SYMPOSIUM S

Nondestructive Methods for Materials Characterization

November 29 – 30, 1999

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* Invited paper

SESSION S1: PROCESS CONTROL AND DEFORMATION BEHAVIOR VIA X-RAY TECHNIQUES

Chair: John H. Kinney
Monday Morning, November 29, 1999
Provincetown/Orleans (M)

8:30 AM *S1.1
OPTIMIZATION OF X-RAY TECHNIQUES FOR NON-DESTRUCTIVE CHARACTERIZATION OF SINGLE CRYSTAL TURBINE BLADES. Robert E. Green, Jr., John M. WInter, Jr., Kirsten G. Lipetzky, Center for Nondestructive Evaluation, The Johns Hopkins University, Baltimore, MD.

The efficiency of modern gas turbine engines, used for both airplane 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. Diffusional creep is resisted most optimally by eliminating the grain boundaries, i.e. using single crystal blades. This use of metallic single crystals 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 loss 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 S1.2
IN-SITU OBSERVATION OF OXIDIZATION PROCESS AT THE MOST UPPER SURFACES BY X-RAY SURFACE PROPAGATION WAVES. Toshihisa Horiuchi, Kenji Ishida, Kazumi Matsushige, Dept. of Electronic Science and Engineering, Kyoto Universitu, Kyoto, JAPAN.

Recently, we have found that when white x-rays are irradiating materias with smooth surfaces at a critical angle, there exist x-ray surface propagation waves (XSPWs) 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 occuring at the surfaces, such as an oxidizaion, a corrosion, and a microcrack generation in atomic or nanometer scales. Experiments were carried out with a Mo rorating anode x-ray generator and total reflection x-ray analysis system to monitor the in-situ oxidization processes in various environmental circumstances such as under ultraviolet irradiation and in an acid atmosphere in ppm level for different types of materials; Cu of metal, H-terminated Si and GaAs of semiconductors. The obtained results revealed that the oxidization does not proceed simply, but there exist several different oxidizing processes. In case of Cu, the time variation of the XSPWs at the inirial stage revealed, firstly a slight increase in the electron density, subsequently an abrupt decrease, and then a steady value, suggesting the sealing-off mechanism in the oxidization phenomena. As described above, this method has been proven to be the notable nondestructive method for evaluating the surface conditions, and become as a powerfull tool for studying the relation between the degradation in macro-scale and initial surface changes such as oxidization and corrosions in nano-scale.

9:15 AM S1.3
NONDESTRUCTIVE, THREEDIMENSIONAL IMAGING OF LOW DENSITY MATERIALS. J.H. Kinney, D.L. Haupt, J.D. Lemay, Lawrence Livermore National Laboratory, Livermore, CA.

The mechanical properties of low-density cellular materials are not well understood. Theoretical models 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 extremal 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 1-2mm 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 18μm resolution. A rotating stage with micrometer driven compression allowed imaging of the foams 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 experimental 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-7405-ENG-48.

9:30 AM S1.4
IMAGE-GUIDED FAILURE ASSESSMENT OF POROUS MATERIALS. Michele Tantillo, 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 assessment of fracture progression can not 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 and maintain 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 not found 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 progression. 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 S1.5
X-RAY MICROTOMOGRAPHY OF FATIGUE CRACK CLOSURE AS A FUNCTION OF APPLIED LOAD IN Al-Li 2090 T8E41 SAMPLES. R. Morano, S.R. Stock, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA; G.R. Davis and J.C. Elliott, Department of Biophysics in Relation to 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 Al-Li 2090 T8E41, and early crack face contact during unloading or prolonged contact during loading seems to reduce the driving "force" for crack extention. 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 fatgiue cracks growth in compact tension samples of Al-Li 2090 T8E41 under two different load ratios, and the inter-relationship between crack path, crack face contact, local crack growth rates and loading conditions are discussed.

10:00 AM S1.6
INTERFACIAL DIFFUSION IN A BARIUM TITANATE FILM. A. Datta, A.G. Richter and J. Kmetko, Department of Physics and Astronomy, Northwestern University, IL; S. Chattopadhyay, 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$\AA$ resolution) the composition profile across a 500$\AA$ thick film of $BaTiO_3$ grown epitaxially on (100) MgO by

MOCVD. Results from both studies indicate diffusion of Mg to about 250Å into the film at film-substrate interface, consistent with the diffuse ferroelectric phase transition observed in this film. The lattice parameter a shows a progressive decrease as we move into the film from the interface, and an anomalously low value in the Mg-free portion of the film.

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. Afsaneh Rabiei, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA; T. Kishi, 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 Al_2O_3 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 showed 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. It is proved that the AE signals caused by void nucleation and growth are almost overlapped and started from very low applied strains. Thereafter 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. Shamachary Sathish, 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 standard X- ray diffraction with an average 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 AFRL 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. Matikas, 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.

11:15 AM S2.4
NONDESTRUCTIVE CHARACTERIZATION OF CORROSION PROTECTIVE COATINGS ON ALUMINUM ALLOY SUBSTRATES. Jochen Hoffmann, Mohammad Khobaib, Theodore Matikas, University of Dayton, Center for Materials Diagnostics, Dayton, OH; Norbert Meyendorf, Udo Netzelmann, Fraunhofer Institut, Materials Characterization Dept., Saarbrucken, GERMANY.

Characterization of corrosion protective coatings is of exceptional importance to enhance the lifetime of an aircraft. Coating systems have to resist against chemical, thermal and mechanical loading and the possible failure of the coating leads to insufficient corrosion protection and finally to the failure of the Al-alloy. Determination of mechanical and physical properties of the coating and the condition of the interface, both under different types of load, are required. Furthermore, it is necessary to detect corrosion damage in the aluminum alloy below the coating. These examinations should provide a better understanding of corrosion initiation due to certain coating defects and should help to develop possibilities of corrosion prevention. To achieve this, the employment of high-resolution nondestructive evaluation (NDE) techniques is necessary. The NDE includes Thermal Wave Microscopy, Scanning Acoustic Microscopy and Eddy Current Microscopy. Thus developing a multisensor approach of several NDE techniques provides complimentary information about the different mechanism of coating degradation and corrosion attack. The nondestructive nature of the techniques allows the sample to be retested in order to monitor the initiation of coating defects or the progression of corrosion with time. High spatial and depth resolution is needed to detect microscale defects. This may lead to the ability to examine early changes of the material before they actually develop in severe failures.

11:30 AM S2.5
NONDESTRUCTIVE EVALUATION OF FATIGUE ON TITANIUM ALLOYS. Theodore Matikas, Jerome Frouin, University of Dayton, UDRI-Center for Materials Diagnostics, Dayton; Norbert Meyendorf, Werner Karpen, Henrik Rösner, Udo Netzelmann, Fraunhofer Institute for Nondestructive Testing (IZFP), Dept of Materials Characterization, Saarbruecken, GERMANY.

High cycle fatigue (HCF) of titanium alloys for aircraft components is characterized by changes of the sub-microstructure, for example an increase of dislocation and microvoid density. During more than 90% of the lifetime microcracks do neither appear nor grow significantly, especially in HCF tests. Therefore, nondestructive methods for characterization of early stages of fatigue have to be sensitive to the sub-microstructure variations. Internal friction processes are the main reason for an increase of temperature during fatigue experiments. The temperature enhancement depends on the number of fatigue cycles, the fatigue parameters and finally on the sub-microstructure of the material. With the help of infrared thermography contact-free temperature measurements in dependence of the fatigue damage accumulation in fatigue experiments and in dependence of fatigue parameters were performed. In addition to the mechanical loading in a fatigue machine a high power ultrasound generator was used for the excitation of dissipative processes resulting in temperature effects. These experiments confirm that on titanium alloys a correlation between the damage accumulation and characteristic temperature effects on the surface of components can be found. Thus temperature measurements could be a possible method for characterization of the damage accumulation for titanium alloys. In order to understand the effects of temperature dissipation, positron annihilation measurements were performed on fatigue specimens. This method is based on the measuremnt of positron lifetime depending on the electron density at the position of the positron. Lifetime is influenced when positrons are trapped to crystal lattice defects. Using this method the concentration of crystal lattice defects, e.g. the dislocation density or the concentration of microvoids in the material can be measured. On fatigued specimens a characteristic increase of the positron lifetime caused by lattice could be found.

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 Morgner, Methodologic 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 properies, it has also turned out to be advantageous to discriminate between structure - sensitive and structure-insensitive properties. In developing methods of 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 in the example of nondestructive thickness measurement of hardened layers on the base of backscattered ultrasound, where the well known relationship existing between the depth of hardened layer and the ultrasonic time 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 siutable physical property. The crucial aspect is that of the highest sensitivity, ie, 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 austenitic 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 als well as elastic properties, - the detection of chill zones in cast iron using magnetic distorion factor.

2:00 PM S3.2
MICROSCOPIC TECHNIQUES FOR CHARACTERIZATION OF MAGNETIC LAYERS. Iris Altpeter, Udo Netzelmann, Norbert Meyendorf, Fraunhofer Institute for Nondestructive Testing (IZFP), Dept of Materials Characterization, Saarbruecken; Jochen Hoffmann, UDRI-Center for Materials Diagnostics, Dayton; Wolfgang Nichtl-Pecher, Exabyte Magnetics, Nürnberg; Hubert Grimm, IBM, Mainz, GERMANY.

In recent years there was an increasing interest in magnetic materials and especially amorphous and nanocrystalline layers. Magnetostrictive and magnetoresistive 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 mictoscopic 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 differnt structures. The latter are corresponding to results from Barkhausen noise microscopy, where Barkhausen noise is measured during continous variation of the external magnetizing field. Barkhausen microscopy is applied to evaluate the distribution of magnetic and mechanical properties such as coercitivity, 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 characteristic position dependent anomalies. These give detailed information about remagnetization processes. Eddy current microscopy is sensititve to fluctuations in conductivity, permeability and the dimension and 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 μ and sigma can be calculated.

2:15 PM S3.3
INFRARED EVALUATION OF HEAT GENERATION DURING THE CYCLIC DEFORMATION OF A CELLULAR Al ALLOY. Afsaneh Rabiei, John W. Hutchinson, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA; Anthony G. Evans, Materials Institute, Princeton University, Princeton, NJ.

The heat generated from a notch upon cyclic deformation of a cellular Al alloy has been measured using an infrared imaging system and compared with that calculated using a model of cyclic plasticity occurring within a Dugdale zone. Heating stops when the notch closes, except for a brief period of additional heating because of friction along the notch faces. The model provides a reasonably accurate characterization of the heat generated that reflects the utility of the Dugdale approach. It is found that the temperatures generated are too small to cause fatigue by thermal softening. A fatigue mechanism based on either geometric softening of the cells or crack growth in the cell walls is implied.

2:30 PM S3.4
GIANT MAGNETORESISTANCE IMAGING FOR NDE OF CONDUCTIVE MATERIALS. E.S. Boltz, S.G. Albanna, A.R. Stallings, Y.H. Spooner, J.P. Abeyta, 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 low 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 highly-sensitive technology, substantial efforts have been focused on designs that minimize the effects of environmental noise sources. So called self-nulling configurations have been used to detect and image defects to a depth of nearly 0.5 in aluminum. These sensors have been interfaced to industry-standard aircraft scanning systems to allow rapid image-based inspection of aircraft structures. The fundamentals of magnetoresisitive sensors and their application to eddy-current NDE will be discussed. In addition, sensor image data for aircraft samples, scanned using a commercial aircraft inspection system will be presented. Finally, preliminary data for two-dimensional imaging sensor arrays will be shown.

2:45 PM S3.5
NON-DESTRUCTIVE EVALUATION OF MECHANICAL PROPERTIES OF MAGNETIC MATERIALS. Z. Zang, David X. Yang and Harsh Deep Chopra, Thin Films and Nanosynthesis Laboratory, Mechanical and Aerospace Engineering Dept., State University of New York at Buffalo, Buffalo, NY; G.E. Hicho and L.J. Swartzendruber, NIST, Gaithersburg, MD.

It has long been known that the mechanical and magnetic properties of ferromagnetic materials are closely related. Mechanical properties such as hardness, strength, ductility, and strain-hardening properties are structure-sensitive, and are directly related to the microstructure, composition, and fabrication method. These same parameters control the magnetic characteristics; structure-sensitive magnetic properties include permeability, coercive force, remanence, the Barkhausen spectrum, and the jump-sum rate. Therefore, measurement of these magnetic properties provides a sensitive probe to the mechanical properties, thereby serving as a magnetic based non-destructive method. In the present study, the changes in yield stress due to plastic deformation (up to 4% strain) in ultra-low carbon steel were systematically investigated. These changes were correlated with the aforesaid magnetic properties that form the basis for the non-destructive method. In addition to a brief description of this non-destructive method, results will be presented which shows that a single magnetic parameter could be related to the yield stress. Furthermore, high resolution Interference-Contrast Colloid method was used to understand the microscopic basis for the interpretation of the measured magnetic properties such as the Bartkhausen jumps. A model is developed that takes into account the effect of magneto-mechanical interactions on an otherwise equi-potential minima for the orientation of magnetic domains in strained samples. This model succeeds in explaining the existence of stress-mediated magnetic domains along the applied tensile axis. It also predicts an anisotropy of the measured Barkhausen spectrum and the jump-sum rate, and will be discussed. The authors gratefully acknowledge the 1999-00 American Society for Non-destructive Testing award to SUNY-Buffalo. This work also benefited from support by the National Science Foundation, Grant No. DMR-97-31733, and this support is gratefully acknowledged.

SESSION S4: NDE FOR CONCRETE AND STEEL
Chair: Rick L. Paul
Monday Afternoon, November 29, 1999
Provincetown/Orleans (M)

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

The paper evaluates the possibilities of using the Impact-Echo method for nondestructive examination of concrete structures. 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

particular attention will be given to a further outlook on practical engineering applications.
The "in-situ" investigations were focused on the determination of the thickness of a solid concrete plate of which only one side was accessible. Further voids and delaminations existing in this plate were localized. Finally, the use of the method for checking the quality of injected cable ducts will be discussed.

3:45 PM S4.2
DUAL BAND INFRARED THERMOGRAPHY AS A NDT TOOL FOR THE CHARACTERIZATION OF THE BUILDING MATERIALS AND CONSERVATION PERFORMANCE IN HISTORIC STRUCTURES. Antonia Moropoulou, Nicolas P. Avdelidis, Maria Koui, National Technical University of Athens, Dept of Chemical Engineering, Section of 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 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 25-40°C), provides interpretation tools to the investigation of transport phenomena occurring at the masonry by IR Thermography. The investigation of porous stones consolidated by various consolidants, acting by filmogenic and deposition mechanisms, and of repair mortars, (of various binding materials, at various mix conditions, admixed 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 micro and macro 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 PROMPT GAMMA-RAY ACTIVATION ANALYSIS AND NEUTRON INCOHERENT SCATTERING.
Rick L. Paul, H. Heather Chen-Mayer and Richard M. Lindstrom, Analytical Chemistry Division, National Institute of Standards and Technology, Gaithersburg, MD; Menno Blaauw, Interfacultair Reactor Instituut, 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 nondestructive 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 8.3×10^8 $cm^{-2}s^{-1}$. A total of 10 locations per sample were measured, as the sample was scanned across the beam in 10 mm increments. Prompt gamma-rays from hydrogen, silicon, calcium, 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 mg of H_2O by the sum of all oxides. NIS measurements were performed using a 5 mm diameter neutron beam, with a fluence rate of 2×10^8 $cm^{-2}s^{-1}$. The sample was scanned across the beam at 5 mm increments. Scattered neutrons were measured using a 40 atm ^{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 scattering (H + matrix). 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.

4:15 PM S4.4
NON-DESTRUCTIVE EVALUATION OF MILD STEELS USING A SCANNING HALL-SENSOR MICROSCOPE. D. Sugiyama, Y. Sakai, H. Aoki, Topy Industries Ltd, Toyohashi, JAPAN; K. Miyake, K. Kawano, T. Ito, A. Oota, Toyohashi University of Technology, Toyohashi, Aichi, JAPAN.

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 over conventional techniques such as eddy current, ultrasound and x-ray imaging, etc. We fabricated a scanning Hall-sensor microscope with an active area 50μmx50μm. This microscope permits us to make highly-sensitive and coolant-free measurements at room temperature so that it can be served as a simple and conventional tool for NDE of magnetic materials. Using this, we succeeded in magnetic detection of small cracks($\sim$10 mm long and $\sim$0.1 mm wide) in mild steels with a yield point of 29 kg/mm^2 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 mmϕ, and succeeded in magnetic detection for the positions, shapes and sizes of all the holes in mild steels through drastic changes in magnetic profile.

SESSION S5: LINEAR AND NONLINEAR ULTRASONICS
Chair: Donna C. Hurley
Tuesday Morning, November 30, 1999
Provincetown/Orleans (M)

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 properties of solid, glass and melt 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 glass 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 softening behavior, 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. Hiser, 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 or irradiation embrittlement, has various manifestations: elevation in strength properties, decreases in ductility, and increases in the brittle-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 capsules (wherein 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 steels composing the RPV. A method using measurements of ultrasonic attenuation is under study to provide a means of nondestructively evaluating 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

Charpy V-notch 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 *S5.3
QUANTITATIVE CONTACT SPECTROSCOPY AND IMAGING BY ATOMIC-FORCE ACOUSTIC MICROSCOPY. W. Arnold, Fraunhofer-Institute for Nondestructive Testing, University, Saarbrcken, 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, quantitative determination of Youngs 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 contact spectra will be discussed as well.

9:30 AM S5.4
QUANTITATIVE MEASUREMENT OF LOCAL CARRIER CONCENTRATION OF SEMICONDUCTOR FROM DISPLACEMENT CURRENT-VOLTAGE CURVE USING A SCANNING VIBRATING TIP. Yutaka Majima, Yutaka Oyama, Mitsumasa 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 at which 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_D-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 accumulation indicating that the majority carriers are depleted at the center of the Si surface. The theoretical I_D-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 is able to be determined by making the theoretical I_D-V curve fit the experimental results without contacting the semiconductor surface in a sub-micron order of a lateral resolution under 10 microns of a top radius of the tip.

9:45 AM S5.5
NONLINEAR ULTRASONIC PARAMETER IN PRECIPITATE-HARDENED STEELS. D.C. Hurley, D. Balzar, and P.T. Purtscher, Materials Reliability Division, National Institute of Standards & Technology, Boulder, CO.

Radiation-enhanced formation of ultrafine, copper-rich precipitates can embrittle steel nuclear reactor containment vessels and thus shorten their useful lifetime. As part of a feasibility study of nondestructive methods to evaluate embrittlement, we have performed nonlinear ultrasonic experiments on a precipitate-hardened surrogate material. The specimens were composed of a high-strength, low-alloy steel with 1.13 % 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 β for each specimen. The ultrasonic longitudinal velocity v_L and attenuation $\alpha(\omega)$ were also measured separately in order to correctly calculate β. Microstructural information to interpret our results was obtained by x-ray diffraction line broadening measurements of the average lattice strain ϵ. 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 β and ϵ 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 S5.6
REAL-TIME MONITORING OF ACOUSTIC LINEAR AND NONLINEAR BEHAVIOR OF TITANIUM ALLOYS DURING CYCLING LOADING. Jerome Frouin, Jeong. K. Na, Theodore. E. Matikas, 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 f-2f 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 future 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 proof 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 fracture. This is the first time that such in-situ experiments were performed for measuring in real-time the linear and nonlinear acoustic parameters as a function of fatigue damage. The new methodology and the related new software and instrumentation enable an entirely new way for studying materials under in-service loading conditions. Real-time health monitoring of the material can greatly contribute to the understanding of material behavior under cyclic loading. However, this technique is not limited to fatigue, but it can also be used for studying materials under creep, tensile, 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 S6: ELECTRIC AND OPTOELECTRONIC NDE

Chair: Robert Gilmore
Tuesday Morning, November 30, 1999
Provincetown/Orleans (M)

10:30 AM S6.1
IMPEDANCE SPECTROSCOPY INVESTIGATION ON THE LOW-TEMPERATURE DEGRADATION OF TETRAGONAL ZIRCONIA: 1- INFLUENCE OF THE MEASUREMENT 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.

Impedance spectroscopy (IS) is a powerful tool for evaluating the microstrutural evolution in ionic conductor ceramics. This nondestructive technique has been used for the characterization the low-temperature degradation of yttria-stabilized tetragonal ZrO_2 polycrystals (2.8 mol%). Annealing experiments, in air, were conducted at 250°C and subsequently at 350, 450 and 600C, both with and without the presence of the sputtered silver electrodes. Each experiment had the duration of 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 diffractometry and IS measurements. This latter was performed at frequencies ranging from 5Hz to 13 MHz at 350°C. Superficial transformations resulting from the aging lead to the raising of an extra relaxation in the impedance spectra, which evolves with the degradation magnitude. The electrical characterization of the degradation phenomenon can not be performed 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 condition was 350°C / 4 hours. It permitted a good definition of the semicircles in the impedance diagrams without affecting the degraded surface. Samples annealed at 450 e 600°C showed gradual reductions on the amount of monoclinic zirconia and also on the extra semicircle resistance.

10:45 AM *S6.2
ELECTRICALLY BASED NON-DESTRUCTIVE MICROSTRUCTURAL CHARACTERIZATION OF ALL CLASSES OF MATERIALS. Rosario 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, size and shape of that interface. Eddy currents and dc 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 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) pore volume in thermal barrier coatings, (2) formation of surface layers due to moisture adsorption 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 [vols. 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 S6.3
NON-DESTRUCTIVE DIELECTRIC ASSESSMENT OF WATER PERMEATION IN COMPOSITE STRUCTURES. Richard A. Pethrick, Pascal Boinard, Gordon S. Armstrong, Eric Boinard, Dept of Pure and Applied Chemistry, Strathclyde University, 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 ageing of adhesively bonded aluminium structures, carbon fibre reinforced plastic (CFRP) bonded structures and bulk glass fibre 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 characterisation 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 (TDR) studies 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 frequency and measurement to non-isotropic materials. The approach for frequency domain analysis of bulk GRP using a coaxial probe technique indicates the potential portability of the technique for in-situ measurements.

11:30 AM S6.4
NONDESTRUCTIVE DAMAGE EVALUATION OF ELECTRO-MECHANICAL COMPONENTS USING A HYBRID, COMPUTATIONAL AND EXPERIMENTAL, APPROACH. Cosme Furlong and Ryszard J. Pryputniewicz, CHSLT - Center for Holographic Studies and Laser Micro-MechaTronics, 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 considerations, 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 at a relatively large number of loading cycles. However, large number 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 representative 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 neither only computational nor only 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 accuracies better than 0.1 μm and 0.000,02 %, respectively, and provide indispensable data for testing and development of reliable electronic packages.

SESSION S7: NDE FOR SILICON WAFERS AND INTERFACES
Chair: Matthew W. Stoker
Tuesday Afternoon, November 30, 1999
Provincetown/Orleans (M)

1:30 PM S7.1
CHARACTERIZATION OF NITRIDED SILICON-SILICON DIOXIDE INTERFACES. M.L. Polignano, M. Alessandri, D. Brazzelli, B. Crivelli, G. Ghidini and R. Zonca, ST Microelectronics, Agrate Brianza,MI, ITALY; A.P. Caricato, INFM-MDM, Agrate Brianza, MI, ITALY; M. Bersani, M. Sbetti and L. Vanzetti, ITC-irst, Povo,Trento, ITALY; G.C. Xing, G.E. Miner, N. Astici, S. Kuppurao, D. Lopes, Applied Materials, Santa Clara, CA.

A newly-developed technique for the simultaneous characterization of the oxide-silicon interface 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 conventional capacitance-voltage measurements of surface state density. As no capacitor structure is required, this technique is very suitable for the characterization of as-grown 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 N_2O and in NO were compared to N_2 annealing processes. The effect of nitridation temperature and duration were also studied, and Rapid Thermal Oxidation/Nitridation processes were compared to conventional furnace nitridation processes. 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. NO treatments are much more effective than N_2O 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 a complete device process surface states are annealed out 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 as-processed wafers.

1:45 PM S7.2
IR TOMOGRAPHY OF LIFETIME AND DIFFUSION LENGTH OF CHARGE CARRIERS IN SEMICONDUCTOR SILICON INGOTS. Vladimir D. Akhmetov, Nikolai V. Fateev, Ins Semicond Phys, 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 F 1535-94). 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 F 1535-94). 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 registration of space-time evolution of an injected packet of charge carriers. The packet of injected carriers is generated by the ray of a pumping laser with a wavelength of 1.15-1.25 microns, and monitored by a probing laser beam with the wavelength 3.39 microns, 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, recombination and diffusion are manifested in the evolution of the package of carriers in varying degrees. This allows one to determine both the lifetime and diffusion length of carriers. The laser beams penetrate into the ingot to be characterized either through its ends or through its ends and side wall. Results of measurements on a 250 mm-long model ingot with an artificially introduced sharp lifetime nonuniformity are presented, with the resolution of 5-20 mm achieved. Extrapolation results show that this method can be used to perform tomography of lifetime as well as diffusion length in silicon ingots with the diameter and length up to 300 and 1000 mm, respectively.

2:00 PM S7.3
MONITORING SILICON QUALITY FROM DIFFUSION FURNACES USING IN-LINE MEASUREMENTS. Matthew Stoker, Kelvin Catmull, Motorola, Chandler, AZ; Brian Letherer, Greg Horner, KLA-Tencor, Milpitas, CA.

Carrier lifetimes provide an excellent indication of the concentration of impurities and defects in semiconducting materials. A new non-destructive in-line, non-contact measurement technique is used to measure generation and recombination lifetimes, both near the surface and in the bulk of the silicon. This technique, which is incorporated into the KLA-Tencor Quantox, provides a highly sensitive method for monitoring the formation of crystalline lattice defects in the silicon, incorporation of metallic impurities, and the formation of oxygen precipitates in silicon wafers during processing. Several applications of these measurements are investigated, including monitoring the effects of various high temperature thermal cycles during the formation of a denuded zone, monitoring furnace contamination after a change of quartzware in a diffusion furnace, and measuring the quality of incoming silicon.

2:15 PM S7.4
CHARACTERIZATION OF Ni- AND Ni(Pt)-SILICIDE FORMATION ON NARROW POLYCRYSTALLINE SI LINES BY RAMAN SPECTROSCOPY. P.S. Lee, National University of Singapore, Dept of Materials Science, SINGAPORE; K.L. Pey, National University of Singapore, Dept of Electrical Engineering, SINGAPORE; D. Mangelinck, Institute of Materials Research and Engineering, National University of Singapore, SINGAPORE; J. Ding, National University of Singapore, Dept of Materials Science, SINGAPORE; T. Osipowicz, National University of Singapore, Dept of Physics, SINGAPORE; C.S. Ho, National University of Singapore, Dept of Electrical Engineering, SINGAPORE; L. Chan, Chartered Semiconductor Manufacturing Ltd., SINGAPORE; G.L. Chen, Data Storage Institute, National University of Singapore, SINGAPORE.

Silicides are used in CMOS technology for interconnects and contacts applications because of the low resistivity and thermal stability of these films. As device dimension shrinks, current silicide materials like $TiSi_2$ and $CoSi_2$ are facing certain limitations for future applications. Recently, nickel monosilicide has been proposed to be one of the candidates for salicidation due to its low resistivity, one step annealing process and low silicon consumption. To improve the thermal stability of NiSi, Pt has been added to delay the nucleation of the high resistivity $NiSi_2$ phase at high temperatures as shown by Mangelinck *et. al* [1].
Fast and accurate tools for phase identification on semiconductor device level are essential for understanding process integration issue. Raman spectroscopy is a nondestructive technique that allows local mapping of phases with micron precision. The ability of micro-Raman spectroscopy for in line process monitoring has created much interest for silicides characterization [2].
The purpose of this study was to monitor the thermal stability of nickel monosilicide on narrow polycrystalline Si lines by Raman spectroscopy. The formation of Ni- and Ni(Pt)-silicide on polycrystalline Si lines with linewidths ranging from 5 μm to 0.25 μm were examined using Raman spectroscopy. The presence of nickel monosilicide gave rise to a dominant Raman peak at around 213 cm^{-1}. This was confirmed by X-ray diffraction and Rutherford backscattering analysis. Ni(Pt)Si was found to be present up to a RTA temperature of 900°C on narrow polycrystalline Si lines.
Reference
1. D. Mangelinck, J.Y. Dai, S.K. Lahiri, C.S. Ho, and T. Osipowicz, Mat. Res. Soc. Spring Meeting, (San Francisco, April 5-9, 1999), accepted in Mat. Res. Soc. Symp. Proc.
2. E.H. Lim, G. Karunasiri, S.J. Chua, H. Wong, K.L. Pey and K.H. Lee, *Monitoring of $TiSi_2$ formation on narrow polycrystalline silicon using Raman spectroscopy*, IEEE Electron Device Lett., vol. 19, no. 5, pp171-173, 1998.

2:30 PM S7.5
RAMAN CHARACTERIZATION OF COMPOSITION AND STRAINS IN $Si_{1x}Ge_x$/Si HETEROSTRUCTURES. Ran Liu, Motorola, Embedded Systems Technology Laboratories, Mesa, AZ.

Germanium composition and strains in epitaxial $Si_{1-x}Ge_x$ layer or Si layer are the essential parameters to tailor the electronic bandstructures in $Si_{1-x}Ge_x$/Si heterostructures for various application, such as heterobipolar transistors (HBT), strain-enhanced mobility field effect transistors (SEMFET) and long wavelength optoelectronic devices. In this paper we demonstrate applications of polarized Raman spectroscopy for characterization of germanium composition in the epi $Si_{1-x}Ge_x$ layer, strains and degree of strain relaxation in both epi Si and $Si_{1-x}Ge_x$ layers. Samples used in this study include strained $Si_{1-x}Ge_x$ epilayers on Si and strained Si epilayers on thick relaxed $Si_{1-x}Ge_x$.

2:45 PM S7.6
NONDESTRUCTIVE MEASUREMENT OF IN-PLANE RESIDUAL STRESS IN SILICON STRIPS. Tieyu Zheng, Steven M. Danyluk, The George W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA.

We report on the development of a shadow Moirè technique to measure the in-plane residual stresses of thin, flat plates. This is an extension of prior work on the measurement of in-plane residual stresses in silicon strips and wafers. Phase stepping shadow Moirè and digital image processing techniques were employed to measure the deflections of the silicon plate specimens subjected to three-point-bending at several different loads. The geometry of the silicon plates is 100x25x0.1 mm. The measured deflections over the area of the silicon plates are fitted with an equation represented by a 2-D polynomial series. The fitting coefficients are used in the calculation of in-plane stresses, which are the summation of in-plane elastic stresses caused by bending load and the residual stresses. The theory of thin plates with large deflection is employed to extract the in-plane stresses. Linear regression on the in-plane stresses versus bending loads yields the stress value at zero bending load. The sensitivity of the technique is in the range of 1 MPa and the resolution of measured deflection is 0.003 mm.

3:00 PM S7.7
SMALL SIGNAL ac-SURFACE PHOTOVOLTAGE TECHNIQUE FOR NON-CONTACT MONITORING OF NEAR SURFACE DOPING AND RECOMBINATION-GENERATION LIFETIME IN THE DEPLETION LAYER. Dmitriy Marinskiy, Jacek Lagowski, Marshall Wilson and Alexandre Savtchouk, Semiconductor Diagnostics, Inc, Tampa, FL; Lubek Jastrzebski, Center for Microelectronics Research, University of South Florida, Tampa, FL; Damon K. DeBusk, Cirent Semiconductor, Orlando, FL.

Small signal non-contact ac-SPV methods for monitoring near surface doping (NSD) have been recently been introduced into commercial diagnostic tools. High frequency, short wavelength light with a very short penetration depth (less than 1 micron) is used to generate this small SPV signal that is monitored using a transparent pickup electrode. This technique has the advantage of fast, non-destructive full wafer measurement. Under certain conditions, the magnitude of this ac-SPV signal is inversely proportional to the depletion layer capacitance which allows the calculation of the concentration of ionized donors or acceptors in the depletion layer. The range of this NSD measurement has recently been extended to 1E18 atoms/cm^3, making it a very attractive technique for monitoring low and medium dose implants and especially for wafer scale mapping of uniformity and implant activation efficiency. In addition, the frequency dependence of the SPV signal can be used to monitor generation lifetime in the near surface depletion region of bulk or epitaxial layers. The influence of factors such as incident light intensity, frequency and silicon surface barrier upon this small ac-SPV signal used to monitor NSD are investigated. The influence of surface states upon SPV signal has never been fully understood. This paper will quantify the very important role that surface states play in the monitoring of NSD and generation lifetime on non-oxidized wafers. The effect of various surface treatments such as HF, SC1 and SC1+SC2 wafer cleans will be examined.

SESSION S8: NOVEL TECHNIQUES AND APPLICATIONS
Chair: W. Arnold
Tuesday Afternoon, November 30, 1999
Provincetown/Orleans (M)

3:30 PM S8.1
HIGH-FREQUENCY STROBOSCOPIC X-RAY TOPOGRAPHY UNDER SURFACE ACOUSTIC WAVE EXCITATION. Emil Zolotoyabko, Technion-IIT, Haifa, ISRAEL.

X-ray imaging techniques are under intensive development driven by the progress in third generation synchrotron sources. Nevertheless, in many cases the contrast achieved with x-ray topography is not enough for defect visualization. For example, important post-implantation defects, such as micron size 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. Crossing crystal regions with extended defects, SAW wavefronts are distorted due to scattering processes. Correspondingly, the ruler in the x-ray image becomes bent, facilitating visualization of defects, even if they are not visible without SAW. In order to realize this idea, first we need to freeze the SAW, because time averaging of SAW deformation completely destroys our ruler. This freezing is done using a stroboscopic mode of measurement, i.e. by synchronizing the x-ray flash frequency with the SAW frequency. Synchrotron radiation is ideally suited for stroboscopic measurements because it intrinsically has the required pulse structure. In this paper the abilities of this technique are illustrated by x-ray topographs taken under 300-600 MHz-SAW excitation from $LiNbO_3$-based SAW devices with He-implanted waveguide layers. Experiments were performed at the European Synchrotron Radiation Facility (ESRF) with an x-ray flash frequency of 5.68 MHz. X-ray topographs showed well-resolved individual wavefronts of traveling SAWs as well as their distortions due to the SAW scattering on linear dislocations. Secondary spherical acoustic waves were observed as a result of the strong SAW interaction with He bubbles.

3:45 PM S8.2
THE DESIGN OF POINT-CONTACT PHONOMETER FOR QUANTUM AND RESONANCE HEAT TRANSPORT IN ATOMIC SCALE CONTACTS. Alexandre G. Shkorbatov, Dmitrii B. Kucher.

The realization of nanotermometers makes possible the characterization of transport properties of the point contacts of atomic scale. The contribution of the ballistic phonons emitted 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 description of the low temperature heat transfer anomalies recently measured in point contacts (J.Phys.: Condens. Matter.10 (1998) 8313-8326).

4:00 PM S8.3
IN-SITU SPECTROSCOPIC ELLIPSOMETRY FOR THE REAL TIME PROCESS CONTROL OF PLASMA ETCHING OF SILICON NITRIDE. I.G. Rosen, T. Parent, B. Fidan, and A. Madhukar, 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 increasingly strict process requirements of advanced semiconductor manufacturing. Spectroscopic ellipsometry (SE) is a 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 to 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, non-invasive nature of SE 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 end-pointing in CF_4/O_2 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 films thickness. Both controllers compensate for drifts in etch rate which occur during a given etch. In addition, the ability of the variable gain, adaptive controller to learn from previous etch runs and adaptively adjust for the run-to-run variability inherent to plasma processing is examined. Experimental results which include comparisons between uncontrolled and controlled etches and between adaptive and non-adaptive controllers will be discussed. This work is supported by AFOSR/DARPA under the MURI program.

4:15 PM S8.4
POLARIZED RAMAN SPECTROSCOPY AS A NONDESTRUCTIVE CHARACTERIZATION TOOL FOR INVESTIGATING THE ORIENTATION OF FLUORINATED POLYMERS. Mei-Wei Tsao, 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 fluorine-containing polymers 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, poly-tetrafluoroethylene (PTFE) and polyhexafluoroproprylene (PHFP) samples with highly oriented -CF_2- moieties are measured with different combination of polarizer and analyzer orientations. From these experimental results along with a detailed group symmetry analysis, Raman-active bands associated with different symmetry species can be assigned for various samples. Ratios between Raman bands with A_1 symmetry, located at 733 cm^{-1} and 1381 cm^{-1} respectively, are used for the determination of the degree of orientation in these fluorinated samples. It is found that the I_{733}/I_{1381} ratio varies from as low as 1.5 for a filament oriented parallel to the polarizer / analyser direction, to as high as 12.0 for a filament 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 PM S8.5
PRECISE DETERMINATION OF THIN METAL FILM THICKNESS WITH LASER-INDUCED ACOUSTIC GRATING TECHNIQUE. A.A. Maznev, M. Gostein, M.A. Joffe, R. Sacco, M.J. Banet, Philips Analytical, Boston, MA; K.A. Nelson, M.I.T., Dept. of Chemistry, Cambridge, MA.

Progress in metal interconnect technology is becoming increasingly critical for the semiconductor integrated circuit industry. Increased requirements to thickness uniformity of metal films, rising number of layers, and introduction of new materials call for a fast noncontact 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 angstrom-level repeatability and 1s 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 S9: POSTER SESSION:
NONDESTRUCTIVE METHODS FOR MATERIALS CHARACTERIZATION
Chair: George Baaklini
Tuesday Evening, November 30, 1999
8:00 P.M.
Exhibition Hall D (H)

S9.1
METHOD AND INSTRUMENTATION FOR NONDESTRUCTIVE CHARACTERIZATION OF SURFACE AREA AND PORE SIZE DISTRIBUTION OF THIN-FILMS IN THEIR DEPOSITED STATE. E.S. Boltz, Y.H. Spooner, S.G. Albanna, D.J. Taylor, A.R. Stallings, J.P. Abeyta, Instrumentation and Sensors Department, TPL, Inc., Albuquerque, NM.

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 films up to 3 :m in thickness and has been used to characterize surface features as small as 20 D. 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.

These data, termed isotherms, are then used to determine the surface area and pore size distribution of the thin-film using the BET and BJH models respectively. Descriptions of the method, instrument and data sets for a wide range of coatings will be presented.

S9.2
STRAIN AND TEMPERATURE DISTRIBUTION IN BROAD-AREA HIGH-POWER LASER DIODES UNDER OPERATION DETERMINED BY HIGH RESOLUTION X-RAY DIFFRACTION AND TOPOGRAPHY. Ute Zeimer, Ferdinand-Braun-Institut für Höchstfrequenztechnik, Berlin, GERMANY; Jörg Grenzer, University Potsdam, Institut für Physik, Potsdam, GERMANY; Tilo Baumbach, Daniel Lübbert, Fraunhofer-Institut für Zerstörungsfreie Prüfverfahren, Dresden, GERMANY; Angel Mazuelas, European Synchrotron Radiation Facility, Grenoble, FRANCE; Götz Erbert, Ferdinand-Braun-Institut für Höchstfrequenztechnik, Berlin, GERMANY.

High-power laser diodes with a wavelength of 808 nm and an optical output power > 1W/facet are important as pump sources for solid-state lasers as well as for direct applications like surface hardening and welding. For most applications a good thermal stability of the laser diode is crucial. The study of the thermal behaviour of a mounted laser diode under operation can help to optimize the soldering and mounting technology. This is especially important since high mechanical stress and high thermal load can lead to defect formation causing device failure. A good spatial resolution is desirable for the determination of strain and temperature distribution along the laser stripe to locate the failure sources. Since high-resolution X-ray diffraction (HRXRD) and topography are nondestructive, these methods are well suited for the in-situ study of strain and temperature distribution during device operation. X-rays with a wavelength of 0.06875 nm from a high-brilliance high-intensity synchrotron source allow to penetrate the about 1 μm thick metal layers on top of the p-side-up mounted laser diode. The high-resolution vertical diffractometer at beam-line ID19 of the ESRF in Grenoble, equipped with high-precision motorized slits, was used to ensure a spatial resolution of about 40 x 40 μm2. To achieve a good angular resolution a Si-analyzer was introduced into the diffracted beam path. Thus the angular positions of the GaAs-substrate peak and the AlGaAs-cladding and waveguide layer peaks could be measured with high accuracy. The rocking curves and topographs were taken at the (008)-reflections. The temperature rise near the active layer at different lateral positions along the laser stripe was calculated from the change of the 2Theta-peak positions and the change of the separation between the GaAs- and AlGaAs-peaks. From the change of Omega (angle between incoming beam and sample surface) the change of the radius of curvature was estimated. Since the laser diodes were operated without cooling, at high driving currents the laser operation ceased due to thermal roll-over and defects were formed resulting in an irreversible drop of the output power. At lateral positions where the highest temperature during operation was determined by HRXRD, regions of dark contrasts indicating defects were detected with cathodoluminescence. Thus a clear correlation between temperature rise and defect formation was found. Most defects were located in the middle of the stripe region. No catastrophic optical damage of the mirror facets was observed. Investigations by transmission electron microscopy reveal, that the origin of the dark contrasts are neither dislocations nor dislocation loops, but diffusion of matrix atoms and accumulation of point defects.

S9.3
EXPERIMENTAL SIMULATION OF CONTAMINATION ARISING FROM ELECTRO ROCKET ENGINE-JET ON SURFACE OF SPACECRAFT UNITS. A.A. Chiriv, A.B. Nadiradze, V.V. Shaposhnikov, Moscow Airspace Institute, Moscow, RUSSIA; V.K. Egorov, Lab. Nuclear Res. IPMT RAS, Chernogolovka, Moscow Dist., RUSSIA.

The longevity of spacecraft units and primarily of it's solar batteries is pollution dependent. The electric rocket engines (ERE) of spacecraft are the same assemblies been largely responsible for the pollution of external spacecraft surfaces. The angle distribution of element contamination in xenon jet of ERE (SPD - M70 type) could show the level of it's 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 shown 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 based of BN compound. The variation of the element concentration on a surface of rings as their wear in during of using was analyzed by RBS 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 situated in pairs at different distances from axis of the jet. To prevent the resputtering effect of contamination atoms precipitated on Be substrate they were parked up to Cu films formed by additional Cu-plate pulverizetion by Xe jet. The control Be targets were packed up by Cu too but theirs were screened from the stream to be used as background standards. The RBS and $B(p;\alpha)^8Be$ data were received by using of ion beam complex Sokol-3 IPMT RAS. The investigation shown that the B-contamination is not depend on the angle to jet axis and presents in the jet in content near $1*10^{13}$ ions/m^3. Moreover the jet plasma contains N^+, F^+ and Si^+ ions too. Their concentration in the jet are defined more precisely.

S9.4
OPTICAL CHARACTERIZATION OF RELIALIBILITY IN BARIUM TITANATE CAPACITORS. Michael Biegalski, Susan Trolier-McKinstry, Yoed Tsur, Material Research Laboratory, Pennsylvania State University, University Park, PA.

A major problem with perovskite ferroelectric devices such as multilayer capacitors (MLCs), is the reliability of the dielectric material under DC electric fields. The failure of $BaTiO_3$ 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 $BaTiO_3$ MLCs can be dramatically increased through the addition of some rare earth elements, namely yttrium, holmium, and dysprosium. However, the mechanism through which the rare earth elements increase the reliability of $BaTiO_3$ is not fully understood. To investigate this problem spectroscopic ellipsometry (SE) was used. SE is a nondestructive tool which can monitor the accumulation of damage in $BaTiO_3$ 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 reducing atmospheres were examined. These materials 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.

S9.5
CHARACTERIZATION OF COPPER SURFACES USED IN ELECTRONIC CIRCUIT BOARDS BY REFLECTANCE FT-IR. James M. Sloan and Charles G. Pergantis, 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 co-planarity of circuit boards. Organic solderability preservative coatings (OSPs) are environmentally and economically advantageous over the more commonly used lead based coatings. Problems arise in assessing the solderability of the bare copper and the integrity of the organic coating. Specular 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 manufacting of electronic circuit boards. By measuring the oxide and protective coating characteristics of these surface metallizations, their solderability performance can rapidly be evaluated in a manufacturing environment. OSP coated test specimens were subjected to Hot-Dry and Hot-Wet environmental conditions using MIL-STD-202F as a guide. The resultant FT-IR spectra provides clear evidence for the formation of various Cu oxides at the Cu/OSP interface over exposure time. IR spectral bands consistent with O-Cu-O and Cu_2O_2 formation appear, while minimal deterioration to the OSP coating was observed. The appearance of the Cu oxide layers grew steadily with increased environmental exposure. The IR data can be directly correlated to solderability performance as evaluated by wet balance testing.

S9.6
METHOD OF PARAMETER CONTROL OF OXIDE FILMS ON CRYSTALS SURFACES. P. Kosoboutski,T. Slyusarchuk, State Univ Lviv Polytechnic, Dept of Physics, Lviv, UKRAINE.

In the present paper the method of three-layer spectroscopy of reflection in phonon 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 $\delta + \varphi = \frac{4\pi n d}{\lambda} + \varphi_{23} = 2\pi$ where n is the refraction index of film with thickness d, φ_{23} is the phase shift of the wave reflected from film-crystal bulk interboundary. Analitical dependence of reflection contour minimum frequency on the parameters d and n is determined as a solution of the equation: $\tan\varphi = \frac{\mathrm{Im}\ \hat{r}_{23}}{\mathrm{Re}\ \hat{r}_{23}}$

S9.7
THERMAL PENETRATION TIMES AS A NONDESTRUCTIVE MEASURE OF ORIENTATION IN POLYIMIDE FILM. Nancy Mathis, Mathis Instruments Ltd., Fredericton, NB, CANADA; Robert Samuels, 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 fabricated, and identify any changes that occur over time as a 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 planar. The molecular structure and orientation in the polyimide film was characterized by waveguide coupling techniques, while the thermal penetration time and thermal conductivity measurements utilized a new rapid nondestructive modified hot wire 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 as they relate to orientation. 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.

S9.8
IN SITU SPECTROSCOPIC ELLIPSOMETRY STUDY OF THE OXIDE ETCHING AND SURFACE DAMAGING PROCESSES ON SILICON CARBIDE AND SILICON UNDER HYDROGEN PLASMA. G. Morell, University of Puerto Rico, Dept. of Physical Sciences; I.M. Vargas, J.Y. Manso, 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 SiC and Si substrates under hydrogen plasma conditions. These measurements were complemented with ex situ characterizations 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 materials studied.

S9.9
PHOTOELASTIC IMAGING OF PROCESS INDUCED DEFECTS IN 300mm-SILICON WAFER. H.D. Geiler, JenaWave, Jena, GERMANY; W. Kürner, O. Storbeck, Dresden, GERMANY.

Shrunk 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 processing 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 (SIRD) of laser light transmitting the wafer. The ability of the SIRD equipment to record a full 300mm-wafer stress image in less than 4 minutes with lateral resolution of 100μm allows to monitor crystal defects and their evolution during the technological process sequence. During each process step the high temperature equipment leaves its finger prints 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 defect evolution causes up stream problems like stress induced diffusion of precipitation degrading the device. The specific role of boat marks for thermal defect generation and evolution is analyzed.

S9.10
IRS AND ESR CHARACTERIZATIONS OF NANOCOMPOSITE THIN FILMS WITH FUNCTIONALIZED OR POLYMERIC THIOLATES AND METAL NANOPARTICLES. Wenxia Zheng, Frank L. Leibowitz, Mathew M. Maye, Steven R. DeLaCruz, Yoon A. Kim, David C. Gilbert, David C. Doetschman, Chuan-Jian 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 thiolates 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 molecular interactions and interfacial reactivities. ESR, on the other hand, probes the conduction electron spin resonance properties of the nanosized cores, and the lineshape analysis can be correlated with the electronic microenvironmental effects on the cores in the nanocomposite films. Results of the IRS spectral characteristics, the ESR lineshape features, and their correlation with the nanoparticle core sizes and the organic shell functionality will be discussed.

S9.11
COMPARISON OF TRANSIENT GRATING OPTOACOUSTIC FILM MEASUREMENT (ISTS) WITH OTHER TECHNIQUES FOR SEMICONDUCTOR THIN FILM METROLOGY. Michael Gostein, Alex A. Maznev, Matthew Banet, Philips Analytical, Boston, MA; Todd C. Bailey, SEMATECH, Austin, TX.

A transient grating optoacoustic technique, Impulsive Stimulated Thermal Scattering (ISTS), has been developed recently for non-destructive measurement of the thickness of thin opaque 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 film stacks used in integrated circuit metallization layers. The technique can be used to measure film uniformity and to detect misprocessed films. In this presentation, ISTS will be compared to other opaque 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 4-point probe. 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 next-generation integrated circuit manufacturing.

S9.12
IN-SITU MONITORING OF ALUMINUM NITRIDE GROWTH BY OPTICAL SPECTROSCOPIES. Steven Allen, Patrick Idwasi, Hugh 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 is is nondestructive and all data are collected during growth. Trimethylaluminum, ammonia, and manganese decacarbonyl are used as precursors in the high temperature ($\sim$1100 K) deposition of undoped and Mn-doped AlN performed by MOCVD at vacuum pressure. The radiation emitted by the sample passes through a beamsplitter 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 on the film thickness, orientation, 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 labor-intensive ex-situ techniques.

S9.13
NEUTRON DEPTH PROFILING OF N- AND P-CHANNEL MOS DEVICE WAFERS. S.C. McGuire*, R.R. Vanfleet and J. Silcox, Cornell University, School of Applied and Engineering Physics, Ithaca, NY; G.P. Lamaze and H. Chen-Mayer, 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 our use of neutron depth profiling (NDP) to examine nondestructively the depth distribution of boron in a test wafer containing boron-dope 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 $^{10}B(n,\alpha)^{7}Li$ reaction were observed with an energy dispersive spectrometer. The neutron flux of 2.5x10^{9} /cm^{2}s (thermal equivalent) at the target enabled a rapid assessment of the boron content. Experiment conditions allowed the structures to be probed to a depth of several microns allowing determinations 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 on the order of 10^{16} at./cm^{2} and a layer thickness of approximately 0.5 microns. Details of the measurement technique and method of analysis with results will be described.

S9.14
REAL-TIME OPTICAL CHARACTERIZATION AND CONTROL OF HETEROEPITAXIAL $Ga_{1-x}In_xP$ GROWTH BY P-POLARIZED REFLECTANCE. N. Dietz[a], K. Ito[b], I. Lauko[b] and V. Woods[a]; [a]Departments of Physics and Materials Science and Engineering, [b]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 $Ga_{1-x}In_xP$ 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 utilizes the PR signals to control thickness and composition during heteroepitaxial growth of $Ga_{1-x}In_xP$ 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 increase per time unit, utilizing the monitored PR signal for validation.

S9.15
IN SITU DIFFUSE REFLECTANCE SPECTROSCOPY FOR MEASUREMENT AND CONTROL OF III-V MOLECULAR BEAM EPITAXY. J.E. Guyer, W.F. Tseng, W.R. Thurber, E.M. Vogel, D.A. Gajewski and J.G. Pellegrino, 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 heterolayer 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 sample 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 / InGaAs / 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 upramp following the lower-temperature deposition of the InGaAs layer. This overshoot was eliminated under DRS control. The DRS controlled sample exhibits a strong peak in gate-drain conductance at $\approx$1 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.

S9.16
OPTICAL ANISOTROPIC DIELECTRIC FUNCTIONS OF HEXAGONAL GaN FILM DETERMINED BY VARIABLE ANGLE SPECTROSCOPIC ELLIPSOMETRY. C.H. Yan, H.W. Yao, University of Nebraska, Lincoln, NE; J.M. Van Hove, A.M. Wowchak, P.P. Chow, SVT Associates, Inc., Eden Prairie, MN; J.M. Zavada, European Research Office.

Variable angle spectroscopic ellipsometry (VASE) was employed to study the anisotropic dielectric response, both ordinary and extraordinary, of hexagonal gallium nitride (GaN) thin films o an 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 6.5eV, 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 miximized to a detectable level at large 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 +3%. The VASE data is analyzed by a Kramers-Kronig consistent parametric semiconductor model which allows non-zero k below the band gap. * This work was supported by US Army Research Office.