. Ngo, Army Research Laboratory, Aberdeen Proving Grounds, MD.

The development of voltage-tunable ferroelectric materials has many applications at microwave frequencies. Some of these applications are tunable phase shifters and delay lines for electronic scanning antennas and tunable filters, resonators and mixers for wireless communication systems. The reduced drive voltages for ferroelectric thin films (<10 V) permit compatibility with semiconductor-based systems, small size, light weight, and geometric flexibility in design. The dielectric requirements for ferroelectric thin films are high tunability, low leakage currents, low dielectric losses at microwave frequencies, relatively good frequency and temperature stability, and reproducibility of dielectric properties. Thin film materials must be single phase, possess crystalline dense microstructure, have smooth surface morphology, be defect- and crack-free, and be thermally stable with the substrate. They must also have good mechanical properties. Substrates must be chosen that minimize film strain and that lead to uniform strain distribution within the deposited film. Microwave dielectric property measurements have been performed on unpatterned barium strontium titanate (BSTO) and Mg-doped BSTO thin films using a coupled dielectric resonator system. With this technique there is no need for microstrip or coplanar waveguide patterning with associated microwave metal losses. It is also noncontacting and nondestructive. The microwave measurement frequency is determined by the geometry and permittivity of high-Q, temperature-stable, coupled cylindrical dielectric resonators. Microwave dielectric

properties are correlated with thin film crystallinity, surface morphology, microstructure, thickness, compositional uniformity, and elemental diffusion at the film-substrate interface.

#### 9:15 AM H4.4

#### CHARACTERIZATION OF FREQUENCY AGILE MATERIALS BY EVANESCENCE-PROBE MICROSCOPY.

M.E. Reeves, Y. Tesfu, Y.G. Wang, N. Navi, J.S. Horwitz, F.J. Rachford.

We will present near-field microwave microscopy data, which allows measurement of dielectric properties of materials at spatial resolutions much smaller than the wavelength of the probing field. Our results demonstrate that such measurements, when combined with finite-element analysis modeling (utilizing lab-written codes, Ansoft Maxwell 3-d, and Ansoft HFSS), yield accurate spatial maps of technologically relevant properties, which include dielectric constant, tand, topography, tunability, and anisotropy of bulk materials and thin films. In particular we will present results of the effects of oxygen and cation stoichiometry on the dielectric properties of thin-film BST. Y.G. Wang, M.E. Reeves, F.S. Rachford, Applied Physics Letters, 76, p 3295 (2000). Y.G. Wang, M.E. Reeves, W.J. Kim, J.S. Horwitz, and F.J. Rachford, Applied Physics Letters, 78, p.3872 (2001).

#### 9:30 AM H4.5

#### LATTICE DYNAMICS AND PHASE TRANSITIONS IN $Ba_xSr_{1-x}TiO_3$ FILMS STUDIED BY RAMAN SPECTROSCOPY.

D.A. Tenne, A. Soukiassian, A.M. Clark, and X.X. Xi, Pennsylvania State Univ., Dept. of Physics, University Park, PA.

Barium strontium titanate thin films grown by pulsed laser deposition on  $SrTiO_3$  and  $LaAlO_3$  substrates with  $SrRuO_3$  buffer layers were studied by Raman spectroscopy in the temperature range from 5 to 300 K. The soft phonon modes were observed in Raman spectra of  $Ba_xSr_{1-x}TiO_3$  films with Ba contents  $x = 0.05, 0.1, 0.2$  and  $0.5$ . The temperature dependence of the soft phonon frequencies and the splitting of the triply-degenerated soft mode into two components of A and E symmetries indicate the ferroelectric phase transition. In the films with higher Ba content the E soft mode is overdamped over a broad range of temperatures. The overdamping region becomes narrower with decreasing Ba concentration. For  $Ba_xSr_{1-x}TiO_3$  films with  $x \leq 0.1$  the E soft mode is not overdamped in the whole range of temperatures. This is attributed to the order-disorder behavior which increases with Ba content. The temperature dependence of the A soft mode frequency in the ferroelectric phase shows that the spontaneous polarization has discontinuity at the Curie point for films with Ba content  $x > 0.1$ , while for  $x = 0.05$  it tends continuously to zero. This indicates that the phase transition is of the first order for the films with  $x$  higher than 0.1, while for smaller Ba concentrations it changes into the second order. The relative Raman intensity of the A soft mode and hard phonon modes decreases gradually over a broad range of temperatures. This indicates a thermally broadened ferroelectric phase transition in the thin films. Raman spectroscopy results are correlated to the temperature dependence of dielectric constant. The results on films grown on  $SrTiO_3$  and  $LaAlO_3$  substrates show the influence of strain on the temperature of the ferroelectric phase transition. The differences in strain state in films grown on these two types of substrates are explained by different structure of underlying SRO layers.

#### SESSION H5: BST BULK MATERIALS AND APPLICATIONS

Chair: Amar S. Bhalla

Wednesday Morning, April 3, 2002

Salon 3/4 (Marriott)

#### 10:15 AM \*H5.1

#### RANDOM FIELD DESCRIPTION OF INDUCED RELAXOR BEHAVIOR IN DOPED BST.

Frank Crowne, Steven Tidrow, Daniel Potrepka, and Arthur Tauber, Army Research Laboratory, Adelphi, MD.

The dc and microwave responses of the barium-strontium titanate (BST) family of ferroelectrics doped with various ion impurity species are analyzed using the random-field technique and the mean-field (Landau-Devonshire) theory of ferroelectricity, along with a self-consistent computation of the dielectric constant of the host material in the presence of the impurity fields. The fields in the material are assumed to arise from associations between positive and negative ions (charge compensation) that lead to dipole formation. It is shown that whereas completely random placement of positive and negative ions generates a Holtsmark distribution of electric field, with infinite second moment and hence extremely large fluctuations in field strength, association into dipoles leads to much lower fluctuations in field and a distribution with finite second moment, which makes a self-consistent dielectric constant meaningful. The theory contains the value of the self-consistent dipole moment as a fitting parameter. Reasons why this quantity can differ from a naive value of the ion

charge times a single lattice spacing, i.e., the distance of closest approach for charge-compensating ions, will be discussed.

#### 10:45 AM \*H5.2

APPLICATION OF FERROELECTRICS IN LOW COST MICROWAVE PHASED ARRAY ANTENNAS. Pyong K. Park, Raytheon Missile Systems, Tucson, AZ; T.K. Dougherty, J.A. Zelik, D.S. Prior, Raytheon Electronic Systems, El Segundo, CA; J.B.L. Rao, D. Patel, Naval Research Laboratory, Washington, DC; L.C. Sengupta, Paratek Microwave, Inc., Columbia, MD.

This presentation will discuss the development of a novel, low-cost, phased-array antenna, being funded by the U.S. Defense Advanced Research Projects Agency (DARPA). The phased-array antenna scans its beam by electronic means without moving mechanical structures. This capability can benefit many defense and commercial applications, however, traditional phased-array antennas are expensive. The new antenna concept uses bulk voltage-tunable dielectrics (VTDs) to provide the phase shifts needed to scan the beam. VTDs currently used are composites of Barium Strontium Titanate and low-loss ceramic dielectrics. The dilution of the BST with a lower dielectric constant material aids in impedance matching of the VTD to a microwave feed and to air. Test results demonstrating electronic beam scanning of prototype antennas will be presented. VTDs for this application can be conveniently characterized by their dielectric constants and figures of merit (FOM), i.e., the loss sustained by a wave passing through the material to achieve 360 degrees of phase shift. At 10 GHz a representative VTD material has a dielectric constant of 100 and a FOM of about 2 dB/360 degree phase shift. Loss increases with frequency for these materials. At 35 GHz current materials exhibit a dielectric constant of approximately 80, with the best FOM of about 5 dB/360 degree phase shift. More information about the VTDs will also be presented.

#### 11:15 AM H5.3

AN S-BAND REFLECTIVE PHASE SHIFTER - A DESIGN EXAMPLE USING FERROELECTRICS. Dongsu Kim, J.S. Kenney, Georgia Institute of Technology, Atlanta, GA; David Kiesling, David Stollberg, MicroCoating Technologies, Inc., Atlanta, GA.

One of the challenges faced in using ferroelectrics in high frequency devices is how to effectively use the material in a circuit design. A compact reflection-type phase shifter fabricated on sapphire substrates coated with ferroelectric barium strontium titanate (BST) thin-films has been built which shows the promise of using BST thin films in the design of tunable microwave devices. The phase shifter, fabricated as one monolithic assembly, consists of a 3dB coupler, meander-line inductors, and tunable interdigital capacitors. A continuously variable phase shift range of more than 120° was obtained at a center frequency of 2.0 GHz, and better than 90° over a 500 MHz bandwidth. The insertion loss minimum was 1.8 dB at 1.85 GHz, and less than 2.0 dB over the 500 MHz bandwidth. The return loss was better than 15 dB for the 90° phase shift range over the 500 MHz bandwidth. Along with presenting the measured data, design data will be shown that will establish practical ranges that must be achieved for the materials electrical properties in order to take the device from the bench-top into the marketplace. The loss of the BST phase shifter presented in this work is on the order of other commercially available RF front-end components, such as bandpass filters and RF switches. This holds promise for the practical realization of smart antenna systems in cellular handsets and wireless LAN cards. This work was supported in part by U.S. Air Force SBIR contact number F33615-01-M-1950.

#### 11:30 AM H5.4

TUNING OF A DIELECTRIC RESONATOR USING FERROELECTRIC THICK FILM. Peter Kr. Petrov, Kumaravinothan Sarma, and Neil McN. Alford, Physical Electronics and Materials Centre, EEIE, South Bank University, London, UNITED KINGDOM.

An electrical tuneable resonator comprising a cavity within which is mounted a dielectric resonator (DR) and ferroelectric thick film grown on a metal substrate is proposed. On applying a dc bias, the relative permittivity of the ferroelectric film decreases and hence affects the dielectric resonator electric field and changes the resonance frequency. The coupling between the DR and BSTO film is balanced so as to be strong enough to provide tuning and yet not so strong as to reduce the Q-factor of the whole device beyond useful values. The thick  $Ba_xSr_{1-x}TiO_3$  (BSTO) films used in this study were prepared by ceramic processing. The Ba/Sr ratio varied from 50/50 to 75/25. Discussed in this paper is dependence of the BSTO films quality and permittivity of the used dielectric resonators on the tuning ratio and Q-factor reducing. We examined dielectric resonators made in our laboratory ( $TiO_2$ ,  $Al_2O_3$  and Ca-Ti-Nd-Al-O with permittivity of 100, 10 and 47 respectively). The preliminary results shown a resonant frequency shifting by 2 MHz while the Qf was above 10,000 GHz.

#### 11:45 AM H5.5

THE DEPENDENCE OF DIELECTRIC PROPERTIES ON COMPOSITION VARIATION IN  $Ba_{0.6}Sr_{0.4}(YTa)_yTi_{1-2y}O_3$ . Daniel Potrepka, Steven Tidrow, Arthur Tauber, Kevin Kircher, Matthew Ervin, Krishna Deb, Bernard Rod, Frank Crowne, U.S. Army Research Lab, Adelphi, MD.

$Ba_{0.6}Sr_{0.4}(YTa)_yTi_{1-2y}O_3$  has been shown to have properties which are promising for tunable applications requiring low dielectric constant [1].  $Ba_{0.6}Sr_{0.4}(YTa)_yTi_{1-2y}O_3$  with  $y < 0.5$  has been synthesized and well-characterized using x-ray diffraction, EDAX, and Raman Spectroscopy. The dependence of the dielectric properties on concentration,  $y$ , of Y and Ta will be presented along with implications for improved performance in device applications. [1] D.M. Potrepka et al., Mat. Res. Soc. Symp. Proc. 656E, DD5.9.1 (2001).

#### SESSION H6: NEW MATERIALS AND APPLICATIONS

Chairs: James S. Horwitz and Mark E. Reeves  
Wednesday Afternoon, April 3, 2002  
Salon 3/4 (Marriott)

#### 1:30 PM \*H6.1

FIELD TUNABLE DIELECTRICS FOR MICROWAVE ELECTRONICS. Amar Bhalla, Materials Research Laboratory, MRI, Pennsylvania State University, University Park, PA.

This paper overviews the scope of designing, developing and exploiting the dielectric materials with field controllable properties that can be utilized in frequency agile components for a wide range of applications in the various desirable temperature zones. Several interesting material systems with low dielectric loss, high tunability and wide temperature range applications have been identified in the polycrystalline, single crystal, composites, and film form. Results on the dielectric and tunability characteristics of  $(BaSr)TiO_3$ ,  $(PbSr)TiO_3$  in the pure compound and in the composites of these compositions with MgO,  $MgTiO_3$ ,  $Al_2O_3$  etc. will be discussed.

#### 2:00 PM H6.2

DIELECTRIC AND ROOM TEMPERATURE TUNABLE PROPERTIES OF MAGNESIUM DOPED BCTZ THIN FILMS ON PLATINUM/MAGNESIUM OXIDE SUBSTRATES. T.S. Kalkur, W.C. Yi, Microelectronics Research Laboratories, University of Colorado, Colorado Springs, CO; Elliott Philofsky and Lee Kammerdiner, Applied Ceramics Research, Colorado Springs, CO.

$Ba_{0.96}Ca_{0.04}Ti_{0.84}Zr_{0.16}O_3$  (BCTZ) has been proposed as a promising material for high dielectric constant device applications since it has a bulk dielectric constant upto 30,000 at room temperature. In this paper we are reporting the results of magnesium (Mg) doped BCTZ film on magnesium oxide (MgO) substrate with platinum (Pt) as the bottom electrode. Mg doped BCTZ films were fabricated by spin-on metalorganic decomposition (MOD) method. The films were annealed in the temperature range of 600-900°C in flowing oxygen environment. The structures of the films were determined by x-ray diffraction. The capacitance vs voltage characteristics of the BCTZ films show very good tunability (greater than 60%) at room temperature.

#### 2:15 PM H6.3

Abstract Withdrawn.

#### 2:30 PM H6.4

EVIDENCE FOR ANTIFERROELECTRIC BEHAVIOR IN  $KNbO_3/KTaO_3$  SUPERLATTICES. David Norton, J. Sigman, Univ. of Florida, Dept of Materials Science and Engineering, Gainesville, FL; Hans Christen, Pam Fleming, Lynn Boatner, Oak Ridge National Laboratory, Oak Ridge, TN; Mark Reeves, George Washington Univ, Washington, DC.

In recent years, the atomic layer-by-layer growth of  $K(Nb,Ta)O_3$  thin films and superlattice structures have been investigated. Specifically, the properties of  $KNbO_3/KTaO_3$  superlattices grown by pulsed laser deposition on  $KTaO_3(001)$  have been studied. We have recently investigated the dielectric properties of 1 unit cell x 1 unit cell  $KTaO_3/KNbO_3$  superlattices. An anomalous dielectric tunability is observed in the superlattice structures, with capacitance increasing with applied dc bias for temperatures just below that corresponding to a structural transition. This behavior is not observed in alloy films of comparable thickness, and is inconsistent with the nonlinear response expected for either paraelectric or ferroelectric material. However, an increase in dielectric constant with applied field is consistent with antiferroelectric behavior. The antiferroelectric ordering appears to be induced by the artificial B-site modulation imposed by the superlattice along the growth direction.



### 2:45 PM H6.5

THE FERROELECTRIC SLAB: A GEOMETRY FOR MICROWAVE COMPONENTS THAT INCORPORATE FERROELECTRIC MATERIALS. Frank Crowne, Steven Tidrow, Army Research Laboratory, Adelphi, MD.

A slab geometry is proposed for incorporating the properties of ferroelectric materials into microwave components such as delay lines, patch antennas, and filters. Using a standard dielectric-slab geometry, the ferroelectric is inserted between two cladding layers and a microstrip electrode is placed on top of it. The wave eigenmodes of this structure distribute the propagating microwave fields between the ferroelectric and the cladding, so that the effective dielectric constant is a weighted average of the dielectric constants of the two materials. The mode spectrum of the structure is discussed, along with issues of impedance matching and power coupling. It is shown that the geometry drastically reduces dissipation due to dielectric losses in the ferroelectric. In addition, if the microstrip line also carries a dc electric field, the dielectric constant of the ferroelectric layer can be varied and with it the propagation properties of the structure. For the specific application of delay lines based on BST, it is shown that this approach is superior to making a uniform composite material by diluting the ferroelectric with a low-dielectric filler such as MgO.

### 3:30 PM H6.6

GROWTH AND CHARACTERIZATION OF HEXAFERRITE-BSTO COMPOSITE THIN FILMS. R. Hajndl, J. Sanders, H. Srikanth, Dept of Physics, Univ of South Florida, Tampa, FL; N.J. Dudney, Solid State Div, Oak Ridge National Laboratory, Oak Ridge, TN.

There is a vital need for tailored materials exhibiting tunable electromagnetic response in the RF and microwave frequency range. This may be best achieved in ferroelectric-ferrite composites, where the combined dielectric and magnetic properties play a role in determining the overall response. We have synthesized a series of such composite thin films by magnetron sputtering in order to study the magnetic and high frequency characteristics. Barium-strontium-titanate (BSTO) and barium-iron-oxide (hexagonal ferrite) class of materials were our choice for the ferroelectric and ferrite components of the composites. Films of varying ratios of those two materials were grown and structurally analyzed. In addition, ion implantation facility at ORNL was used to implant Cobalt dopants to possibly explore changes in magnetic anisotropy due to altered microstructure of the films. The X-ray diffraction scans of the composites show a clear distinction between the phases of the two materials, which indicates that there is no degradation of the different components. Ba-ferrite grains of size  $\sim 0.5 \mu\text{m}$  are visible on the SEM images. Magnetic measurements done using a Physical Property Measurement System (PPMS) reveal a very interesting trend in the hysteresis loops. While the pure hexaferrite films show a well-defined M-H loop, the composite films show a distinct double-transition in the M-H loops. These results will be discussed in the context of the microstructure and surface modification of the thin films. The frequency-dependent impedance of the films up to 1 GHz will also be presented.

### 3:45 PM H6.7

ROLE OF THE LASER REPETITION RATE FOR LASER DEPOSITED YTTRIUM IRON GARNET FILMS. Sören Kahl, Sergey I. Khartsev, Vasyil Denysenkov, Alexander M. Grishin, Dept of Microelectronics and Information Technology, Royal Institute of Technology, Stockholm, SWEDEN; Sebastian Kranzusch, Laser-Laboratorium Göttingen e.V., Göttingen, GERMANY.

Yttrium iron garnet (YIG) films have been deposited onto single crystal gadolinium gallium garnet (GGG) substrates by the technique of pulsed laser deposition. The investigation focuses on the influence of the laser repetition rate on the properties of the films. For a given set of experimental parameters, the oxygen background pressure and the substrate temperature were optimized to achieve the smallest linewidth of the ferromagnetic resonance (FMR). The repetition rate was then varied from 10 to 50 Hz. There is a clear transition from films with low saturation magnetization  $M_S \approx 300$  Gs, high coercive fields  $H_C > 20$  Oe, and broad FMR lines  $\Delta H > 100$  Oe to films with  $M_S > 1400$  Gs,  $H_C < 10$  Oe, and  $\Delta H < 10$  Oe. This transition occurs when the laser repetition rate is changed from 20 to 30 Hz. Films deposited at 10 and 20 Hz possess a large number of holes. No significant differences could be detected in all other of the investigated properties: crystalline structure, composition, and surface roughness do not depend on the repetition rate. The results are discussed in terms of the different times between the laser pulses and the resulting different energies transferred from the ablated species to the film per unit time.

### 4:00 PM H6.8

RAPID PROTOTYPING OF FERROELECTRIC COMPOSITES. Jennifer Synowczynski, Samuel Hirsch, and Bonnie Gersten, Weapons

and Materials Research Directorate, Army Research Laboratory, Aberdeen Proving Grounds, MD.

The objective of this paper was to develop a method to allow RF designers the flexibility to create high precision complex three-dimensional components from high-dielectric constant, low-loss BaSrTiO<sub>3</sub> / MgO ferroelectric composites. This was accomplished through a combination of lost wax rapid prototyping and ceramic gelcasting. This new method also allows material scientists to create new materials based on composite structures where the connectivity between the phases in the composite can be directly controlled on a submillimeter scale. In the gelcast/lost wax method, the inverse of the structure was deposited from a low melting point thermoplastic using a high precision Sanders Rapid Toolmaker. The inverse served as a mold into which a stable ceramic slurry (viscosity between 200-300cp) was cast. The slurry contained between 50-80wt% of the ceramic powders (BaSrTiO<sub>3</sub>, MgO), a 12-20wt% solution of monomers and crosslinkers, and a free radical initiator. The ceramic powders were immobilized in place by thermally activating a polymerization reaction at 50°C. The wax mold was then removed by drying the green body in a high humidity oven at 120°C. The green body contained between 5-15 wt% binder depending on the solids loading. It was then thermally treated at elevated temperatures between 1350-1450°C to remove any residual organics and fully densify the part. SEM investigations of the unfired part determined that if the atmosphere is not controlled during the reaction, the binder distribution on the surface of the part is different from the bulk. SEM analysis was also used to characterize the microstructure and precision of the sintered part. The low frequency dielectric properties (permittivity, loss tangent, tunability) of the gelcast were reported. This study addressed the effect of casting process, binder removal, and shrinkage during sintering on the final net shape. It also discusses methods to stabilize the ceramic suspension, monitor the reaction, and the correlation between the solids loading and the green and sintered densities.