SYMPOSIUM S
Nondestructive Methods for Materials Characterization

November 29 – 30, 1999

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*Invited paper
SESSION SI: PROCESS CONTROL AND DEFORMATION BEHAVIOR VIA X-RAY TECHNIQUES
Chair: John H. Kinney
Monday, November 29, 1999
Provincetown, Orleans (M)

8:30 AM SI.1 OPTIMIZATION OF X-RAY TECHNIQUES FOR NON-DESTRUCTIVE CHARACTERIZATION OF SINGLE CRYS TALS. Jenelle Miller, Jr., Kirsten G. Lipetsky, Center for Nondestructive Evaluation, The Johns Hopkins University, Baltimore, MD.

The efficiency of modern gas turbine engines, used for both aerospace propulsion and electric power generation, increases with increasing combustion temperature. The physical requirements which limit the choice of turbine blade materials for high temperature operation are low density, thermal stability, toughness, and resistance to fatigue, high-temperature oxidation, and creep. Creep caused by dislocation motion is resisted by addition of alloying elements in solid solution and formation of stable hard precipitates, which serve as dislocation pinning points. Diffusion creep is resisted most optimally by eliminating the grain boundaries, i.e., using single crystal blades. This use of metallic single crystal for structural engineering applications places new requirements on nondestructive techniques for process control. Of particular importance is the need for an improved inspection procedure after crystal growth for determination of the overall crystalline perfection of the final blades, both internal and external. Multicrystal and poor quality single crystal blades can result in cost of aircraft engines with possible accompanying loss of life. For gas turbines used in the electric power industry the failure of a blade may also indirectly result in loss of life, but certainly will result in loss of electric power with innumerable problems for users of this power. This paper will describe nondestructive x-ray characterization techniques for process control of single crystal turbine blade growth which detect macroscopic and microscopic defects, determine the overall crystallographic perfection, and detect any unwanted secondary crystals both on the external surface as well as in the interior of single crystal turbine blades.

9:00 AM SI.2 IN SITU OBSERVATION OF OXIDATION PROCESS AT THE MOST UPPER SURFACES BY X-RAY SURFACE PROPAGATION WAVES. Toshihisa Horiehara, Kenji Ishida, Katsumi Matsushige, Dept. of Electronic Science and Engineering, Tokyo University, Tokyo, JAPAN.

Recently, we have found that when white x-rays are irradiating materials with smooth surfaces at a critical angle, there exist x-ray surface propagation waves (XSPW's) at just near the surface, and the energies of which give the information of the number densities of electrons at the most upper surfaces. Here, we utilized this findings to monitor the several phenomena occurring at the surfaces, such as an oxidation, a corrosion, a microstructure observation in atomic or nanometer scales. Experiments were carried out with a Mo rotating anode x-ray generator and total reflection x-ray analysis system to monitor the in-situ oxidation processes in various environmental circumstances such as under ultraviolet irradiation and in an atmosphere in ppm level for different types of materials; Cu of metal, H-terminated Si and GNS of semiconductors. The obtained results revealed that the oxidation does not proceed simply, but there exist several different oxidizing processes. In case of Cu, the time variation of the XSPW's at the initial stage revealed, firstly a slight increase in the electron density, subsequently an abrupt decrease, and then a steady value, suggesting the scaling-off mechanism in the oxidation phenomenon. As described above, this method has been proven to be the notib nondestructive method for evaluating the surface conditions, and become as a powerful tool for studying the relation between the degradation in micro-scale and initial surface changes such as oxidation and corrosion in nano-scale.


The mechanical properties of low-density cellular materials are not well understood. Nondestructive methods that attempt to describe the elastic behavior of these materials are either based upon highly idealized approximations of the cellular architecture or are valid only in the extreme limits of pore density. The purpose of our study was to develop a three-dimensional imaging technique to study the deformation behavior of low-density materials during loading, and to implement image-based finite element methods to solve the equations of elasticity. Specimens of silicone foam, 13.2 mm in diameter by 1-2 mm thick, were used for this study. The nominal pore density was 50%, and the pores approximated interconnected spheres. The specimens were imaged with microtomography at 18 µm resolution. A low-angle x-ray with micrometer-driven compression allowed characterization of the forms during deformation with precise registration of the images. A finite element mesh, generated from the image voxels, was used to calculate the mechanical properties of the structure, and was compared with conventional mechanical testing. Though the base material (silicone) was linear elastic, the porous foam exhibited significant nonlinear behavior in compressive loading. The finite-element calculations from the images, which were in excellent agreement with experiment data, suggested that nonlinear behavior in the load-displacement curves arises from distortion of the pores during compression. High-resolution microtomography, coupled with efficient finite-element modeling, shows promise for improving our understanding of the deformation behavior of cellular materials. This work was performed under the auspices of the U.S. Department of Energy, contract W-31-109-ENG-38.

9:30 AM SI.4 IMAGE-GUIDED FAILURE ASSESSMENT OF POROUS MATERIALS. Michele Tarli, Ralph Mueller, Orthopedic Biomechanics Laboratory, Beth Israel Deaconess Medical Center and Harvard Medical School, Boston, MA.

Lightweight metallic cellular materials have the potential for use in composite structures, such as plates, shells, and tubes. In order to evaluate the usefulness of these porous materials, an understanding of their failure mechanisms is essential. Until recently the structural analysis of fractures has been limited to 2-dimensional sections. Due to the inherent destructiveness of this method, dynamic behavior of fracture progression cannot be obtained. We have developed a technique called image-guided failure assessment that allows for the 3-dimensional analysis of fracture progression on the microscopic level. The objective of this study was to validate the technique by determining its accuracy and precision. Image-guided assessment involves the use of a novel micro-compression device to apply loads, and the use of micro-computed tomography, providing 68 µm isotropic resolution, to image the specimens. The technique was validated using an 8% density porous aluminum alloy with highly reproducible microstructural properties. Specimens were compressed and imaged at intervals of increasing strain, up to 32% strain, and compared to specimens compressed continuously from 0% to 32% strain. The load at corresponding strains, ultimate load, ultimate strain, stiffness, and Young's modulus were found not to be statistically different between the two tests. The advantage of using a nondestructive approach to assess failure is that subsequent images of increasing strain can be animated to give visual information regarding fracture initiation and propagation. Damage was seen to accumulate and propagate in local bands rather than being evenly distributed throughout the structure. This implies that local strains may be much larger than the apparent strain. In summary, image-guided failure assessment has been validated to obtain visual and mechanical data regarding fracture progression. This technique can be used to nondestructively evaluate the properties of various porous materials in order to assess their mechanical failure patterns.

9:45 AM SI.5 X-RAY MICROCTOMOGRAPHY OF FATIGUE CRACK CLOSURE AS A FUNCTION OF APPLIED LOAD IN AlLi 2000 T641 SAMPLES. R. Morano, S.L. Stank, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA; G.R. Dinwiddie and J.C. Elliott, Department of Biomedical Sciences, College of Dentistry, Queen Mary and Westfield College, London, UK.

Crack closure is held to be responsible for very low fatigue crack growth rates in many alloys such as AlLi 2000 T641, and early crack face contact during unloading or prolonged contact during loading seems to reduce the driving force for crack extension. High resolution x-ray computed tomography (i.e., microtomography) allows one to image the entire volume of samples and to quantify opening as a function of applied load over the entire crack surface. Results are reported for fatigue cracks growth in compact tension samples of AlLi 2000 T641 under two different stress ratios, and the interrelationship between crack path, crack face contact, local crack growth rates and loading conditions are discussed.

10:00 AM SI.6 INTERFACIAL DIFFUSION IN A BARUUM TITANATE FEE. M.A. Dintin, A.G. Richter and J. Kimetto, Department of Physics and Astronomy, Northwestern University, IL; S. Chatopadhyay, Department of Electrical Engineering, North Carolina Agricultural and Technical State University, Greensboro, NC.

A combination of two nondestructive techniques, Grazing Incidence X-ray Reflectivity and High Resolution X-ray Diffraction, is used to study (at around 10 Å resolution) the composition profile across a 500Å thick film of BaTiO3 grown epitaxially on (100) MgO by
SESSION S2: NDE FOR FRACTURE FATIGUE AND CORROSION
Chair: Jody L. Schroeder
Monday Morning, November 29, 1999
Provincetown/Orleans (M)

10:30 AM S2.1
ACOUSTIC EMISSION SOURCE CHARACTERIZATION AND THE FRACTURE EVOLUTION IN METAL MATRIX COMPOSITES. Akbars Rahi, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA: T. Kashi, and M. Enoki, Research Center for Advanced Science and Technology, The University of Tokyo, Tokyo, JAPAN.

A two channel AE source characterization has been used to evaluate the dynamic process of fracture during three point bending test of Al2O3 particle reinforced composites with 5 and 10 μm of particle sizes. It is observed that the dominant fracture mechanism parameters are void nucleation, growth and coalescence. AE source characterization confirms that there are two main different sources for acoustic emissions signals in these materials. Void nucleation is classified in one group and void coalescence or micro pop-in between primary voids in another group. In fine particle reinforced materials, when the AE events’ amplitude for void nucleation at fine particles is not high enough to be detected, the main source for AE events is only the coalescence of primary micro-voids. By increasing the particle size, the number of detectable events during void nucleation and growth will increase and it is proved that the AE signals caused by void nucleation and growth are almost overlapped and started from very low applied strains. Therefore the micro or macro pop-ins are occurring by linking up these voids and finally a catastrophic failure will happen through the whole thickness of material.

10:45 AM S2.2
SCANNING ACoustIC MICROSCOPY AND X-RAY DIFFRACTION CHARACTERIZATION OF NEAR CRACK TIP STRESSES IN Ti-6Al-4V. Shamsuddin Siahish, Richard Martin, University Of Dayton Research Institute, Dayton, OH.

Scanning Acoustic Microscopy is used to measure and map both Rayleigh wave velocity and Surface Skimming Longitudinal wave velocity near a crack tip in a sample of Ti-6Al-4V. Velocities are calculated at points in a typical raster scan over an area. The Local Youngs modulus variation is calculated at each point using these velocities, and then used to generate an image in a C-scan type format. X-ray diffraction measurements are performed in order to map the stress in the same region of the sample. Stress variation is measured using a standard X-ray diffraction with an Inverse Youngs modulus and is compared with the measurements that include local variation in the modulus determined using acoustic microscopy. Sponsored by the NDE Branch of the APRIL Materials and Manufacturing Directorate at Wright-Patterson Air Force Base, Ohio.

11:00 AM S2.3
DEVELOPMENT OF NONDESTRUCTIVE APPROACH TO PREDICT FATIGUE CRACK INSTABILITY. J.L. Schroeder, E.B. Shell, T.E. Matis, D. Eylon, Center for Materials Diagnostics, University of Dayton, Dayton, OH.

The objective of the this work is to develop a methodology for predicting material failure by evaluating changes in material characteristics directly prior to unstable crack growth. In an effort to establish and document these changes several Ti-6Al-4V flat, notched samples have been subjected to fatigue loading to partial life. After a fatigue crack was initiated characterization was performed during in-situ application of an incrementally increased static load. White light interference microscopy was found to be a successful nondestructive tool for characterizing changes in the deformation zone in front of the crack tip. A relationship between the applied load and the surface area of the deformation zone was obtained. This relationship was exponential directly prior to failure of the specimen. Surface observations during in-situ testing allowed samples to be brought near to failure without complete fracture. This result may aid efforts to optimize the service life of airframe structural components.

SESSION S3: STRUCTURE-SENSITIVE PROPERTIES FOR NDE CHARACTERIZATION
Chair: Norbert Meyendorf
Monday Afternoon, November 29, 1999
Provincetown/Orleans (M)

1:30 PM S3.1
THE ROLE OF STRUCTURE-TO-PROPERTY RELATIONSHIPS IN MATERIALS CHARACTERIZATION. Winfried Borgert, Metallurgical and Diagnostic Center for Materials Testing, Magdeburg, GERMANY.

As all NDT methods of materials characterization are based on physical principles, methods to characterize materials in a nondestructive approach are required to be developed systematically in the light of the relationships existing between the material structure and the physical properties. Experience has shown that it is useful to differentiate between macroscopic physical properties and
microscopic effects. In utilizing macroscopic physical properties, it has also turned out to be advantageous to discriminate between structure-sensitive and structure-insensitive properties in developing nondestructive characterization of materials, the following steps are consistently required: (1) Reveal the structure-to-property-relationships. (2) Select the most suitable structure-to-property relationships. (3) Convert the structure-to-property-relationships to a nondestructive testing method. Some practical examples will be discussed. It is only in a few cases that natural interrelationships can be utilized in revealing the structure-to-property relationships, such as the diffusion behavior of the active layer of an hf-magnetic recording head. The electrochemical characterization of hardened layers on the base of backscattered ultrasound, where the well known relationship existing between the depth of hardened layer and the ultrasonic wave of flight can be directly used. In most cases empirical stochastic correlations have to be resorted to by adopting multiple regression analysis between two or more parameters. Various aspects have to be considered in selecting the most suitable physical property. The crucial aspect is that of the highest sensitivity, i.e., which of the properties studied shows the most marked change for the process parameter to be ascertained. Other factors too play an essential role: which reproducibility or measuring uncertainty can be expected and how can a change in property be translated into a nondestructive method? As examples are discussed: the measurement of carburized depth of austenite steel tubes during the cracking process, the hardening of ball bearing steel, the tempering of malleable cast iron using coercive force - NDE of tensile strength of cast iron using magnetic as well as elastic properties, - the detection of chill zones in cast iron using magnetic distortion factor.

2:00 PM S3.2 MICROSCOPIC TECHNIQUES FOR CHARACTERIZATION OF MAGNETIC LAYERS. Irz Alpeter, Udo Netzelmann, Norbert Meyendorf, Fraunhofer Institute for Nondestructive Testing (ZfTI), Dept. of Materials Characterization, Saarbrücken, Jürgen Hoffmann, University of Applied Sciences, Dortmund, Wolfgang Nickel-Pecher, Elektrische Magnetics, Nürnberg, Hubert Grimm, IBM, Mannheim, GERMANY.

In recent years there was an increasing interest in magnetic materials and especially amorphous and nanocrystalline layers. Magnetoresitive and magnetoviscoelastic effects are of high interest for applications of these materials in sensors, actuators and memories. Therefore there is a requirement for methods to characterize magnetic and mechanical properties of magnetic materials. Some novel micromagnetic techniques will be presented in this paper. Local thermal modulation by a laser beam is used to image magnetic domain structure in metallic glasses and in polycrystalline nickel, taking profit of the temperature dependent magnetization. When the amorphous test objects are magnetized to a sufficient level, the photothermally modulated stray field (PMS) images show different structures. The later are corresponding to results from Barkhausen noise microscopy, where Barkhausen noise is measured during continuous variation of the external magnetizing field. Barkhausen microscopy is applied to evaluate the distribution of magnetic and mechanical properties such as coercivity, hardness and residual stress in polycrystalline sendust layers and in very thin magnetoresistive layer systems. For very thin layers (10-20nm) only a small number of Barkhausen jumps are observed during one hysteresis loop. The noise signal shows a characteristic position-dependent anomalies. These give detailed information about remagnetization processes. Eddy current microscopy is sensitive to fluctuations in conductivity, permeability and the distortion of the magnetic or topography of the tested layers. Amplitude and phase images at different frequencies can be measured simultaneously. Using a least square fit technique, images of material parameters depending on \(\mu\) and \(\sigma\) can be calculated.

2:15 PM S3.3 INFRARED EVALUATION OF HEAT GENERATION DURING THE CYCLIC DEFORMATION OF A CELLULAR AL ALLOY. Akio Hasegawa, Robert M. Miller, Division of Engineering, Applied Sciences, Harvard University, Cambridge, MA; Anthony G. Evans, Materials Institute, Princeton University, Princeton, NJ.

Time-Resolved Photoacoustic Spectroscopy has been used to study the cyclic deformation of a cellular Al alloy. The results show the potential of this technique to study the heat generation associated with cyclic deformation of cellular structures.

2:30 PM S3.4 GIANT MAGNETORESISTANCE IMAGING FOR NDE OF CONDUCTIVE MATERIALS. E.S. Bleeck, G.S. Althoff, A.R. Stallings, Y.H. Spooner, J.P. Ahearn, Instrumentation and Sensors Department, TPL, Inc., Albuquerque, NM.

TPL has developed an electromagnetic sensor suite based on a synergistic coupling of conventional field generation electronics with high performance magnetoresistors. The result is a powerful new sensing technology with broad implications for detection of deeply buried anomalies in conductive materials. With high noise and high sensitivity from DC to well over 1 GHz, and a characteristic dimension of only a few microns, these sensors provide performance nearly equaling that of a superconductive quantum interference device (SQUID) but with substantially improved spatial resolution, ruggedness and a much lower fabrication cost. As with any such high-sensitivity technology, substantial efforts have been focused on designs that minimize the effects of environmental noise sources. So-called nulling configurations have been used to detect and image defects to a depth of nearly 0.5 in aluminum. These sensors have interfaced to industry-standard aircraft scanning systems to allow rapid image-based inspection of aircraft structures.

SESSION 4: NDE FOR CONCRETE AND STEEL

Chair: Rick L. Paul

Monday Afternoon, November 29, 1999

Provincetown/Olreans (M)

3:30 PM S4.1 IMPACT-ECHEL - NEW GENERATION OF NONDESTRUCTIVE IN SITU TEST SYSTEMS FOR CONCRETE STRUCTURES. A.T. Meczek, M. Meczek, Wroclaw University of Technology, POLAND; P. Stroeven, Delft University of Technology, Delft, THE NETHERLANDS.

The paper evaluates the possibilities of using the Impact-Echel method for nondestructive examination of concrete. The general principles of this technique will be presented, as well. It was shown that spectrum analysis based on Fourier Transformation offers a powerful tool for the evaluation of experimental data. This will be discussed in the framework of in-situ test results, whereby
3:45 PM  S4.2 DUAL INFRARED THERMOGRAPHY AS A NDT TOOL FOR THE CHARACTERIZATION OF THE BUILDING MATERIALS AND CONSERVATION PERFORMANCE IN HISTORIC STRUCTURES. Antonio Monopolou, Nicolaos P. Avdelidis, Maria Kani, National Technical University of Athens, Dept of Chemical Engineering, Materials Science and Engineering, Athens, GREECE; Nikos K. Kanellopoulos, NCSR Demokritos, Materials Laboratory, Athens, GREECE.

Infrared thermography is a non-destructive investigation technique, which is largely used because of the outstanding advantages that is capable to provide in a variety of applications and especially for conservation purposes of historic monuments, where destructive sectioning is prohibited. In the present work, dual band infrared thermography (3.5 - 4.2 μm and 8 - 12 μm) is used, to evaluate the humidity distribution through capillary rise in reference samples of porous materials, in the laboratory, in order to validate the examination of real scale material systems in situ. The combined study of transport phenomena, in terms of vapor/moisture transport, in prototypes, simulating porous materials, under controlled environmental conditions (Relative Humidity 60-80% and Temperature 20-40°C), provides interpretation tools to the investigation of transport phenomena occurring at the masonry by IR Thermography. The investigation of porous samples consolidated by various consolidants, acting by filmogenic and deposition mechanisms, and of various binding materials, at various mixing conditions, admired with pozzolanic and crushed-brick additives and air entraining agents), permits to evaluate, in lab, the performance of conservation materials, regarding their compatibility to the porous building stones. Pilot investigation, which has been performed regarding ancient, Byzantine, Venetian and recent monuments in Greece, allows for the comparison among various types of historic mortars and restoration ones. Infrared Thermography allows for recording the thermal maps of the real surfaces under study and provides information on the differential behaviour of the various materials on the masonry scale regarding the water impregnation and evaporation phenomena, which are controlling the weathering effects in porous media. Hence, a macro and micro scale NDT is developed to evaluate compatibility of conservation interventions and materials.

4:00 PM  S4.3 CHARACTERIZATION OF HYDROGEN IN CONCRETE BY COLD NEUTRON PUMPED GAMMA-RAY ACCELERATION ANALYSIS AND NEUTRON INCOHERENT SCATTERING. Rick L. Paul, H. Heather Cheng-Mayer and Richard M. Lindstrom, Analytical Chemistry Division, National Institute of Standards and Technology, Gaithersburg, Interfaculty Reactor Institute, Technische Universiteit Delft, THE NETHERLANDS.

A combination of cold neutron prompt gamma-ray neutron activation analysis (PGAA) and neutron incoherent scattering (NIS) has been used for non-destructive characterization of hydrogen as a function of position in concrete. Measurements were made on 100 mm diameter semicircular cross sections cut from concrete cylinders of different compositions. For PGAA, each sample was irradiated in various locations using a 5 mm diameter neutron beam with a fluence rate of 8.3 x 10^6 cm^-2 s^-1. A total of 10 locations per sample were measured, as the sample was scanned across the beam in 10 mm increments. Prompt gamma-rays of chlorine, sodium, potassium, and aluminum were measured using a high purity germanium detector. Milligrams of each element were determined and converted to milligrams of oxide. The fraction of water at each location was calculated by dividing the mg of H2O by the sum of all oxides. NIS measurements were performed using a 5 mm diameter neutron beam, with a fluence rate of 2 x 10^8 cm^-2 s^-1. The sample was scanned across the beam at 5 mm increments. Scattered neutron yields were measured using a 44 cm^3 He detector. Because of the large incoherent scattering cross section of H, a rapid scan (10 min per point for < 0.5% counting statistics) was employed to determine the total amount of scattered intensity. The sample was subsequently baked at 112°C for 3.5 hours and the measurement repeated. The amount of scattered signal was found to have reduced by 25%. Polypropylene films were used to calibrate the amount of H in the form of water evaporated. The results were compared with those obtained by PGAA. These measurements demonstrate the feasibility of the techniques for 2 dimensional compositional mapping of hydrogen and other elements in materials. A possible future application is to determine the uniformity of hydrogen in concrete during drying.


Development for magnetometers utilizing highly-sensitive magnetic sensors such as superconducting quantum interference devices and micro-Hall sensors provides a new and promising technique for non-destructive evaluation (NDE) of magnetic materials, because of many advantages. Section of conventional techniques such as eddy current ultrasound and x-ray imaging, etc. We fabricated a scanning Hall-sensor microscope with an active area 50μm x 50μm. This microscope permits us to make highly-sensitive and coolant-free measurements at room temperature so that it can be used as a simple and conventional tool for NDE of magnetic materials. Using this, we succeeded in magnetic detection of small cracks (<10 μm and ~0.1 mm wide) in mild steels with a yield point of 29 kgf/mm² caused by a fatigue process, independent of whether or not the sample surface is covered with nonmagnetic paint. To check further its potential, we applied the microscope to mild steels having circular holes with various diameters from 0.2 to 1.2 mm), and succeeded in magnetic detection for the penetration depth of 10 μm, and determined the looks in mild steels through drastic changes in magnetic profile.

8:30 AM  S5.1 ULTRASONIC DETERMINATION OF MATERIALS PROPERTIES. Ming-ming Duan, Steven Pilgrim, NY State College of Ceramics at Alfred University, Alfred, NY.

A measuring system using an ultrasonic pulse-echo technique has been built to determine the acoustic, elastic properties of solid, glass and melts, materials as a function of temperature (RT to 1200°C). When coupled with the measurement of the coefficient of thermal expansion, this system can be used to determine the baseline shear and longitudinal moduli, Poisson ratio, and attenuation coefficients of refractory and glassy samples. From knowledge of these baseline data, the temperature and effective thermal gradient in furnace insulation can be determined. Such data obtained can be used to a) perform in-situ evaluation of wall-thickness and temperature gradients, b) improve the accuracy of modeling of furnaces and melters, and c) improve the understanding of high temperature structures. Results will be presented on a variety of commercial refractory materials and typical glass compositions.

8:45 AM  S5.2 EVALUATION OF ULTRASONIC ATTENUATION MEASUREMENTS FOR ESTIMATING NEUTRON EMBRITTLEMENT OF IRRADIATED RPV STEELS. Allen L. Hiner, Jr., Robert E. Green, Jr., The Johns Hopkins University, Center for Nondestructive Evaluation, Baltimore, MD.

In commercial nuclear power plants, neutron radiation emitted from the reactor core causes a degradation in the material properties of the low alloy steel composing the reactor pressure vessel (RPV). This degradation, termed neutron irradiation embrittlement, has various manifestations: elevation in strength properties, decreases in ductility, and increase in the brittle-to-ductile transition temperature. The latter is of principal importance to the safety of nuclear power plants, as various plant transient and accident scenarios could place the highly irradiated RPV in a highly stressed condition at a temperature for which fully ductile behavior of the vessel cannot be assured. Conventionally, the degradation in RPV steel fracture toughness caused by neutron embrittlement is monitored by surveillance cracks (though small specimens of the RPV steel are irradiated in the vessel as the plant operates and subsequently destructively tested), or evaluated through empirical calculations based upon the chemical composition of the RPV steel and the operating history of the plant. At present, there is no nondestructive method for directly measuring the actual condition of the steel composing the RPV. A method using measurements of ultrasonic attenuation is under study to provide a means of nondestructively measuring neutron embrittlement in fracture toughness. This method uses a pulse-echo immersion ultrasonic test method. The geometry of the test samples is identical to that used in RPV surveillance capsules, specifically the
Charyp Vonch specimen with a square 10-mm cross-section. To assess this test method, measurements have been made on four RPV materials representing eight different irradiation conditions.

9:00 AM *55.3* QUANTITATIVE CONTACT SPECTROSCOPY AND IMAGING BY ATOMIC-FORCE ACOUSTIC MICROSCOPY. W. Arnold, Fraunhofer-Institute for Nondestructive Testing, University, Saarbrücken, GERMANY.

In Atomic Force Microscopy (AFM) deflection of a microfabricated elastic beam with a sensor tip at its end is used to generate high-resolution images of surfaces. Dynamic modes, where the cantilever is vibrated while the sample surface is scanned, belong to the standard equipment of most commercial instruments. With a variety of these techniques, such as Force Modulation Microscopy, Ultrasonic Force Microscopy, Scanning Local Acceleration Microscopy, or Pulsed Force Microscopy, images can be obtained which depend on the elasticity of the sample surface. However, the determination of Young’s modulus of a sample surface with AFM is still a challenge especially when stiff materials such as hard metals or ceramics are encountered. In this contribution the evaluation of the cantilever vibration spectra at ultrasonic frequencies is presented in order to discern local elastic data quantitatively. Nanocrystalline magnetic materials, multidomain piezoelectric materials, silicon and other materials have been examined, some as a reference material. In addition images will be shown which display the domain structure of PZT ceramics with the contrast caused by the local elasticity. Nonlinear phenomena in the contrast spectra will be discussed as well.

9:30 AM *55.4* QUANTITATIVE MEASUREMENT OF LOCAL CARRIER CONCENTRATION OF SEMICONDUCTOR FROM DISPLACEMENT-INDUCED-VOLTAGE CURVE USING A SCANNING VIBRATING TIP. Yutaka Majima, Yutaka Oyama, Mitsuhiro Iwamoto, Tokyo Institute of Technology, Dept of Physical Electronics, Tokyo, JAPAN.

A new scanning probe method for evaluating the local carrier concentration of a semiconductor is presented. The distance between a semiconductor surface and a vibrating tip is changed sinusoidally, and is adjusted as small as some few nm, so that tunneling current $I_T$ between the semiconductor surface and the vibrating tip flows. Displacement current $I_D$ due to the change in electric flux from the semiconductor surface to the vibrating tip flows periodically in accordance with the vibration of the tip. $I_T$ also flows when the distance becomes a minimum value. Both $I_T$ and $I_D$ flow simultaneously when a direct tip voltage $V$ is applied, and these two currents are separated by using a two-phase lock-in amplifier. $I_T-V$ curve is analyzed by taking into account two-dimensional electric flux profile under the tip. In $V$ range within which majority carriers are accumulated, $I_T-V$ curve is extrapolated by a straight line. On the contrary, the slope of the curve decreases as $V$ is reduced from $V$ range of the major carriers, indicating that the majority carriers are depleted at the center of the Si surface. The theoretical $I_T-V$ curves are in good agreement with the measurement in $V$ region where the majority carriers are both accumulated and depleted. The local carrier concentration can be made by making the theoretical $I_T-V$ curve fit the experimental results without contacting the semiconductor surface in a submicron order of a lateral resolution with 10 microns of a top radius of the tip.

9:45 AM *55.5* NONLINEAR ULTRASONIC PARAMETER IN PRECIPITATE-HARDENED STEELS. D. C. Hulley, D. Bahr, and P.T. Parlascher, Materials Reliability Division, National Institute of Standards & Technology, Boulder, CO.

Radiation-enhanced formation of ultrafine, copper-rich precipitates can embrittle reactor contain vessel vessels, and thus shorten their useful lifetime. As part of a feasibility study of nondestructive methods, for embrittlement of high-strength, low-alloy steel with 1.3 % copper by mass. The final aging treatment (temperature and time) was varied to obtain specimens with a range of hardness and precipitate configuration. Harmonic generation experiments using an infrared Michelson interferometer were performed to determine the nonlinearity parameter $\beta$ for each specimen. The ultrasonic longitudinal velocity $v_L$ and attenuation $\alpha(\omega)$ were also measured separately in order to correctly calculate $\beta$. Microstructure to interpret our results was obtained by x-ray diffraction line broadening measurements of the mean lattice strain $\epsilon$. Measurements showed only small variations in $v_L$ and $\alpha(\omega)$ and no consistent trends with hardness or strain. However, an approximately linear relationship between $\beta$ and $\epsilon$ was observed. Our results will be interpreted using a microstructural model for harmonic generation that includes the effect of precipitates. Relationships between lattice strain, state of precipitate coherency, and hardness will be discussed and used to interpret the nonlinear ultrasonic behavior.

10:00 AM *55.6* REAL-TIME MONITORING OF ACOUSTIC LINEAR AND NONLINEAR BEHAVIOR OF TITANIUM ALLOYS DURING CYCLING LOADING. Jerome Frouin, Jeong. K. Na, Theodore. E. Martin, University of Dayton, Center for Materials Diagnostics, OH.

An in-situ technique to measure sound velocity, ultrasonic attenuation and acoustic nonlinear property has been developed for characterization and early detection of fatigue damage in aerospace materials. A previous experiment using the F2F technique on Ti-6Al-4V dog bone specimen fatigued at different stage of fatigue has shown that the material nonlinearity exhibit large change compared to the other ultrasonic parameter. Real-time monitoring of the nonlinearity may be a possible tool to characterize early fatigue damage in the material. For this purpose we have developed a computer software and measurement technique including hardware for the automation of the measurement. New transducer holder and special grips are designed. The automation has allowed us to test the long-term stability of the electronics over a period of time and so the reproducibility of the linearity of the system. A real-time experiment has been performed on a dog-bone specimen from zero fatigue all the way to the final fatigue. The first time that such in-situ experiments were performed for measuring in real-time the linear and nonlinear acoustical parameters as a function of fatigue damage. The new methodology and the related new software and instrumentation enable an experiment under in-service loading conditions. Real-time health monitoring of the material can greatly contribute to the understanding of material behavior under cyclic load. However, this technique is not limited to titanium, but can also be used for studying metal fatigue, composite, or other mechanical and/or thermal testing conditions. This new methodology was developed with an objective to predict the initiation of fatigue microcracks, and to detect, in-situ, fatigue crack initiation as well as to quantify early stages of fatigue damage.

SESSION 56: ELECTRIC AND OPTOELECTRONIC NDE
Chair: Robert Gilmore
Tuesday Morning, November 30, 1999
Provincetown/Orleans (M)

10:30 AM *56.1* IMPULSE SPECTROSCOPY INVESTIGATION ON THE LOW-TEMPERATURE DEGRADATION OF TETRAGONAL ZIRCONIA. I. INFLUENCE OF THE MATERIAL CONDITIONS. A.P. Santos, Centro de Desenvolvimento da Tecnologia Nuclear - CDTN/CNEN, Belo Horizonte, MG, BRAZIL; R. Z. Domingues, Universidade Federal de Minas Gerais, Dept of Chemistry, Belo Horizonte, MG, BRAZIL.

Impulse spectroscopy [15] is a powerful tool for evaluating the microstructural evolution in ion conductor ceramics. This nondestructive technique has been used for the characterization the low-temperature degradation of yttria-stabilized tetragonal ZrO$_2$ polycrystals (28 mol%). Annealing experiments, in air, were conducted at 250°C and subsequently at 350°C, 450° and 600°C, both with and without the presence of the sputtered silver electrodes. Each experiment lasted for 4 hours. The aim of this work was to define some experimental conditions for the future experiments. The degraded surfaces were examined by both X-ray diffraction and IR measurements. This latter was performed at frequencies ranging from 5Hz to 13 kHz at 350°C. Superficial transformations resulting from the aging lead to the raising of an extra relaxation in the impulse spectra, which evolves with the degradation magnitude. The electrical characterization of the degradation phenomenon cannot be achieved during the annealing because the silver coating retards the degradation. The samples annealed with silver showed less monoclinic phase content and smaller extra semicircle than those with no coating. The degradation was better observed when the temperature/time was 350°C / 4 hours. It permitted a good definition of the semicircle in the impulse diagrams without affecting the degraded surface. Samples annealed at 450° and 600°C showed gradual reductions on the amount of monoclinic zirconia and also on the extra semicircle resistance.

10:45 AM *56.2* ELECTRICALLY BASED NON-DESTRUCTIVE MICROSTRUCTURAL CHARACTERIZATION OF ALL CLASSES OF MATERIALS. Rocco A. Gerhardt, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA.
It is well known that dissimilar phases will have different electrical responses under an applied electric field. The actual response will be dependent on the material’s individual resistance, capacitance and/or inductance values. Thus, when two dissimilar phases are joined together, additional interactions may occur which will be dependent on the type, shape, and size of the material and the resistivity measurements have been used in the past but they are often not sensitive enough to detect small changes caused by the presence of defects, grain boundary layers or cracks. In this presentation, we will discuss the usage of impedance and/or for dielectric spectroscopy for the detection of microstructural features at all length scales in a wide variety of materials. Examples of the types of features that have been detected non-destructively include: (1) pores in thick thermal barrier coatings, (2) formation of surface layers due to moisture absorption in a wide variety of materials, (3) orientation of fibers and whiskers in ceramic matrix and polymer matrix composites, (4) crack detection in metals and (5) stacking faults in semiconductors to name a few. Many more examples can be found in proceedings books of two MRS symposia [vol. 411 and 500]. While data acquisition can easily be obtained using a variety of commercially available equipment, data interpretation is more complex. This talk will concentrate on the above examples and highlight the most important steps needed in order to obtain useful information from the materials being measured. Proper data collection and data interpretation of a specific set of materials can lead to in-line process monitoring, quality control monitoring, mechanical damage monitoring and environmental degradation monitoring of those materials.

11:15 AM AS 6.3
NON-DESTRUCTIVE DIELECTRIC ASSESSMENT OF WATER PERMEATION IN COMPOSITE STRUCTURES. Richard A. Ceplick, Pascal Bonnard, Gordon S. Armstrong, Eric Bonnard, Dept of Physics and Astronomy, University of Strathclyde, Glasgow, UK, William M. Banks, Dept of Mechanical Engineering, Strathclyde University, Glasgow, UK, Robert L. Crane, Air Force Research Laboratory, OH.

Over the last ten years, the application of high frequency dielectric spectroscopy techniques for the assessment of composite structures has been investigated. Novel approaches to assess non-destructively the evolution during aging of adhesively bonded aluminum structures, carbon fibre reinforced plastic (CFRP) bonded structures and bulk glass fiber reinforced plastic (GFRP) structures are presented in this paper. The applicability and limitations of dielectric measurements, in both frequency and time domain, to the monitoring of water ingress at temperature between 30°C and 65°C are examined. The correlation between gravimetric, mechanical and high frequency dielectric spectroscopy data demonstrates the suitability of the technique regarding the characterization and assessment of composite structures having been exposed to hot and moist environments and illustrates its potential as a non-destructive evaluation (NDE) technique. The dielectric time domain response (TDIR) technique does not only indicate a new way to assess the state and integrity of adhesively bonded structures but are also producing new insight into the application of dielectric spectroscopy to non-destructive evaluation of composite structures. The approach to high frequency analysis of bulk GFRP using a capacitive probe technique indicates the potential portability of the technique for in-situ measurements.

11:30 AM AS 6.4
NONDESTRUCTIVE DAMAGE EVALUATION OF ELECTRO-MECHANICAL COMPONENTS USING A HYBRID, COMPUTATIONAL AND EXPERIMENTAL, APPROACH. Cosme Barlong and Raymond J. Poppovitch, CHILP. Center for Holographic Studies and Laser Micro-Technologies, Mechanical Engineering Department, Worcester Polytechnic Institute, Worcester, MA.

With the electronic industry being one of the most dynamic, in terms of new technologies, electronic packages have to be designed and optimized for new and ever more demanding applications in relatively short periods of time while satisfying electrical, thermal, and mechanical consideration, as well as cost and manufacturability. In addition, reliability and durability have to be taken into consideration. As a consequence, effective quantitative methodologies, such as computational and experimental, should be applied in the study and optimization of electro-mechanical components. For applications subjected to repeated cyclic loading conditions, fatigue failure is an important factor. Fatigue failure usually appears as a relatively high frequency, low-level, cyclic loading. Large numbers of loading cycles implies extensive testing which is not compatible with short development time periods. Therefore, effective nondestructive quantitative techniques for detecting, locating, and sizing damage early in the design process are required. In this paper, a novel hybridized use of nondestructive, noninvasive, remote, full field of view, quantitative opto-electronic holography techniques with computational modeling is presented. The hybridization is illustrated with a representational application, which shows that the combined use of opto-electronic holography techniques and computational modeling provides an effective engineering tool for nondestructive study of electro-mechanical components subjected to fatigue loading conditions. With the representative application, it is also demonstrated that novel non-destructive and experimental techniques should be applied to an engineering application, because applying only one of the techniques can provide erroneous information. Using this hybrid approach, displacements and strains are determined with an accuracy of 0.1 mm and 0.001%, respectively, and provide indispensable data for testing and development of reliable electronic packages.

SESSION 57: NDE FOR SILICON WAFERS AND INTERFACES
Chair: Matthew W. Skoler
Tuesday, Afternoon, Monday, 30 June, 1999
Provincetown/Olans (M)

1:30 PM 57.1

A newly-developed technique for the simultaneous characterization of the oxide-silicon interfacial and of bulk impurities was used for a systematic study of the nitridation process of thin oxides. This technique is based upon surface recombination velocity measurements, and was previously validated by comparison with capacitance-voltage measurements of surface state density. As no capacitor structure is required, this technique is very suitable for the characterization of interpoly interfaces. In addition, it can accurately provide the distribution of surface recombination velocity over wafer surface, and therefore gives important information about process uniformity. Oxides grown both in dry and in wet environments were considered, and nitridation processes in N2O and in NO were compared. N2 annealing processes and the effect of oxidation temperature and duration were also studied, and Rapid Thermal Oxidation /Nitridation processes were compared to conventional furnace nitridation processes. The surface recombination velocity was correlated with nitrogen concentration at the oxide-silicon interface obtained by Secondary Ion Mass Spectroscopy (SIMS) measurements. Additional information on interface chemical states was provided by x-ray photoemission spectroscopy (XPS). Surface recombination velocity (hence surface state density) decreases with increasing nitrogen pile-up at the oxide-silicon interface, indicating that in nitrided interfaces surface state density is limited by nitridation. In addition, as nitridation proceeds a positive oxide charge is developed. Nitridation processes are much more effective than NO treatments in the formation of a nitrogen-rich interface layer, and, as a consequence, in surface state reduction. Surface state density was measured in fully processed wafers before and after constant current stress. After complete device process surface states were neutralized by hydrogen passivation, however they are reactivated by the electrical stress, and surface states results after stress were correlated with data of surface recombination velocity in reprocessed wafers.

1:45 PM 57.2
IR TOMOGRAPHY OF LIFETIME AND DIFFUSION LENGTH OF CHARGE CARRIERS IN SEMICONDUCTOR SILICON INGOTS. Vladimir D. Akmether, Nikolai V. Feseev, Ins Semiconductor Physics, Novosibirsk, RUSSIA.

Lifetime as well as diffusion length, closely related to it, is one of the main parameters indicative of the quality of silicon as a semiconductor material. Usually, the lifetime in ingots is estimated from measurements on two disks cut from the ends of an ingot (ASTM F1535-94). This characterization method, however, has the following disadvantages: 1) no direct information about the lifetime inside ingot is available, 2) a certain part of the ingot is lost because of fabrication of the disks and 3) it is difficult to accurately determine the bulk lifetime because of the influence of surface recombination (ASTM F1535-94). In this report, a new nondestructive method for measurement of three-dimensional pattern of both the lifetime and diffusion length in silicon ingots, free from the above disadvantages, is presented. This method consists in the in-situ evolution of an injected pocket of charge carriers. The pocket of injected carriers is generated by the ray of a pumping laser with a wavelength of 1.15-1.25 microm, and monitored by a probing laser beam with the wavelength 3.38 microm, via free-carrier absorption. Depending on
the distance between the axes of the rays in the ingot under study, as well as on the ratio between the diameter of the rays and the diffusion length, which are determined in the electronic bimorphs. These parameters are determined in the wafer-like structures in Si$_x$Ge$_{1-x}$ layers. The ratio between the diameter of the rays and the diffusion length is crucial for the stability of the wafer-like structures in Si$_x$Ge$_{1-x}$ layers.

Germinium composition and strains in epitaxial Si$_x$Ge$_{1-x}$ layers are the essential parameters influencing the electronic properties and the stability of the wafer-like structures in Si$_x$Ge$_{1-x}$ layers. Si$_x$Ge$_{1-x}$/Si heterostructures for various applications, such as heterojunction bipolar transistors (HBTs), strain-engineered field effect transistors (SEG-FETs), and long wavelength photodetectors, are designed to exploit the unique electronic properties of Si$_x$Ge$_{1-x}$ layers. The composition and strain in Si$_x$Ge$_{1-x}$ layers are determined using high-resolution imaging techniques, such as HRTEM and X-ray diffraction. The strain is determined using high-resolution X-ray diffraction and Raman spectroscopy, while the composition is determined using X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS).
many cases the contrast achieved with x-ray topography is not enough for defect visualization. For example, important post-implantation defects, such as microcracks, cavities or bubbles, are invisible in x-ray images. In order to enhance the x-ray sensitivity we introduced into the crystal high-frequency surface acoustic waves (SAWs), having a 5-10 µm-wavelength. Traveling SAWs cause a long-range variation of elastic deformation which, at a given moment of time, exhibits wavelength-periodicity over a whole crystal area. X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Comparison of regions with different wavefronts shows that wavefronts are distorted due to scattering processes. Correspondingly, the ruler in the x-ray image becomes bent, facilitating visualization of defects. Then if the SAW is used in combination with this idea, we first need to freeze the SAW, because time merging of SAW deformation completely destroys our ruler. This freezing is done using a threestage mode of measurement, i.e. by synchronizing the x-ray flash frequency with the SAW frequency. Synchrotron radiation is ideal for this purpose, because the incoherent radiation practically has the required pulse structure. In this paper the abilities of this technique are illustrated by x-ray topographs taken under 300-800 MHz-SAW excitation from LiNbO₃-based SAW devices with He-implemented waveguide layers. Experiments were performed at the European Synchrotron Radiation Facility (ESRF) with an x-ray flash frequency of 3.08 MHz. X-ray topographs showed well-resolved individual wavefronts of traveling SAWs as well as their distortions due to the SAW scattering on local inclusions. Secondary spherical acoustic waves were observed as a result of the strong SAW interaction with He bubbles.

3:45 P.M. SS.2
THE DESIGN OF POINT-CONTACT PHONOMETER FOR QUANTUM AND RESONANCE HEAT TRANSPORT IN ATOMIC SCALE CONTACTS. Alexander G. Skokov, Dmitry B. Kasper

The elimination of nanometer wiggles makes possible the characterization of transport properties of the point contacts of atomic scale. The combination of the ballistic phonons excited by the point contact to the resistivity of the metallic nanowire opens the possibility of electrical measurement of the quantum phonon flux. The combination of the surface contact and metallic nanowire treated as a new device point contact phonometer. We investigate the ballistic phonon heat flow in the regime in which the phonon characteristic wavelength is of the same order as point contact diameter. In this regime the wave character of the phonons should be observed. We show that the energy flux due to monochromatic phonons is described by the formula similar to the Landauer equation for electron conductivity. The calculations enable the description of local temperature heat transfer anomalies recently measured in point contacts [J. Phys.: Condens. Matter 10 (1998) S118-S120].

4:00 P.M. SS.3
IN SITU SPECTROSCOPIC ELLIPSOLOGY FOR THE REAL TIME PROCESS CONTROL OF PLASMA ETCHING OF SILICON NITRIDE. L.G. Rosen, T. Parent, B. Fidan, and A. Mudrakur, Center for Intelligent Manufacturing of Semiconductors (CIMOS), Departments of Mathematics and Materials Science, University of Southern California, Los Angeles, CA.

Real time feedback control of semiconductor plasma etching is becoming desirable to achieve the degree of process reproducibility demanded by the increasing strict requirements on high-volume production of advanced semiconductor manufacturing. Spectroscopic ellipsometry (SE) is commonly used in-situ sensor for dry etching. SE measures the change in the polarization state of light reflected from a surface. The change in the polarization state of the reflected light is dependent on the wavelength of light, the angle of incidence, the structure of the sample and the optical constants of the sample. Sample properties of interest into which ellipsometry is sensitive are obtained by fitting a model to the experimental ellipsometry data where the sample property or properties of interest are the fit parameters. The strength of ellipsometry lies in its sensitivity to film thickness and the non-destructive nature of the measurements. In the present work, we describe the design, testing and evaluation of SE based real time feedback controllers for etch rate regulation and etch depth endpointing in C₄F₉O₂ plasma etching of silicon nitride films. A fixed gain controller as well as an adaptive, variable gain controller are designed, tested, and evaluated. The feedback variable for both is the current etch rate as determined from the in-situ SE measurements of the film thickness. Both controllers compensate for drifts in etch rate which occur during an etch. In addition, the ability of the variable gain, adaptive controller to learn from previous etch runs and adaptively adjust the run-to-run variability inherent to plasma processing is examined. Experimental results which include comparisons between controlled and uncontrolled etches and between adaptive and non-adaptive controllers will be discussed. This work is supported by AFOSR/DARPA under the MURI program.

4:15 P.M. SS.4
POLARIZED RAMAN SPECTROSCOPY AS A NONDESTRUCTIVE CHARACTERIZATION TOOL FOR INVESTIGATING THE ORIENTATION OF FLUORINATED POLYMERS. Mei-Wei Tan, John F. Rabolt, Department of Materials Science and Engineering, University of Delaware, Newark, DE.

This paper demonstrates the use of Polarized Raman Spectroscopy (PRS) as a non-destructive characterization tool for the analysis of fluorocarbon-containing polymeric films. PRS, commonly found in electronic and biomedical devices. The recent advance in photonic and fiber optic technologies has made the once bulky, hard-to-use Raman spectroscopy instrumentation a lightweight, compact analytical tool capable of point-and-probe operation. In this study, polytetrafluoroethylene (PTFE) and polyhexafluoroarylene (PFPE) samples with highly oriented CF₂ moieties are measured with different combination of polarizer and analyzer orientations. From this experiment results along with a detailed group symmetry analysis, Ramans-active bands associated with different symmetry species can be assigned for various samples. Ratios between Ramans bands with A₁ symmetry, located at 733 cm⁻¹ and 1391 cm⁻¹, respectively, are used for the determination of the degree of orientation in these fluorinated samples. It is found that the I₃₃/I₇₃ ratio varies from as low as 1.5 for a film oriented parallel to the polarizer / analyzer direction, to as high as 12.0 for a film oriented perpendicular to the direction of the polarizing optics. This result strongly suggests that PRS can be applied to material processing environments as a non-destructive method to obtain molecular orientation information.

4:30 P.M. SS.5

Progress in metal interconnect technology is becoming increasingly critical for the semiconductor integrated circuit industry. Increased requirements to thickness uniformity of metal films, raising number of layers, and introduction of materials call for a high-precision measurement technique suitable for in-line metrology on product wafers. The laser-induced grating technique also called ISTS (Impulsive Stimulated Thermal Scattering) provides metal film thickness measurements with 1% level accuracy over a wide measurement time. The technique is based on the laser generation and detection of surface acoustic waves, and can be realized in a compact and robust optical set-up. The challenges are the ability to measure the thickness of several layers simultaneously, measurements on very thin (<200 Å) films and on small structures, and eliminating the effect of parasitically scattered light. Solutions to these problems and applications of the technique to the control of different processes involved in metal interconnect technology will be discussed.

SESSION 30 POSTER SESSION
NONDESTRUCTIVE METHODS FOR MATERIALS CHARACTERIZATION
Chair: George Budniki

Tuesday Evening, November 30, 1999
8:00 P.M.
Exhibition Hall D (H)


TPL Inc. has developed an instrument for determining the surface area (BET) and pore size distribution (BJH) of thin films in their deposited state. The method is based on a synergistic combination of a surface mode ultrasonic measurement and gas adsorption techniques, and is compatible with most materials and deposition techniques. The technique has been applied to many materials up to 100 µm thick, and has been used to characterize surface features as small as 20. Unlike traditional BET methods, the ultrasonic technique does not require removal of the film from its substrate and a complete characterization can be completed in less than two hours. The technique makes use of the inherent sensitivity of ultrasonic waves to density changes. As the film is dosed with the interrogation gas, an ultrasonic wave is used to detect the amount of gas adsorbed by the film as a function of partial pressure. Data is acquired for both adsorption and desorption cycles.
SO. 3 EXPERIMENTAL SIMULATION OF CONTAMINATION ARISING FROM ELECTRO ROCKET ENGINE-JET ON SURFACE OF SPACECRAFT UNITS. A.A. Chirv, A.B. Nadiadze, V.V. Shaposhnikov, Moscow Airspace Institute, Moscow, RUSSIA; V.K. Egorov, Lab. Nuclear Res. IPM RAS, Chernogolovka, Moscow Dist., RUSSIA.

The longevity of spacecraft units and primarily of its solar batteries is pollution dependent. The electric rocket engines (ERE) of spacecraft are the same assemblies being largely responsible for the pollution of external spacecraft surfaces. The angle concentration of element contamination in xenon jet of ERE (SPD - SPQ type) could show the level of its responsibility in pollution problem was studied. The investigation were carried out in conditions of a model vacuum 1*10⁻⁶ torr. The calculation of the plasma jet parameters and experimental measurement showed that the main deliver of the pollution was the ceramics of isolating rings placed in the discharge space. The material of the rings was created on basis of BN compound. The variation of the element contamination on a surface were carried out by means of UAS of H⁺ ions. The angle contamination analysis by ERE plasma jet was studied by comparative RBS He⁺ and nuclear reactions diagnostics of the experimental and control Be targets were petrified on different materials surfaces. To prevent the gasifying effect of contamination atoms precipitated on Be substrate they were parked up to Cu films formed by additional Cu-plate pulverization by Xe jet. The control Be targets were parked up by Cu too but theirs were screened from the stream to be used as background signal. The RBS and RIS of ions were received by using of ion beam complex Sols-3 IPMT RAS. The investigation showed that the R-contamination is not depend on the angle to jet axis and presents in the jet in certain near 1*10⁻¹⁰ ions/cm². Moreover the jet plasma contains Cu+, P+, and Si ions too. Their concentration in the jet are defined more precisely.

SO. 4 OPTICAL CHARACTERIZATION OF RELIABILITY IN BARIUM TITANATE CAPACITORS. Michael Biegalski, Susan Toller-McKinstry, Yedl Taur, Material Research Laboratory, Pennsylvania State University, University Park, PA.

A major problem with perovskite ferroelectric devices such as multiyayer capacitors (MLCs), is the reliability of the dielectric material under DC electric fields. The failure of BaTiO₃ based MLCs has been traced to the electromigration of oxygen vacancies. MLCs with Ni electrodes are processed in a reducing atmosphere, this increases the oxygen vacancy concentration and decreases the reliability. The reliability of BaTiO₃ MLCs can be dramatically increased through the addition of some rare earth elements, namely yttrium, helium, and dysprosium. However, the mechanism through which the rare earth elements increase the reliability of BaTiO₃ is not fully understood. To investigate this problem spectroscopic ellipsometry (SE) was used. SE is a nondestructive tool which can monitor or the accumulation of damage in BaTiO₃ by measuring the changes in the optical properties of the material. In this study undoped and yttrium doped barium titanate samples fired in air and reduced atmospheres were examined. These samples were analyzed prior to and after degradation in a DC electric field. Results on sensitivity of SE to damage accumulation as well as the impact of rare earth doping on oxygen vacancy concentration and mobility will be presented.

SO. 5 CHARACTERIZATION OF COPPER SURFACES USED IN ELECTRONIC CIRCUIT BOARDS BY REFLECTANCE FT-IR.

James M. Sloan and Charles G. Pengaur, U.S. Army Research Laboratory, Polymers Research Branch, APG, MD.

Organic and organo-metallic coatings are presently being applied over bare copper as an approach to improve the plasmosphericity of circuit boards. Organic solubility preservative coatings (OSPs) are environmentally and economically advantageous over the more commonly used lead based coatings. Problems arise in assessing the solubility of the bare copper and the integrity of the organic coating. Spectral reflectance Fourier Transform infrared spectroscopy (FT-IR) has been utilized to monitor the formation of Cu oxides occurring on these copper substrates used in the manufacturing of electronic circuit boards. By measuring the oxide and protective coating characteristics of these surface metallizations, their solubility behavior can be evaluated in a controlled manufacturing environment. OSP coated test specimens were subjected to Hot-Dry and Hot-Wet environmental conditions using MIL-STD-202F as a guide. The resulting FT-IR spectra provide clear evidence for the formation of various Cu-Oxide species over exposure time. IR spectral bands consistent with Cu₃O and Cu₂O formation appear, while minimal deterioration to the OSP coating was observed. The appearance of the Cu oxide layers grew steadily with increased environmental exposure. IR data can be directly correlated to solubility-performance as evaluated by wet balance testing.

SO. 6 METHOD OF PARAMETER CONTROL OF OXIDE FILMS ON CRYSTALS SURFACES. P. Kosačovič, T. Süsswarth, State Univ Ljubljana, Dept. of Physics, Ljubljana, UKRAINE.

In the present paper the method of three-layer spectroscopy of reflection in photon range of spectrum for nondestructive control of parameters of oxide films on crystal surface is considered. The essence of the approach consists in satisfying the condition $\delta + \phi = \frac{2\pi}{\lambda}$, where $\delta$ - the reflection index of film with thickness $d$, $\phi$ - the phase shift of the wave reflected from film-crystal bulk interboundary. Analytical dependence of reflection coefficient minimum frequency on the parameters $\delta$ and $n$ is determined as a solution of the equation: $\tan \varphi = \frac{\lambda}{2 \pi d}$. 

SO. 7 THERMAL PénéTRATION TIMES AS A NONDESTRUCTIVE MEASURE OF ORIENTATION IN POLYMER FILM.

Nancy M. Hinds, Hinds Instruments Ltd., Fredericton, NB, CANADA; Robert Samuel, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA.

Material properties depend on their fabricated structure and...
orientation. It is thus important to develop rapid nondestructive techniques that will both allow determination of the orientation fabrication, and to occur over times that is result of subsequent in service environmental conditions. The present study examines the relationship between the thermal penetration time and planarity in polyimide films. Polyimides are advanced materials that have good high temperature stability, excellent dimensional stability, and excellent mechanical, electrical, and chemical resistance properties. The samples tested were specially prepared to range in orientation from three dimensionally random to highly plane. The most common orientation is characterized by waveguide coupling techniques, while the thermal penetration time and thermal conductivity measurements utilized a new rapid non destructive high temperature instrument. This correlation will be the first time thermal penetration time has been measured by modified hot wire techniques and related to the internal structure of a polyimide. The work contributes to a deeper theoretical understanding of heat transfer mechanisms and their relevance to the orientation. The thermal penetration time evaluation could provide a new tool in the arsenal of structural characterization techniques. This relationship between thermal penetration time and orientation is key for film manufacturers. Such a correlation has potential to speed the development cycles of new materials and assure properties during production and end-use applications.

**SO.11**

**COMPARISON OF TRANSIENT GRATING OPTOACOUSTIC F.normal measurement (ISTS) WITH OTHER TECHNIQUES FOR SEMICONDUCTOR THIN FILM MICROPHOTOGRAPHY**. Michael Grein, Alex A. Manzke, Matthew Bennett, Philips Analytical, Boston, MA; Todd C. Bailey, SEMATECH, Austin, TX.

A transient grating optoacoustic technique, Impulsive Simulated Thermal Scattering (ISTS), has been developed recently for nondestructive measurement of the thermal conductivity of thin films used in the semiconductor industry. This technique uses a transient optical grating to initiate an acoustic response in the sample film. Observation of the response of the film to the optical grating permits rapid measurement of film thickness for single and multilayer films deposited on insulating or metal substrates. ISTS can be used to measure film uniformity and to detect misprocessed films. In this presentation, ISTS will be compared to other noncontact film thickness measurement techniques used in the semiconductor industry, including electron microscopy (SEM/TEM), x-ray fluorescence (XRF), Grazing Incidence X-Ray Reflection (GIXR), and profilometry. The presentation will focus on a series of measurements of Cu metal layers and their underlying Ta and TaN barrier films. These films are important for non-generation integrated circuit manufacturing.

**SO.12**

**IN SITU MONITORING OF ALUMINUM NITRIDE GROWTH BY OPTICAL SPECTROSCOPY**. Stephen Alles, Patrick Javits, High Richardson, Ohio Univ, Department of Chemistry and Biochemistry, Athens, OH.

Group III nitrides have been under intense study because of their numerous possible applications in new electronic and optical devices. The use of in-situ spectroscopies is a potentially valuable technique in the study of nitride semiconductor growth as it is non-destructive and all data are collected during growth. Trimethylaluminum, ammonia, and manganese decarbonyl are used as precursors in the high temperature [1100 K] deposition of undoped and M-doped AlN performed by MOCVD at vacuum pressure. The radiation emitted by the sample passes through a monochromatic and is analyzed by FTIR spectroscopy for the optical modes of AlN, while an UV monochromator monitors interference fringes used to calculate film thickness. The spectral dependence of the film on the gas composition, and other properties are under investigation. The thickness data is correlated with the background-subtracted emission attributed to the emerging AlN film, then used to characterize various film properties accessible only by time and intensification techniques.

**SO.13**

**NEUTRON DEPTH PROFILING OF N- AND P-CHANNEL MOS DEVICES WITH USE OF A 300 MeV Neutron Beam**. S.C. McGarry, R.R. Vanfleet and J. Silcox, Cornell University, School of Applied and Engineering Physics, Ithaca, NY; G.P. Lannuzel and H. Chen, Mayber, Chemical Sciences and Technology Laboratory, NIST, Gaithersburg, MD; Present address: Department of Physics, Southern University and A&M College, Baton Rouge, LA.

We present results from the use of neutron depth profiling (NDP) to examine nondestructively the depth distribution of boron in a test wafer containing boron-doped N- and P-channel MOS devices. A guided beam of neutrons from the NIST cold neutron source was used to probe the specimen and charged particle products from the 197Au(n,2n) reaction were observed with an energy dispersive spectrometer. The neutron flux of 2.5 x 10^9/cm^2/keV at the target enabled a rapid assessment of the boron content. Experiment conditions allowed the structures to be probed to a depth of 25 mm while allowing determination to be made on most circuit structures of this type. Monte Carlo simulations of the energy spectra have been obtained that are consistent with a boron layer buried a few microns beneath the surface. Analysis of the energy spectra yielded boron concentrations of the order of 1 x 10^14 cm^-2, a layer thickness of approximately 0.5 microns. Details of the measurement technique and method of analysis with results will be described.

**SO.8**

**IN SITU SPECTROSCOPIC ELLIPSOMETRY STUDY OF THE OXIDE ETCHING AND SURFACE DAMAGING PROCESSES ON SILICON CARBIDE AND SILICON UNDER HYDROGEN FLASHPASSIVEATION**. M.J. Vierstra, J.Y. Mamo, J.R. Guzman, B.R. Weiner, University of Puerto Rico, Dept. of Chemistry; A. Martinez, University of Puerto Rico, Dept. of Physics. We have employed in situ spectroscopic ellipsometry in the monitoring and characterization of Si and Si substrates under hydrogen plasma conditions. These measurements were complemented with ex situ characterization including Raman, FTIR, XRD and SEM. The hydrogen plasma first attacks the native oxide layer of the substrate etching it away. This process can be slowed down by controlling the plasma energy, its distance to the substrate, and the substrate temperature. We monitored this process in real time with in situ spectroscopic ellipsometry in order to stop the plasma when the surface is at its cleanest condition. When the plasma remains on beyond this point, substrate surface roughening takes place followed by surface damage. Comparison of the in situ ellipsometry spectra with the ex situ characterizations allowed to establish correlations between the degree of surface roughness and damage, and the corresponding changes in the dielectric function of the material studied.

**SO.9**

**PHOTOLETIC IMAGING OF PROCESS INDUCED DEFECTS IN 300 mm SILICON WAFER. H.D. Geiler, Jenawave, Jen.; Germany; W. Kürner, O. Starbeck, Dresden, Germany.**

Shrink structural dimensions on enlarged wafer areas increase the influence of crystal defects on device function and integrity. Nondestructive and noncontact measurement techniques for rapid defect monitoring on the product wafer meet the demands of device integrity and cost efficiency. Temperature gradients during high temperature process steps cause lattice defects like slip lines, which can be detected by their local stress fields. Visualization of local stress is realized by scanning infrared depolarization measurement (SIDR) of laser light transmitted through the wafer. The ability of the SIDR equipment to record a full 300 mm wafer stress image in less than 4 minutes with lateral resolution of 100 mm allows to monitor crystal defects and their evolution during the technological process sequence. During each process step the high temperature equipment leaves its fingerprint at the support points of the wafer by creation of local temperature gradients. The slip lines caused by these gradients can be evaluated by their stress field and characterized by a specified danger potential. Further defects can be caused by problems like stress induced diffusion of precipitation degrading the device. The specific role of these marks for thermal defect generation and evolution is analyzed.

**SO.10**

**IRS AND ESR CHARACTERIZATIONS OF NANOCOMPONET THIN FILMS WITH FUNCTIONALIZED OR POLYMER THIOLATES AND METAL NANO PARTICLES**. Wenjin Zheng, Frank L. Leslie, Maria A. Morris, M. Marion, Y. Sun, Y. Xiong, Y. A. Kim, David C. Gilbert, David C. Dzochkman, Chun-Jin Zhong, State University of New York, Binghamton, NY.

A key to the ultimate technological applications of core-shell nanoparticle materials is the understanding of the interparticle structural and electronic properties in the nanocomposite. This paper focuses on the characterizations of such structural properties for the composite thin films derived from metal nanoparticles and functionalized or polymeric thiols using infrared reflectance spectroscopic (IRS) and electron spin resonance (ESR) techniques. IRS provides information on molecular packing and ordering of the shell components in the nanocomposite films, and the information directly relates to the nanocrystalline interactions and interfacial reactivity. ESR, on the other hand, probes the conduction electron spin resonance properties of the nanocrystalline cores, and the linewidth analysis can be correlated with the electronic microwavematerials in the cores as well. Both IRS results show the characteristic of the ESR line shapes, and their correlation with the nanoparticle core sizes and the organic shell functionality will be discussed.
90.14 REAL-TIME OPTICAL CHARACTERIZATION AND CONTROL OF HETEROEPITAXIAL GaN$_2$-In$_2$P GROWTH BY P-POLARIZED REFLECTANCE. N. Dettz, K. Ike, I. Lisko, and V. Woods, "Departments of Physics and Materials Science and Engineering, Center for Research in Scientific Computation, North Carolina State University, Raleigh, NC.

The development of surface-sensitive real-time optical sensors that are able to characterize thin film formation at the point where the growth occurs are essential not only to advance optoelectronic integrated circuits with stringent control of thickness and composition but also to gain a better understanding about the growth process itself. In this contribution we present the application of p-polarized reflectance spectroscopy (PRS) for real-time monitoring and control of pulsed chemical beam epitaxy (PCBE) during low temperature growth of epitaxial GaN$_2$-In$_2$P heterostructures on Si(001) substrates by PCBE. We present results on open- and closed loop control using nonlinear control algorithms (based on nonlinear Kalman filtering) that utilize the PRS signals to control thickness and composition during heteroepitaxial growth of GaN$_2$-In$_2$P on Si (001). The decomposition kinetics in the SRL is captured/approximated by a reduced order kinetic model and linked to the PRS response, surface reaction chemistry, composition, film growth rate, and film properties. These data are linked to compute the composition and thickness increment per time unit, utilizing the monitored PRS signal for validation.

90.15 IN SITU DIFFUSE REFLECTANCE SPECTROSCOPY FOR MEASUREMENT AND CONTROL OF III-V MOLECULAR BEAM EPITAXY. J.E. Giger, W.F. Tsang, W.R. Tharler, E.M. Vogel, D.A. Gajewski and J.C. Pollegrino, Semiconductor Electronics Division, National Institute of Standards and Technology, Gaithersburg, MD.

Measurement and control of substrate temperature is a common difficulty for molecular beam epitaxy (MBE) growth of semiconductor structures. Conventional MBE employs a thermocouple (TC) in the vacuum gap between the heater coils and the substrate. In steady-state, the temperature offset between the TC and the sample surface can be of the order of 100°C, but can be calibrated with pyrometry or by observing known surface changes with reflection high energy electron diffraction. Even under calibrated conditions, the TC reading can significantly lag the actual substrate surface temperature during transients, resulting in heterobar deposition well above or well below optimal growth temperatures. Diffuse reflectance spectroscopy (DRS) has been used in situ to monitor the absorption edge of the semiconductor substrate, which can be correlated to the temperature. Because it is a direct measure of the bulk substrate temperature, DRS does not suffer from the time lag during transients that thermocouples do. We have exploited this capability to monitor and control the substrate temperature with the DRS and substrate heater in active, closed-loop. To examine the effect of the temperature lag experienced during conventional MBE, we have grown identical pairs of GaAs / AlGaAs pseudomorphic high electron mobility transistors (pHEMTs). For one pHEMT in each pair, the input signal for substrate temperature control was the TC, for the other, it was the DRS. Under TC control, an overshoot of up to 50°C was observed during the temperature ramp following the lower temperature deposition of the InGaAs layer. This overshoot was eliminated when DRS control. The DRS controlled sample exhibits a strong peak in gate-drain conductance at 21 V depletion; this peak is absent in the TC controlled sample. We will discuss the implications of this difference for pHEMT growth procedures and for device performance.


Variable angle spectroscopic ellipsometry (VASE) was employed to study the anisotropic dielectric response, both ordinary and extraordinary, of hexagonal gallium nitride (GaN) thin films on important material for blue and UV light emitting device applications. The GaN films were grown by molecular beam epitaxy (MBE) on c-plane sapphire substrates. Room temperature isotropic and anisotropic mode VASE measurements were made, in the range of 0.75 to 6.5 eV, at the angles of incidence in between of 20 and 80 degrees. Evidence of anisotropy is observed via generalized VASE measurements, reflecting the nature of wurtzite crystal structure of GaN. VASE data simulations by isotropic and anisotropic models indicate that the anisotropic effect can be minimized to a negligible level at small angle of incidence, or to be maximized to a detectable level at angle of incidence. Thus the ordinary dielectric function are precisely determined by the VASE measurements at incident angles of 20 and 40 degrees in the range of 0.75 to 6.5 eV. The film thickness and surface roughness were also determined by the VASE analysis. The extraordinary dielectric functions are then fully determined by the VASE measurements at 60 and 80 degree angles of incidence. The average difference between extraordinary and ordinary reflective indices is about 0.03. The VASE data is analyzed by a Kramer-Kronig consistent parametric semiconductor model which allows non-zero k below the band gap. This work was supported by US Army Research Office.