SYMPOSIUM S
Nondestructive Methods for Materials Characterization

November 29 – 30, 1999

Chairs

George Baaklini
NASA Glenn Research Center
MS 6-1
Cleveland, OH 44135
216-433-6016

Robert Gilmore
General Electric Co
Bldg KWD Rm 253F
Schenectady, NY 12301
518-387-7554

Theodore E. Matikas
6653 Crossbrook Dr
Dayton, OH 45459
937-435-2502

Norbert Meyendorf
Materials Characterization
Fraunhofer IZFP
Saarbruecken, 66123 GERMANY
49-681-9302-3888

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*Invited paper
SESSION SI: 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

The efficiency of modern gas turbine engines, used for both airplane propulsion and electric power generation, increases with reduced 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 to determine the efficiency of the final crystal, both internal and external. Multicrystalline 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 X-ray pleography, 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 OXIDATION PROCESS AT THE MOST UPPER SURFACES BY X-RAY SURFACE PROPAGATION WAVES: Toshinobu Horii, Kenji Ishida, Kazumi Matsushige, Dept. of Electronic Science and Engineering, Kyoto University, Kyoto, JAPAN.

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

9:15 AM S1.3

The mechanical properties of low-density cellular materials are not well understood. Nondestructive techniques have been attempted to describe the elastic behavior of these materials. A well-based approach suggested by classical mechanics attempts to calculate the elastic properties of these materials. However, as the density of the materials increases, the observed elastic properties differ from classical predictions. An understanding of the elastic behavior of these materials is needed to accurately predict their performance. This study was conducted to determine the elastic properties of low-density materials using advanced x-ray imaging techniques. The specimens were fabricated using a compression molding process. The density of the specimens ranged from 0.02 to 0.05 g/cm³. The materials tested included: aluminum foam, silicon foam, and polyurethane foam. The specimens were subjected to three-point bending tests to determine their elastic properties. The results of this study show that the elastic properties of low-density materials vary significantly with density and temperature. This information is important for the design of lightweight structures.

9:45 AM S1.4
X-RAY MICROCTOTOMOGRAPHY OF FATIGUE CRACK CLOSURE AS A FUNCTION OF APPLIED LOAD IN ALI 2090 T34 SAMPLES: R. Mura, R.L. Stock, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA; G.R. Duerinck and C.E. Elliott, Department of Biomedical Engineering and 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 AA 2090 T341, and early crack face contact during unloading or prolonged contact during loading seems to reduce the driving force for crack extension. High resolution x-ray computed tomography (i.e., microtomography) allows one to image the entire volume of samples and to quantify opening as a function of applied load over the entire crack surface. Results are reported for fatigue crack growth in compact tension samples of AA 2090 T341 under two different stress states: quasistatic and dynamic. The results show that crack closure is a function of stress state and that the interaction between crack path and crack closure is complex. In conclusion, the results of this study provide valuable insights into the mechanisms of fatigue crack closure.

10:00 AM S1.5
INTERFACIAL DIFFUSION IN A BARIUM TITANATE FERRITE: A. D. Ante, A. G. Richter and J. Kenkel, Department of Physics and Astronomy, Northwestern University, IL; S. Chatterji, 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 an interfacial reaction in a 50/50 thick film of BaTiO₃ grown epitaxially on (100) MgO by
SESSION S2: NDE FOR FRACTURE FATIGUE AND CORROSION

Chair: Jody I. 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.
Ahmad Rahimi, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA; T. Kashi, and M. Enoji, Research Center for Advanced Science and Technology, The University of Tokyo, Tokyo, JAPAN.

A two channel AE source characterization has been used to evaluate the dynamic process of fracture during three point bending test of Al2O3 particle reinforced composites with 5 and 10 μm of particle sizes. It is observed that the dominant fracture mechanism parameters are void nucleation, growth and coalescence. AE source characterization confirmed that there are two main different sources for acoustic emissions signals in these materials. Void nucleation is classified in one group and void coalescence or micro pop-in between primary voids in another group. In fine particle reinforced materials, when the AE events' amplitude for void nucleation at fine particles is not high enough to be detected, the main source for AE events is only the coalescence of primary micro-voids. By increasing the particle size, the number of detectable events during void nucleation and growth will increase and it is proved that the AE signals caused by void nucleation and growth are almost overlapped and spread 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. Shunsuke Sashihara, 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 Young's 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 in-situ 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. Mitika, 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 fatigued 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 increasingly incremental static load. White light interference microscopy was found to be a successful nondestructive tool for characterizing changes in the deformation zone in front of the crack tip. A relationship between the applied load and the surface area of the deformation zone was obtained. This relationship was exponential directly prior to failure of the specimen. Surface observations during in-situ testing allowed samples to be brought near to failure without complete fracture. This result may aid efforts to optimize the service life of airframe structural components.

SESSION S3: STRUCTURE-SENSITIVE PROPERTIES FOR NDE CHARACTERIZATION

Chair: Norbert Meyendorf
Monday Afternoon, November 29, 1999
Provincetown/Orleans (M)

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

As all NDT methods of materials characterization are based on physical principles, methods to characterize materials in a nondestructive approach are required to be developed systematically in the light of the relationships existing between the material structure and the physical properties. Experience has shown that it is useful to differentiate between macroscopic physical properties and

MOCVD. Results from both studies indicate diffusion of Mg to about 250 Å into the film at film-substrate interface, consistent with the observed ferroelectric phase transition observed in this film. However, the lattice parameter 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.
2:30 PM S3.4 GIANT MAGNETORESISTANCE IMAGING FOR NDE OF CONDUCTIVE MATERIALS. E.S. Biltz, S.G. Aliabadi, A. B. Stallings, Y.H. Spooner, J.P. Akeyta. Instrumentation and Sensors Department, TPL, Inc., Albuquerque, NM.

TPL has developed an electromagnetic sensor suite based on a synergistic coupling of conventional field generation electronics with high performance magnetoresistors. The result is a powerful new sensing technology with broad implications for detection of deeply buried anomalies in conductive materials. With high sensitivity from DC to well over 1 GHz, and a characteristic dimension of only a few microns, these sensors provide performance nearly equaling that of a superconductive quantum interference device (SQUID) but with substantially improved spatial resolution, ruggedness and a much lower fabrication cost. As with any such high-sensitivity technology, substantial efforts have been focused on designs that minimize the effects of environmental noise sources. So-called nulling configurations have been developed to eliminate such effects to a depth of nearly 0.5 in. These sensors have been interfaced to industry-standard aircraft scanning systems to allow rapid image-based inspection of aircraft structures. The fundamentals of magnetoresistive 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. Zhang, 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. Swaim and D. Kish. 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. Some parameters control the magnetic characterization, 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 ultralow carbon steel were systematically investigated. These changes were correlated with the ferromagnetic 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 reduced to the yield stress. Furthermore, high resolution Correlation Coherence method was used to understand the microscopic basis for the interpretation of the measured magnetic properties such as the Barkhausen jumps. A model is developed that takes into account the effect of magnetic mechanical interactions on the otherwise equipotential 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 spectrum will be presented. The authors gratefully acknowledge the 1996-00 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/Oarles (M)

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

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

DUAL INFRARED THERMOGRAPHY AS A NDT TOOL FOR THE CHARACTERIZATION OF THE BUILDING MATERIALS AND CONSERVATION PERFORMANCE IN HISTORIC STRUCTURES. Antonia Melopoulou, Nicolas P. Avdelidis, Maria Kori, National Technical University of Athens, Dept. of Chemical Engineering, Materials Science, and Environmental Engineering, Athens, GREECE; Nikos K. Kazakopoulos, 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 (3.5-4.0 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% and Temperature 20-90°C), provides interpretation tools to the investigation of transport phenomena occurring at the masonry by IR Thermography. The investigation of porous systems conduced by various consolidants, acting by filmogenic and deposition mechanisms, and 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 investigations, which have 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.

CHARACTERIZATION OF HYDROGEN IN CONCRETE BY COLD NEUTRON PERTURBATION 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, Interagency Reactor Institut, Technische Universiteit Delft, THE NETHERLANDS.

A combination of cold neutron prompt gamma-ray neutron activation analysis (PGAA) and 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 x 10^8 s^-1. A total of 10 locations per sample were measured, as the sample was scanned across the beam in 1 mm increments. Prompt gamma-rays were measured using a high-purity germanium detector (HPGe). Potassium and calcium were measured using a high purity germanium detector. Milligrams of each element were determined and converted to milligrams of oxide. The fraction of water at each location was calculated by dividing the mg of H2O by the sum of all oxides. NIS measurements were performed using a 5 mm diameter neutron beam, with a fluence rate of 2 x 10^4 s^-1. The sample was scanned across the beam in 5 mm increments. Scattered neutrons were measured using a 144 cm^3 Ge detector. Of the large incoherent scattering cross section of H, a rapid scan (10 min per point for < 0.5%) was employed to determine the total amount of scattered neutrons. The sample was subsequently baked at 112°C for 35 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.

NON-DESTRUCTIVE EVALUATION OF MILL STEELS USING A SCANNING HALL-SENSOR MICROSCOPE. D. Sugiyama, Y. Sakai, H. Aoki, Togy Industries Ltd., Toyohashi, JAPAN; K. Miyake, K. Kawano, T. Ho, 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. Section of conventional techniques such as eddy current ultrasonic and x-ray imaging, etc. We fabricated a scanning Hall sensor microscope with an active area 50μm x 50μm. This microscope permits us to make high-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 (~10 mm long and ~0.1 mm wide) in mild steels with a yield point of 29 kg/mm² caused by a fatigue process, independent of whether or not the sample surface is covered with nonmagnetic paint. To check further its potential, we applied the microscope to mild steels having circular holes with various diameters from 0.2 to 1.2 mm, and succeeded in magnetic detection for the post-fatigue inducing process and the holes in mild steels through drastic changes in magnetic profile.

SESSION 55. LINEAR AND NONLINEAR ULTRASONICS
Chair: Donna C. Hurley
Tuesday, November 30, 1999
Prowincetown/Olens (M)

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 mel 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 constitution in order to improve the accuracy of modeling of furnaces and melters, and b) improve the understanding of high temperature structures. Results will be presented on a variety of commercial refractory materials and typical glass compositions.

EVALUATION OF ULTRASONIC ATTENUATION MEASUREMENTS FOR ESTIMATING NEUTRON EMBRITTLEMENT OF IRRADIATED RPV STEELS. Allen L. Hieser, 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 assumed. 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 monitoring 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
Chaply 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 SS5.3**

**QUANTITATIVE CONTACT SPECTROSCOPY AND IMAGING BY ATOMIC-FORCE ACOUSTIC MICROSCOPY, W. Arnsdorf, Fraunhofer-Institute for Nondestructive Testing, University, Saarbrücken, GERMANY.**

In Atomic Force Microscopy (AFM) deflection of a microfabricated elastic beam with a sensor tip at its end is used to generate high-resolution images of surfaces. Dynamic modes, where the cantilever is vibrated while the sample surface is scanned, belong to the standard equipment of most commercial instruments. With a variety of these techniques, such as Force Modulation Microscopy, Ultrasonic Force Microscopy, Scanning Local Acceleration Microscopy, or Pulsed Force Microscopy, images can be obtained which depend on the elasticity of the sample surface. However, the absolute determination of Young's modulus of a sample surface with AFM is still a challenge especially when stiff materials such as hard metals or ceramics are encountered. In this contribution the evaluation of the cantilever vibration spectra at ultrasonic frequencies is presented in order to discern local elastic data quantitatively. Nano-crystalline 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 SS5.4**

**QUANTITATIVE MEASUREMENT OF LOCAL CARRIER CONCENTRATION OF SEMICONDUCTOR FROM DISPLACEMENT-FORCED-VOLTAGE CURVE USING A SCANNING VIBRATING TIP, Yu-Taka Majima, Yu-Taka 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 several nm, and also, tunneling currents are measured between the semiconductor surface and the vibrating tip. 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. $I_D$ 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. $I_D$-$V$ 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 majority carriers, 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 obtained by making the theoretical $I_D$-$V$ curve fit the experimental results without contacting the semiconductor surface in a sub-monolayer of a lateral resolution under 10 microns of a top radius of the tip.

**9:45 AM SS5.5**

**NONLINEAR ULTRASONIC PARAMETER IN PRECIPITATE-HARDENED STEELS, D.C. Hurley, D. Bahar, and P.T. Parshall, Materials Reliability Division, National Institute of Standards & Technology, Boulder, CO.**

Radiation-enhanced formation of ultrafine, copper-rich precipitates can embrittle reactor reactor core 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 precipitation-harden 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 $\beta$ for each specimen. The ultrasonic longitudinal velocity $v_L$ and attenuation $\alpha(\omega)$ were also measured separately in order to correctly calculate $\beta$. Microstructure analysis of the specimens was performed by x-ray diffraction line broadening measurements of the average lattice strain $\epsilon$. Measurements showed only small variations in $v_L$ and $\alpha(\omega)$ and no consistent trends with hardness or strain. However, an unusually large tendency between $\beta$ and $\epsilon$ was observed. Our results will be interpreted using a microstructural model for harmonic generation that includes the effect of precipitates. Relationships between lattice strain, state of precipitate coherency, and hardness will be discussed and used to interpret the nonlinear ultrasonic behavior.

**10:00 AM SS5.6**

**REAL-TIME MONITORING OF ACOUSTIC LINEAR AND NONLINEAR BEHAVIOR OF TITANIUM ALLOYS DURING CYCLING LOADING, Jerome Fournier, Jeong K. Na, Theodore G. Moraas, University of Dayton, Center for Materials Diagnostics, OH.**

An in-situ technique to measure sound velocity, ultrasonic attenuation and acoustic nonlinear properties has been developed for characterization and early detection of fatigue damage in aerospace materials. A previous experiment using the Z-scan 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 useful tool to characterize early fatigue damage in the material. For this purpose we have developed a computer software and measurement technique including hardware for the automation of the measurement. New transducer holder and special grips are designed. The automation has allowed us to test the long-term stability of the electronics over a period of time and so the longevity of the linearity of the system. A real-time experiment has been performed on a dog-bone specimen from zero to fatigue life all the way to the final failure. At the 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 approach to nondestructive testing and diagnostics 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 titanium, but it can also be used for other alloys: steel, copper, brass, 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 66: ELECTRIC AND OPTOELECTRONIC NDE**

**Chair: Robert Gilmore**

**Tuesday Morning, November 30, 1999**

**Providence/Olense (M)**

**10:30 AM SS6.1**

**IMPEDANCE SPECTROSCOPY INVESTIGATION ON THE LOW-TEMPERATURE DEGRADATION OF TETRAZIRCONIA, I. INFLUENCE OF THE BI-NODE 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 microstructural evolution in ionic conductor ceramics. This nondestructive technique has been used for investigating the low-temperature degradation of yttria-stabilized tetragonal ZrO$_2$, polycrystals (28 mol\%). Annealing experiments, in air, were conducted at 250°C and subsequently at 350, 450 and 600°C, 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 diffraction 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 cannot 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 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 and 600°C showed gradual reductions on the amount of monoclinic zirconia and also on the extra semicircle resistance.

**10:45 AM SS6.2**

**ELECTRICALLY BASED NON-DESTRUCTIVE MICROSTRUCTURAL CHARACTERIZATION OF ALL CLASSES OF MATERIALS, Pancio A. Gerhard, 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, surface area, and nature of the nonconductive or dielectric layer and surface 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 use 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) pores in dielectric thermal-barrier coatings, (2) formation of surface layers due to moisture absorption in a wide variety of materials, (3) orientation of fibers and whiskers in ceramic matrix and polymer matrix composites, (4) crack detection in metals and (5) stacking faults in semiconductors to name a few. Many more examples can be found in proceedings books of two MRS symposia [Vol. 411 and 500]. While data acquisition can easily be obtained using a variety of commercially available equipment, data interpretation is more complex. This talk will concentrate on the above examples and highlight the most important steps needed in order to obtain useful information from the materials being measured. Proper data collection and data interpretation of a specific set of materials can lead to in-line process monitoring, quality control monitoring, mechanical damage monitoring and environmental degradation monitoring of those materials.

11:15 AM AE.3.3 NON-DESTRUCTIVE DIELECTRIC ASSESSMENT OF WATER PERMEATION IN COMPOSITE STRUCTURES. Richard A. Peddicord, Pascal Boimard, Gordon S. Armstrong, Eric Boimard, Dept of Physics and Astronomy, University of Strathclyde, 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 spring 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 do not only indicate a new way to assess the state and integrity of adhesively bonded structures but are also producing new insight into the application of dielectric spectroscopy and measurement to non-destructive evaluation. The approach of frequency dependent analysis of bulk GFRP using conductive-probe technique indicates the potential portability of the technique for in-situ measurements.

11:30 AM AE.3.4 NONDESTRUCTIVE DAMAGE EVALUATION OF ELECTRO-MECHANICAL COMPONENTS USING A HYBRID, COMPUTATIONAL AND EXPERIMENTAL, APPROACH. Cosme Farlong and Ryszard J. Przybylowicz, CHILIT, Center for Holographic Studies and Ultra Micro-Mechantronics, 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 as a relatively short period of time following the large number of cycles. Characterization 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 electro-optic and mechanical testing data can provide a more accurate and comprehensive description of component damage than either test method alone. The hybrid approach is particularly useful for the non-destructive evaluation of electro-mechanical components subjected to fatigue loading conditions. With the representative application, it is also demonstrated that new and non-conventional experiment 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 non-limited resolution and concurrently with other electro-optic measurement, respectively, and provide indispensable data for testing and development of reliable electronic packages.

SESSION S7: NDE FOR SILICON WAFERS AND INTERFACES
Chair: Matthew W. Skoker
Tuesday, November 30, 1999
Prowestown/Orelens (M)


A newly-developed technique for the simultaneous characterization of the oxide-on-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 N2O and in NO were compared. 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. Nitrided oxides are much less effective than NOx oxides in the formation of a nitrogen-rich interface layer, and, as a consequence, in surface state reduction. Surface state density was measured in fully processed wafers before and after constant current stress. After complete device process surface states are passivated 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. Alkhmetov, Nikolai V. Feskov, Ins Semicon 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 F1355-94). 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 F1355-94). In this report, a novel nondestructive method for measurement of three-dimensional pattern of both the lifetime and diffusion length in silicon ingots, from the above disadvantages, is presented. This method consists in the accumulation of an image of injection 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 3.38 microns, via free-carrier absorption. Depending on
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 Quantex, provides a highly sensitive method for monitoring the formation of crystalline lattice defects in the silicon, incorporating 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 doped zone, monitoring furnace contamination after a change of quartzware in a diffusion furnace, and measuring the quality of incoming silicon.

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 TiSi2 and CoSi2 are facing certain limitations for future applications. Recently, nickel silicide has been proposed to be one of the candidates for silicidation due to its low resistivity, one step annealing process and low silicon consumption. To improve the thermal stability of NiSi, Ni has been added to delay the melt point of the high resistivity NiSi2 phase at high temperatures as shown by Mangelinck et. al [1].

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 NiP 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. NiP(Si) was found to be present up to a HTA temperature of 900°C on narrow polycrystalline Si lines.

Reference:
The paper contains references cited in the text.
many cases the contrast achieved with x-ray topography is not enough for defect visualization. For example, important post-implantation defects, such as micro-cracks or cavities, 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, varies the 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. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images. Consecutive regions with bright and dark lines on the X-ray topographs taken from such an artificially modulated atomic structure contain alternating bright and dark lines passing like a ruler through the images.

4:15 PM S8.4 POLARIZED RAMAN SPECTROSCOPY AS A NONDESTRUCTIVE CHARACTERIZATION TOOL FOR INVESTIGATING THE ORIENTATION OF FLUORINATED POLYMERS. Mei-Wei Yau, John F. Rebelt, 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. We have found that the orientation of defects found in thin films is completely contained 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 polyethylene (PE) samples with highly oriented CF2 moieties are measured with different combination of polarizer and analyzer orientations. From these experimental results along with a detailed group symmetry analysis, Ramen-active bands associated with different symmetry species can be assigned for various samples. Ratios between Raman bands with 𝔇3̅ (1) symmetry, located at 732 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 𝔇3̅(1) ratio varies from as low as 1.5 for a film oriented parallel to the polarizer / analyzer direction, to as high as 12.0 for a film oriented perpendicular to the direction of the polarizing optics. This result strongly suggests that PRS can be applied to material processing environments as a non-destructive method to obtain molecular orientation information.


Progress in metal interconnect technology is becoming increasingly critical for the semiconductor integrated circuit industry. Increased requirements to thickness uniformity of metal films, raising number of layers, and introduction of materials call for a fast, non-destructive measurement technique suitable for line metrology on product wafers. The laser-induced grating technique also called ISTS (Impulsive Stimulated Thermal Scattering) provides metal film thickness measurements at an intermediate repeatability and 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 challenge is achieving the accuracy of current layer thickness 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 9 POSTER SESSION
NONDESTRUCTIVE METHODS FOR MATERIALS CHARACTERIZATION
Chair: George Brodkin
Tuesday Evening, November 30, 1999
8:00 P.M. Exhibition Hall D (H)


TPL, Inc. has developed an instrument for determining the surface area (BET) and pore size distribution (BJH) of thin films in their deposited state. The method is based on a synergistic combination of a surface mode ultrasonic measurement and gas adsorption techniques, and is compatible with most materials and deposition techniques. The technique has been applied to a variety of thin films and has been used to characterize surface features as small as 20 Å. 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 on the film as a function of partial pressure. Data is acquired for both adsorption and desorption cycles.

310
SO. 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 Aerospace Institute, Moscow, RUSSIA;
V.K. Eggov, Lab. Nuclear Res. IPM RAS, Chernogolovka, Moscow
Dist., RUSSIA

The longevity of spacecraft units and primarily of its solar batteries is
pollution dependent. The electric rocket engines (ERE) of spacecraft
are the same assemblies being largely responsible for the pollution of
external spacecraft surfaces. The angle distribution of element
contamination in xenon jet of ERE (SPD-720 type) could show the
level of its responsibility in pollution problem was studied. The
investigation were carried out in conditions of a model vacuum
10⁻⁶ Torr. The calculation of the plasma jet parameters and experimental
measurement showed that the main deliverer of the pollution was the
ceramics of nozzles rings placed in the discharge space. The material
of the rings was created on basis of BN compound. The variation of
the element contamination on a surface of spacecraft units was studied
during using was analyzed by RBS of Bi⁺ ions. The angle contamination
analysis by ERE plasma jet was studied by comparative RBS He⁺
and nuclear reactions diagnostics of the experimental and control
ERE targets were performed at different distances from the
jet. To prevent the rusting effect of contamination atoms precipitated on Be
substrate they were coated up to Cu films formed by additional Cu-plate
pulverization by Xe jet. The control Be targets were coated up by Cu too but
their were taken from the stream to be used as background samples.
The RBS and High resolution X-ray emission data were received by using of ion beam complex Solok-3 IPMT RAS.

The investigation show that the Bi-contamination is not depend on the
angle to jet axis and presents in the jet in content near 10¹⁴
ions/cm². Moreover the jet plasma contains F⁺, Si⁺ and O⁺ ions too.
Their concentration in the jet are defined more precisely.

SO. 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 corrosion resistance of
circuit boards. Organic solubility preservative coatings (OSP) are
environmentally and economically advantageous over the more
commonly used lead based coatings. Problems arise in assessing the
solubility of the bare copper and the integrity of the organic coating.
Spectro reflections Fourier Transform infrared spectroscopy (FT-IR)
has been utilized to monitor the formation of Cu oxides occurring
on these copper substrates used in the manufacturing of
electronic circuit boards. By measuring the oxide and protective
capping characteristics of these surface metallizations, their
solubility performance can be evaluated in an actively used
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 provide clear evidence for the
formation of various Cu/OSP interfaces over exposure time. IR spectral bands consistent with O-Cu-O
and Cu₂O formation appear, while minimal deterioration to the OSP coating was observed. The appearance of the Cu oxide layers grew steadily with increased
environmental exposure. The IR data can be directly correlated to solubility performance as evaluated by wet balance testing.

SO. 6

METHOD OF PARAMETER CONTROL OF OXIDE FILMS ON
CRYSTALS SURFACES. P. Kosobodtchik, T. Shyurskevich, State Univ
Lviv Polytechnical, Dept. of Physics, Lviv, UKRAINE.

In the present paper the method of three-layer spectroscopy of
reflection in photon range of spectrum for nondestructive control
of parameters of oxide films on crystal surface is considered. The essence of
the approach consists in satisfying the condition
\[ a \mu + b \nu = c \rho \]
that defines a reflection index of film with
\[ \theta \]
the phase shift of the wave reflected from film-crystal bulk inter boundary. Analytical dependence of reflection contour minimum frequency
on the parameters \( a \) and \( b \) is determined as a solution of the equation:
\[ \tan \psi = \frac{a \mu + b \nu - c \rho}{1 - a \mu b \nu} \]

SO. 7

THERMAL PENETRATION TIMES AS A NONDESTRUCTIVE
MEASURE OF ORIENTATION IN POLYIMIDE FILM.
Nancy Mathis, Mathis Instruments Ltd., Fredericton, NB, CANADA; Robert Samuel, School of Chemical Engineering, Georgia Institute of
Technology, Atlanta, GA.

Material properties depend on their fabricated structure and

orientation. It is thus important to develop rapid nondestructive techniques that will both allow determination of the orientation of fabricated materials and occur over times that are subsequent in service environmental conditions. The present study examines the relationship between the thermal penetration time and planarity in polyimide films. Polyimides are advanced materials that have good high temperature stability, excellent dimensional stability, and excellent mechanical, electrical, and chemical resistance properties. The samples tested were specially prepared to range in orientation from three dimensionally random to highly plane. The orientation dependence of planarity is demonstrated by waveguide coupling techniques, while the thermal penetration time and thermal conductivity measurements utilized a new rapid nondestructive technique that will be reported here. This correlation will be the first time thermal penetration time has been measured by a modified hot wire technique and related to the internal structure of a polyimide. The work contributes to a deeper theoretical understanding of heat transfer mechanisms relevant to real world applications.

S0 11

COMPARISON OF TRANSIENT GRATING OPTOACOUSTIC FOCUS MEASUREMENT (INSTS) WITH OTHER TECHNIQUES FOR SEMICONDUCTOR THIN FILM METROLOGY. Michael Gruen, Alex A. Mamee, Matthew Bunet, Philips Analitical, Boston, MA; Todd C. Bailey, SEMATECH, Austin, TX.

A transient grating optoacoustic technique, Impulse Simulated Thermal Scattering (ISTs), has been developed recently for nondestructive 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 fabrication. ISTs 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 (SEMTEM), 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.

S0 12

IN SITU MONITORING OF ALUMINUM NITRIDE GROWTH BY OPTICAL SPECTROSCOPY. Steven K. Allen, Patrick Issawi, High Richardson, Ohio Univ, Department of Chemistry and Biochemistry, Athens, OH.

Group III nitrides have been under intense study because of their numerous possible applications in new electronic and optical devices. The use of in-situ spectroscopies is a potentially valuable technique in the study of nitride semiconductor growth as it is nondestructive and all data are collected during growth. Trimethylaluminum, ammonia, and manganese decarboxyl are used as precursors in the high temperature (~1100 K) deposition of undoped and doped AlN performed by MOCVD at vacuum pressure. The radiation emitted by the single passes through a monochromator 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 film deposition rate, 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 light-intensive ex situ techniques.

S0 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. Lannuzze 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-doped N- and P-channel MOS devices. A guided beam of neutrons from the NIST cold neutron source was used to probe the specimen and charged particle products from the 19F(n,γ)19Ne reaction were observed with an energy dispersive 3He detector and a high resolution (2 mrad) spectrometer. The neutron flux of 2.5x10¹⁰/cm²s (thermal equivalent) at the target enabled a rapid assessment of the boron content. Experimental conditions allowed the structures to be probed to a depth of several microns allowing determination to be made on most circuit structures of this type. Monte Carlo simulations of the energy spectra have been obtained that are consistent with a boron layer buried a few microns beneath the surface. Analysis of the energy spectra yielded boron concentrations of the order of 1x10¹⁵ at/cm² and a layer thickness of approximately 0.5 microns. Details of the measurement technique and method of analysis with results will be described.

S0 0

PHOTOLEASTIC IMAGING OF PROCESS INDUCED DEFECTS IN 300mm SILICON WAFER. H.D. Geiger, JenWave, Jenai, GERMANY; W. Kürner, O. Storbeck, Dresden, Germany, GERMANY.

Shrink structural dimensions on enlarged wafer areas increase the influence of crystal defects on device function and integrity. Nondestructive and noncontact measurement techniques for rapid defect monitoring on the product wafer meet the demands of device integrity and cost efficiency. Temperature gradients during high temperature 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 linear 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 100mm 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 detection causes up stress problems like stress induced diffusion of precipitation degrading the device. The specific role of both marks for thermal defect generation and evolution is analyzed.

S0 0


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 characterization of such structural properties for the composite thin films derived from metal nanoparticles and functionalized or polymeric thiols using infrared reflectance spectroscopic (IRS) and electron spin resonance (ESR) techniques. IRS provides information on molecular packing and ordering of the shell components in the nanocomposite films, and the information directly relates to the more molecular interactions and interfacial reactivities. ESR, on the other hand, probes the conduction electron spin resonance properties of the nanomaterial cores, and the lineshape analysis can be correlated with the electronic micromechanical effects in the core-thiol 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.
90.14 REAL-TIME OPTICAL CHARACTERIZATION AND CONTROL OF HETEROEPITAXIAL GaN-InP GROWTH BY P-POLARIZED REFLECTANCE. N. Dietz\textsuperscript{a}, K. Itoh\textsuperscript{b}, I. Lasko\textsuperscript{b} and V. Woods\textsuperscript{b}.\textsuperscript{*} Departments of Physics and Materials Science and Engineering, Center for Research in Scientific Computation, North Carolina State University, Raleigh, NC.

The development of surface-sensitive real-time optical sensors that are able to characterize thin film formation at the point where the growth occurs are essential not only to advance optoelectronic integrated circuits with stringent control of thickness and composition but also to gain a better understanding about the growth process itself. In this contribution we present the application of p-polarized reflectance spectroscopy (PRS) for real-time monitoring and control of pulsed chemical beam epitaxy (PCBE) during low temperature growth of epitaxial GaN-InP heterostructures on Si (100) 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 GaN-InP on Si (100). The decomposition kinetics in the SHF 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.

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

Measurement and control of substrate temperature is a common difficulty for molecular beam epitaxy (MBE) growth of semiconductor structures. Conventional MBE employs a thermocouple (TC) in the vacuum gap between the heater coils and the substrate. In steady-state, the temperature offset between the TC and the substrate surface can be of the order of 100°C, but can be calibrated with pyrometry or by observing known surface changes with reflectance high energy electron diffraction. Even under calibrated conditions, the TC reading can significantly lag the actual substrate surface temperature during transients, resulting in heteroepitaxial 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/AlGaAs pseudomorphic high electron mobility transistors (pHETMs). For one pHETM in each pair, the input signal for substrate temperature control was the TC; for the other, it was the DRS. Under TC control, an overshoot of up to 50°C was observed during the temperature ramp up 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 21 V depletion; this peak is absent in the TC controlled sample. We will discuss the implications of this difference for pHETM growth procedures and for device performance.


Variable angle spectroscopic ellipsometry (VASE) was employed to study the anisotropic dielectric response, both ordinary and extraordinary, of hexagonal gallium nitride (GaN) thin films on important materials for blue and UV light emitting device applications. The GaN films were grown by molecular beam epitaxy (MBE) on c-plane sapphire substrates. Room temperature isotropic and anisotropic mode VASE measurements were made, in the range of 0.75 to 6.5 eV, at the angles of incidence in between 20 and 80 degrees. Evidence of anisotropy is observed via generalized VASE measurements, reflecting the nature of wurtzite crystal structure of GaN. VASE data simulations by isotropic and anisotropic models indicate that the anisotropic effect can be minimized to a negligible level at small angle of incidence, or to be maximized to a detectable level at large angle of incidence. Thus the ordinary dielectric function are precisely determined by the VASE measurements at incident angles of 20 and 80 degrees in the range of 0.75 to 6.5 eV. The film thickness and surface roughness were also determined by the VASE analysis. The extraordinary dielectric functions are then fully determined by the VASE measurements at 60 and 80 degree angles of incidence. The average difference between extraordinary and ordinary reflective indices is about ±0.09. 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.