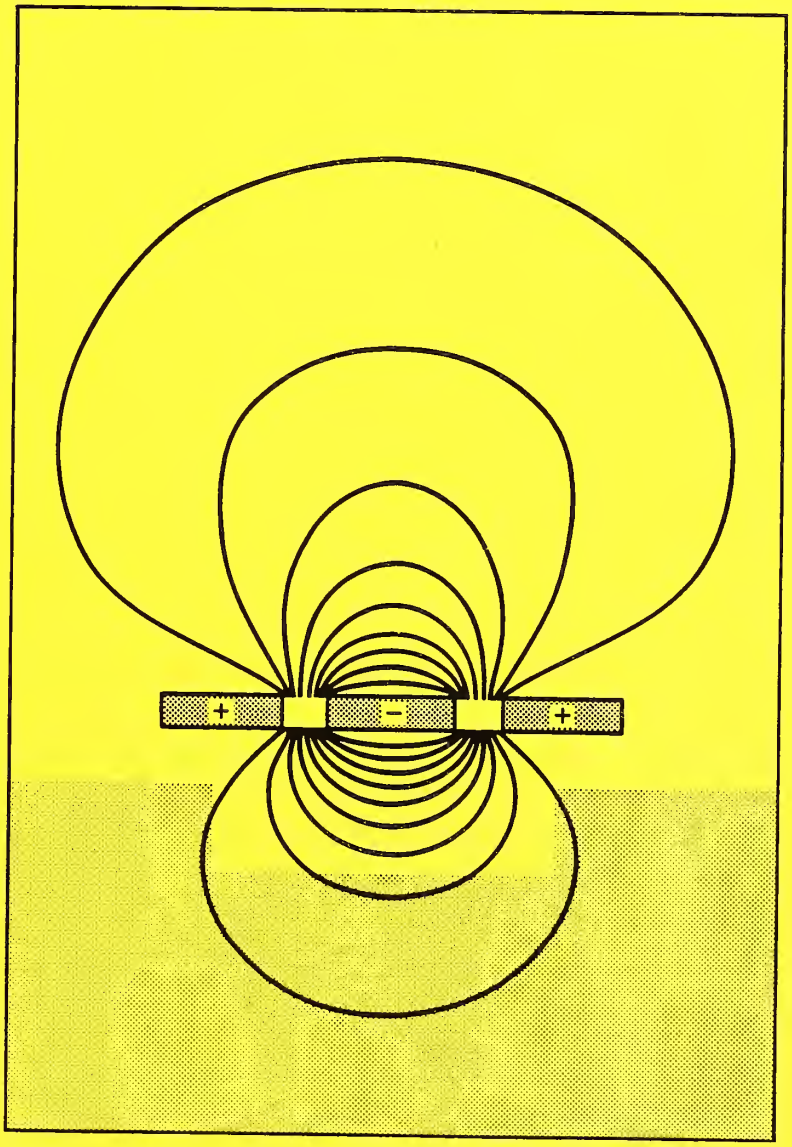
IMSE

Institute for Materials Science and Engineering

NONDESTRUCTIVE EVALUATION



NBSIR 87-3611
U.S. Department of Commerce
National Bureau of Standards

Technical Activities 1987

Schematic diagram of a capacitive array probe for sensing changes in AC impedance due to surface and subsurface features and variations of dielectric properties of insulating materials. The figure shows a three element array over a dielectric containing a groove, i.e., a simulated flaw, and equipotential field lines for this configuration. In practice, more than three elements may be used, not only to determine lift-off error, but also to probe various depths in the dielectric material. See the report, "Capacitive Array Sensor Applied to NDE" by P.J. Shull, J.C. Moulder, M. Gimple, and B.A. Auld.

IMSE

Institute for Materials Science and Engineering

NONDESTRUCTIVE EVALUATION

H. Thomas Yolken, Chief
Leonard Mordfin, Deputy Chief

NBSIR 87-3611
U.S. Department of Commerce
National Bureau of Standards
October 1987

Technical Activities 1987

TECHNICAL ACTIVITIES

Certain commercial equipment, instruments, or materials are identified in this report in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the materials or equipment identified is necessarily the best available for the purpose.

TECHNICAL ACTIVITIES

Table of Contents

IV. NDE STANDARDS AND METHODS

APPENDICES

INTRODUCTION

This report provides brief reviews of technical activities in nondestructive evaluation (NDE) that were carried out by or for the National Bureau of Standards (NBS) in fiscal year 1987 (October 1, 1986, through September 30, 1987). Collectively, these technical activities constitute the Bureau's NDE Program which is managed programmatically and administered on a Bureau-wide basis by the Office of Nondestructive Evaluation (ONDE).

Traditionally, the most common application of NDE is for the characterization of cracks, voids, inclusions, and other kinds of flaws in materials, components, assemblies, and structures. This usage of NDE is the basis of modern in-service inspection procedures as applied, for example, to aircraft, bridges, pipelines, and pressure vessels. Recognizing that NDE measurements for in-service inspection must be reproducible and quantitative, a key component of the NBS NDE Program is providing traceability for NDE measurements to national measurement standards. To this end, NBS conducts research to achieve an adequate understanding of the physical basis of the NDE measurement techniques and procedures that require standardization. Furthermore, the results of the Bureau's research and development work on NDE measurements are applied to specific and meaningful problems in order to demonstrate the validity of the results and to help disseminate them to the user communities. Thus, it is possible to think of the NDE program as comprising three types of activities: research, standardization, and applications.

The most valuable effect of standardization has been to change NDE from a go/no-go system for the detection of flaws to a quantitative system for the characterization of flaws and also for the nondestructive measurement of material properties and characteristics such as microstructural texture, particle size and agglomeration, polymer cure state, density, surface roughness, etc. The capability for reliable and reproducible nondestructive measurements of this kind is vital to the national effort to raise the quality of manufactured products. Clearly, the ability to monitor important material properties or characteristics during a manufacturing process can often be used to guide or control the process and, thus, to raise both the quality and the uniformity of the product. In recent years, the NDE Program has placed strong emphasis on the application of nondestructive measurement principles to the evaluation of material properties and characteristics and on selected demonstrations of the applicability of these measurements to quality enhancement in the processing of advanced materials.

The reviews in this annual report are arranged in the following sections that reflect the NDE Program's four major activity areas of: (1) NDE for Metal and Ceramic Powder Production and Consolidation, (2) NDE for Formability of Metals, (3) NDE for Composites Processing and Interfaces, and (4) NDE Standards and Methods. Each of these sections is preceded by an introduction.

Reports such as this one have been issued on an annual basis since 1978 and are commonly referred to as the "NBS NDE Annual Reports." A parallel series of reports, also issued annually, presents bibliographies and abstracts for the Bureau's technical reports and publications on NDE and its supporting technologies. The purpose of both of these report series is to serve as an introduction to the Bureau's NDE Program. Many readers will want further details on specific aspects of the work or its outputs, and such inquiries are

welcomed and encouraged, both by the principal NDE investigators (whose names and affiliations precede each of the articles in the report) and by ONDE. Either can be addressed in care of NBS, Gaithersburg, Maryland 20899 or reached by telephone via (301) 975-2000, FTS 879-2000, or AV 851-1285 ext. 2000. Requests for further information and suggestions regarding the Program always receive prompt and careful attention.

I. NDE FOR CERAMIC AND METAL POWDER PRODUCTION AND CONSOLIDATION

This activity is broadly concerned with developing approaches, sensors, and procedures for nondestructively determining those properties of ceramic and metal powders and of consolidated materials that relate to the quality and performance of the materials and manufactured parts. The emphasis is on measurements which can be made during the manufacturing process to sense the pertinent properties of the product during critical stages of its formation and to provide the data required to control the process to optimize quality and productivity. This activity includes: characterization of ceramic slips and compacted and sintered ceramics by AC spectroscopy, near-surface characterization of ceramics by thermal waves, determination of the elasticity of ceramic samples by measuring sound velocities, and investigation of appropriate sensors for metal atomization powder systems. Although all of these projects are aimed at developing sensors for the manufacturing process, the project on metal powders represents the most complete approach to this end. It involves the development of a process model, the investigation of a variety of sensors, the study of their performance in an actual inert gas/metal atomization facility, and the development of, and ultimately the use of, an expert system in process control. This project, which is viewed as a model system to obtain experience and insight on how to approach the complete problem of process sensing and control, has been augmented by the formation of an industrial consortium.

A project on small angle neutron scattering (SANS) was completed by achieving its goal of showing that detailed information on particle size and distribution can be determined on undensified $A\ell_2O_3$ powder. A new project on the determination of the complex AC impedance of ceramic slips and compacted and sintered ceramics by AC spectroscopy was initiated. The determination of electrical properties by AC spectroscopy, elastic properties by ultrasonics, and thermal properties by thermal waves are all sensitive to the microstructure of ceramics. Moreover, they have the potential for process sensing.

Representative Accomplishments:

- A project was initiated to investigate the potential of AC spectroscopy in two steps in the processing of ceramics; the preparation of slurries and final sintering. In AC spectroscopy, the complex AC impedance of the material is measured as a function of frequency. Such data can be analyzed to obtain information about the microstructure of the material. Initial measurements on zirconia slurries revealed a surprising relationship between impedance and concentrations of solid material that need to be clarified by further work. Initial complex impedance measurements on fully dense, sintered, zirconia samples clearly showed the effects of the grains and grain boundaries.

- Previous ultrasonic measurements in green-state (unsintered) alumina specimens demonstrated the effect of a naturally occurring flaw, hard agglomerates, on the elasticity of powder and compacted samples. In the work this year, the longitudinal wave speeds in normal alumina samples and in those containing flaws were studied as a function of pressure during complete loading and unloading cycles in a die. The results differed significantly for these two kinds of samples but were repeatable, indicating that such ultrasonic measurements should be useful in monitoring the

compaction process. In order to understand such results on a microscopic level and help in developing a process model, theoretical work was initiated on the influence of pore size and morphology on the elastic moduli of ceramic materials.

- Work was initiated last year on developing sensors for measuring particle size in real time for monitoring metal powder atomization systems. A number of modifications of the atomization chamber was made this year to allow for high-speed Schlieren photography and particle-size measurements. Suitable methods were determined for measuring particle size by laser scattering techniques for diagnostics of the jet break-up and droplet formation. Schlieren photography was used to visualize the pressure fields in the gas jets. A second set of techniques for measuring particle-size distribution at the exit section of the inert gas atomizer has also been evaluated in the atomizer chamber. An industrial consortium consisting of Crucible Materials, Hoeganaes, and General Electric joined with NBS in a three year program to develop an automated processing technology for rapidly solidified metal powders. The program plans involve the development of in situ sensors, process models, an expert computer system, and control devices.

Nondestructive Characterization of Ceramic Processing Using AC Spectroscopy

J. E. Blendell, J. F. Kelly, and A. Dragoo
Ceramics Division
Institute for Materials Science and Engineering

The objective of this work is to study the potential of AC spectroscopy for characterizing the following steps in ceramic processing: (1) slip preparation and (2) final sintering. The complex AC impedance of a sample as a function of frequency is determined, and analysis of these spectra yields information about the microstructure of the material.

(1) The AC electrical characteristics of zirconia slurries were investigated using a Hewlett-Packard 4194A Network Analyzer operated in the gain phase mode. In this mode the instrument has a frequency range of 10 Hz to 100 MHz and measures the ratio of the test channel to the reference channel voltages, T/R, and the relative phase shift, θ, in degrees. Preliminary investigation of the AC characteristics of several slurries composed of zirconia powder and a dispersant were carried out. The effect of solid concentration on the AC response was measured for slips having a range of solid to liquid ratios. T/R values were found to be independent of frequency over the mid-frequency range, from 1 kHz to 10 MHz, whereas θ showed a minimum value at about 30 kHz. To test whether a relationship existed between the electrical response and the slip composition, T/R values at θ minimum were compared with slip composition. A rapid change was found in T/R for concentrations between 0.2 and 0.25 volume fraction solid. The reason for this change has not been determined.

(2) As a ceramic sinters it densifies and the grains coarsen; they increase in size and change shape, and the pore volume and morphology change. These effects can be monitored using AC spectroscopy as the microstructure evolves from the "green" compact, where no grain boundaries exist, to the sintered

sample. Zirconia (ZrO_2) samples with a variety of densities and grain sizes have been prepared. The powders were pressed into pellets with densities estimated to be 40-45% of theoretical, and sintered in air between 1000 °C and 1600 °C for 2-6 hours. The density of the sintered samples ranged from 50% to 100% of the theoretical value. Initial AC spectra were obtained on thin disks of the fully dense ZrO_2. Platinum electrodes were sputtered onto both sides of the sample and the electrical leads were pressed into contact with the electrodes. Measurements were made from 56 Hz to 13 MHz between 350 and 400 °C in air. Figure 1 shows such a spectrum where Z′ and Z′′ are the real and imaginary parts of the complex impedance. The contributions of the grains, grain boundaries, and the electrodes were determined and are identified in the figure. This result indicates that it may be possible to monitor microstructural changes that occur during sintering. The pore size distribution and porosity are not directly determined from these measurements but, rather, must be correlated with other measurements such as ultrasonics, or measured directly.

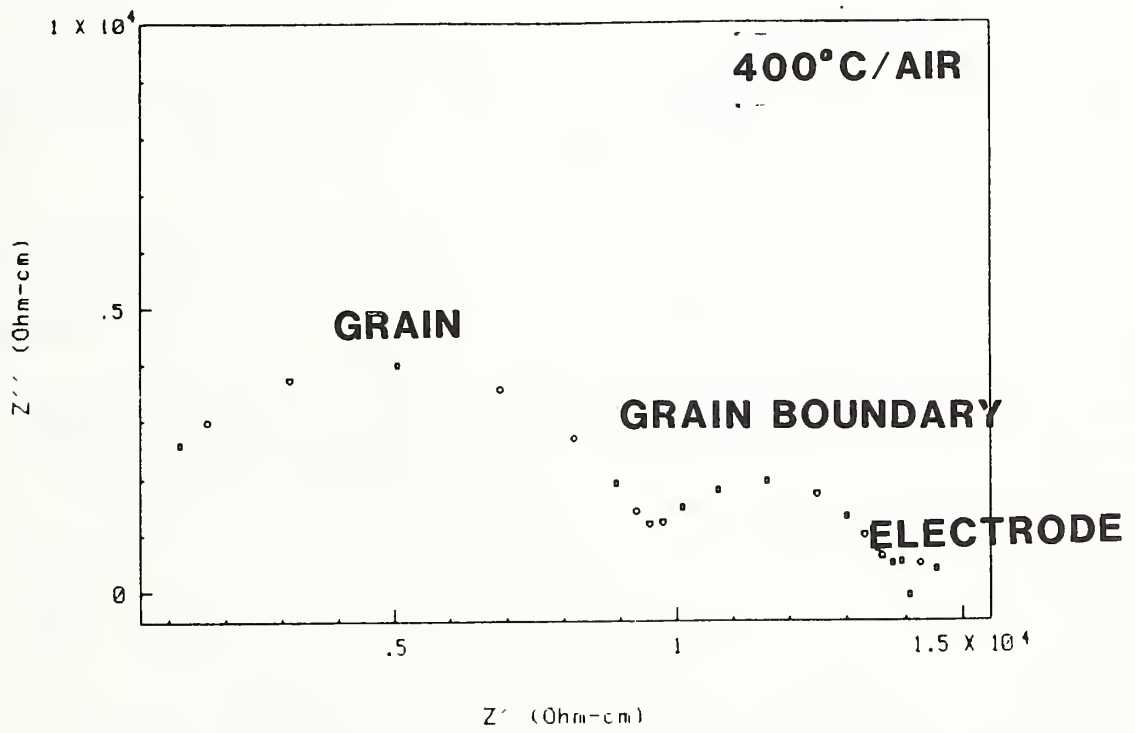


Figure 1. Complex impedance data diagram for fully dense Y-doped ZrO_2 specimen at 400 °C in air.

It was observed that during the above measurements the samples degraded severely, to the point where they had no mechanical strength. This result has been reported to occur only in the temperature range of 100-450 °C in air. At high temperatures the samples show no degradation. The degradation is thought to be due to a reaction of the sample with OH^- present in the atmosphere. However, in order to observe the contribution of the grains, the grain boundaries and the electrodes in the 50 Hz to 100 MHz range, the AC spectra must be obtained between 300-400 °C. To prevent this degradation that occurs in this temperature range, we have constructed a sample holder that allows the atmosphere to be controlled.

During FY88 the AC spectra of samples will be measured over the entire range of densities (from the green compacts to fully sintered) and as a function of grain size at constant density. These data will be correlated with the microstructure of the sample and with ultrasonic measurements.

Near Surface Characterization of Ceramics by Thermal Waves

G. S. White
Ceramics Division
Institute for Materials Science and Engineering

Last year, systems were completed which allow thermal wave experiments to be conducted using either photoacoustic effect (PAE) or mirage effect detection techniques. The mirage system, designed by Dr. Lorretta Inglehart, a scientist on contract from Johns Hopkins University, was adapted for computer control by Eric H. LeGal La Salle, a guest scientist from the ESPCI in Paris, France. This year the work includes: (1) evaluating the effectiveness of the photoacoustic effect (PAE) in detecting porosity changes in alumina, (2) closed crack detection in silica and silicon carbide (SiC), and (3) thermal diffusivity measurements of ceramics.

(1) In previous work, thermal wave signals were shown to vary systematically in specimens fired at different temperatures. However, these variations could not be attributed uniquely to changes in porosity because of possible effects of residual binder on the thermal wave signal, and because the porous specimens lacked the strength to receive the same surface treatment as the dense specimens. New specimens with porosity varying from 3 to 20% were formed by uniaxially pressing powder without binder into wafers and firing the wafers for various times at 1600 °C. This approach eliminated the above problems but resulted in specimens containing layered residual stresses; many of these specimens failed during firing.

The specimens were placed in a PAE cell where the magnitude of the thermal wave signal was found to be: (a) much greater for the most porous specimen than for the other specimens and (b) more sensitive to porosity the shorter the probe length. However, while these and other thermal wave measurements detected changes correlated with large variations in porosity in alumina, the lack of existing theories and the insensitivity of the thermal wave signal to small (~2%) variations in porosity suggest that other NDE methods may be more useful for measuring porosity.

(2) Mirage imaging experiments have been conducted to detect cracks in vitreous silica (SiO_2) and SiC. Cracks were formed in the silica specimens by scribing the surface with a diamond and initiating visible cracks in the damaged area that were longer than the scribe line. Cracks in SiC were formed as a result of machining damage and were visible under the optical microscope. The laser heat source had a focused beam size of ~100 μm. The thermal probe length, a function of the material thermal diffusivity, was 760 μm in the SiC and ~80 μm in the SiO_2. Use of various configurations of the mirage effect apparatus failed to detect the cracks in the silica specimen. In contrast, cracks in the SiC specimen were readily detected for all experimental configurations. A possible explanation of the observed results is that the thermal

probe length in SiO_2 did not extend sufficiently beyond the scanned heating spot to provide a signal detectable over the thermal noise generated by the heating beam. On the other hand, the probe length in the SiC was many times larger than the heating spot size and could detect the crack far from the heating beam position. It is worth noting that the crack widths were much less than the probe length in both of the materials. To deal with materials of small thermal diffusivity such as SiO_2, long thermal wavelengths are required. Further work at lower frequencies would be useful to confirm this suggestion, and these measurements are planned for next year.

(3) There was a cooperative effort this year with AVX, a multilayer capacitor manufacturing company, to employ PAE measurements for determining the thermal diffusivities (α) of a set of dielectric specimens considered for use in capacitors. Preliminary experiments on an alumina specimen showed that α could be determined with an accuracy of 10%. Results showed that changes in processing conditions designed to decrease porosity had no effect on α but that changes in composition, in the 2 to 5% range, could change α from 6×10^{-3} cm^2/s to 12×10^{-3} cm^2/s. These measurements have been successfully used to explain thermal shock failure of some of the dielectric specimens. This work is continuing with a new set of specimens from AVX with different chemical compositions and different processing conditions.

Plans for future work include thermal wave measurements on composites which have undergone mechanical loading to look for the onset of microcracking and delaminations.

References:

1. G. S. White and E. H. LeGal La Salle, "Nondestructive Characterization of Ceramic Materials: Thermal Waves," Proceedings of the NBS-BAM Conference, National Bureau of Standards, 1987.

2. G. S. White, B. Rawal, and C. Nguyen, "Investigation of $BaTiO_3$ Capacitor Materials by Thermal Wave and Ultrasonic Techniques," submitted at the Conference on Nondestructive Testing of High Performance Ceramics, August, 1987.

Ultrasonic Characterization of Ceramic Processing

G. V. Blessing
Automated Production Technology Division
Center for Manufacturing Engineering

E. R. Fuller, Jr.
Ceramics Division
Institute for Materials Science and Engineering

To facilitate the use of ultrasonic probes for on-line monitoring of ceramic processing, the objectives of this work are to develop: (1) an experimental base and (2) predictive models of wave propagation characteristics for both "green" (unfired) and partially sintered ceramics.

(1) Experimental studies this year have investigated the influence of compaction pressure on the dynamic-elasticity properties of ceramics during die compaction. Previous work [1,2] has demonstrated the potential of material sound speed, a dynamic elasticity parameter, for a real time, in situ evaluation of ceramic powder compaction. Here, the influence of various additives, a polyvinyl alcohol (PVA) binder and a glycerol plasticizer, was examined on the compaction process. The addition of additives such as these to a ceramic slurry before spray drying it into powders imparts a thin additive layer on the individual powder particles, which facilitates powder compaction into a dense "green" state. Removal of these additives **before** compaction is not a normal processing procedure, but provides a model "defective" state for studying the compaction of ("hard") agglomerated powders without additives. Additive removal was accomplished here by a calcination step at 500 °C.

Using compaction pressure as a process variable, ultrasonic wave speed was measured during die compaction of an alumina powder, both with additives ("normal") and following the removal of additives ("calcined") [3]. Results during the loading and unloading cycles are shown in Figure 1 for the longitudinal wave speed. As this figure illustrates, wave speed is affected both by sample density (increased pressure giving increased density through particle rearrangement and compaction) and by the extent of particle-particle adhesion (as reflected by normal versus calcined powders). The presence of the binder and plasticizer in the normal sample acts to cohesively bond the particles, resulting in a stiffer material as reflected by the greater sound speed. This stiffness increases significantly upon compaction, with the stiffness of the normal sample increasing slightly more than the calcined sample. Upon unloading, the normal sample retains significantly more of its enhanced stiffness than the calcined sample due to cohesive action of the binder. These observed trends in sound speeds were repeatable, with differences due to like-sample processing being significantly less than those between the normal and calcined samples.

Additional studies were performed on specimens sintered from these powders, both normal and calcined, to determine if acoustic properties could elucidate differences in internal microstructure. Two sintered disks were interrogated using acoustic microscopy techniques [4]. Somewhat surprisingly, at a relatively low ultrasonic frequency of 30 MHz, the normal sample displayed greater internal scattering than the sample from calcined powders. At 100 MHz, however, differences between the two different sample types were overwhelmed by variations within each specimen. A possible explanation of this phenomenon is that the scattering was dominated by interfacial scattering from surface roughness. At 100 MHz, the wavelength of the ultrasonic probe in water is 15 μm, and accordingly is sensitive to average surface roughnesses as small as a fraction of a μm. To illustrate the point further, surface ultrasonic reflection measurements on a similar pair of samples at 4.5 MHz in air (with a greater wavelength of 75 μm) resulted in more than a 50% greater reflected amplitude from the normal sample than from the calcined sample. Future plans are to repeat the 100 MHz tests after polishing both samples to the same surface finish.

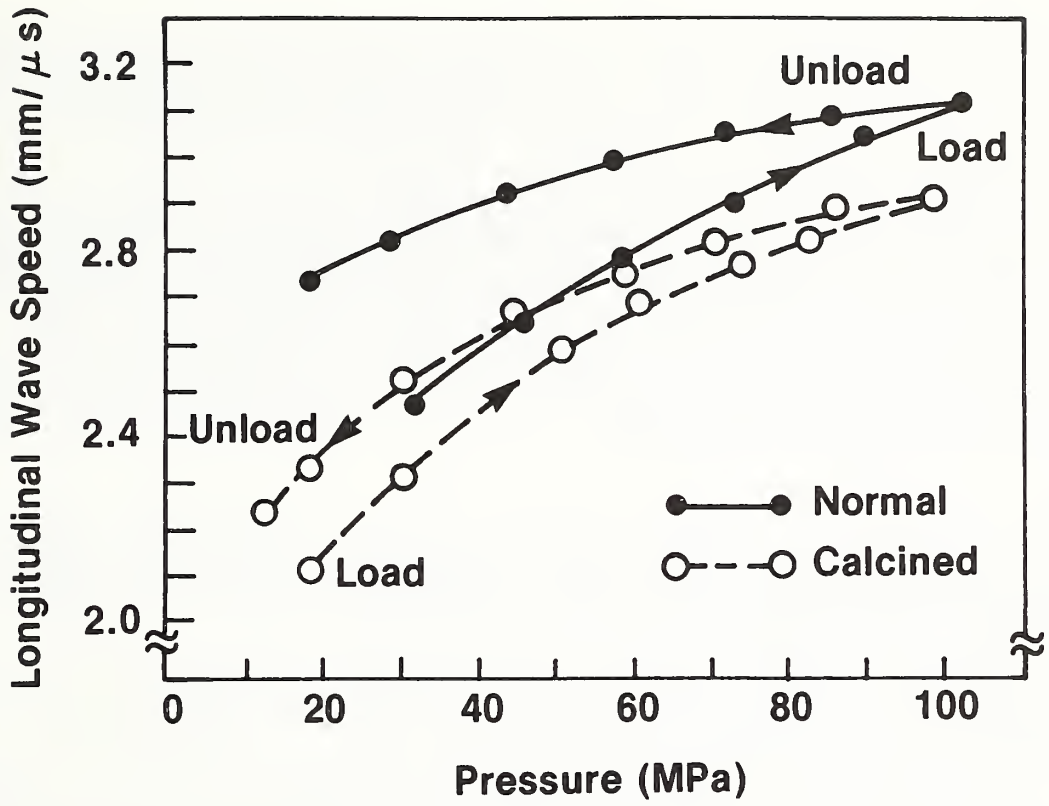


Figure 1. Longitudinal wave speeds in normal alumina powders and in alumina powders with the additives burned out (calcined) are plotted as a function of compaction pressure during loading and unloading.

(2) Theoretical modeling of the elastic properties of various ceramic microstructures was initiated this year to describe the interaction of the ultrasonic waves with the material microstructure that develops during processing. Work was initiated in two areas: (a) consolidation of ceramic powders ("green" ceramics) and (b) densification of ceramics (partially sintered ceramics).

(a) Previous experimental studies [1] have demonstrated the extreme sensitivity of ultrasonic properties of ceramic compacts to processing conditions. For instance, the Poisson's ratio of a ceramic specimen compacted from calcined powder varied from -0.72 to -0.14 to 0.23 in going from a "green" compact to a "partially" sintered compact to a dense ceramic, respectively. This is truly a remarkable range of variation for Poisson's ratio, especially the negative values, which are rarely observed and whose exact meaning is unclear. To understand such results, micromechanical models of the elastic properties of "green" ceramics are being developed using colloid forces to describe the interparticle interactions. Ultrasonic wave propagation in these models is characterized as a function of microstructural morphology and processing conditions. Initial results have demonstrated that noncentral interparticle potentials are necessary to obtain Poisson's ratio other than 0.25.

(b) Well-established procedures are available in the literature to calculate the influence of spherical porosity on elastic moduli of partially sintered ceramics. Applying these procedures to recently measured elastic moduli for partially sintered $Ba_2YCu_3O_7$ superconducting ceramics [5], however, have not given consistent results. For example, the variations of more than a factor of six in the measured bulk moduli before correction persisted after correction by more than a factor of two and a half. Possible explanations for these results might include the morphology and distribution of pores (initial calculations assumed they were monosized and spherical) and the existence of microcracks. Future research will explore the influence of both pore morphology and distribution on elastic moduli; varying the pore shape from microcracks to oblate spheroids to spheres to prolate spheroids, and possibly considering mixed distributions of these shapes.

References:

1. M. P. Jones, G. V. Blessing, and C. R. Robbins, "Dry-Coupled Ultrasonic Elasticity Measurements of Sintered Ceramics and Their Green States," Materials Evaluation 44, pp. 859-862, June 1986.

2. G. V. Blessing and M. P Jones, "Characterization of Ceramics by Ultrasonics," Technical Activities of the Office of Nondestructive Evaluation, NBSIR 86-3434, pp. 7-9, 1986.

3. M. P. Jones and G. V. Blessing, "Ultrasonic Evaluation of Spray-Dried Ceramic Powders During Compaction," Proc. of the Second International Symposium on the Nondestructive Characterization of Materials, Montreal, Canada, 21-23 July 1986 (to be published).

4. Private communication of acoustic microscopy research by L. Kessler, Sonoscan, Bensenville, IL.

5. J. E. Blendell, C. K. Chiang, D. C. Cranmer, S. W. Freiman, E. R. Fuller, Jr., E. Drescher-Krasicka, W. L. Johnson, H. M. Ledbetter, L. H. Bennett, L. J. Swartzendruber, R. B. Marinenko, R. L. Myklebust, D. S. Bright, and D. E. Newbury, "Processing-Property Relations for $Ba_2YCu_3O_{7-x}$ High T_c Superconductors," Adv. Ceram. Mat. 2 [3B], 512-529 (1987).

Sensors for Metal Powder Atomization Systems

S. D. Ridder and F. S. Biancaniello
Metallurgy Division
Institute for Materials Science and Engineering

H. G. Semerjian
Optical Diagnostics Group
Center for Chemical Engineering

G. E. Mattingly
Chemical Process Metrology Division
Center for Chemical Engineering

T. Hopp
Factory Automation Systems Division
Center for Manufacturing Engineering

Many of the current "advanced" metal powder products owe their improved mechanical properties to particular microstructural features obtained by rapid solidification. Previous work in our laboratories has shown that the type of solidification microstructure seen in metal powders is strongly dependent on droplet size prior to solidification [1]. These studies show that droplet undercooling and, therefore, solidification velocity increase as droplet size decreases. It then becomes important to monitor and control the particle size produced during metal atomization in order to obtain particular microstructures that are desired.

The research reported here is aimed at developing an instrumentation system that is capable of monitoring and controlling atomization processes to achieve the desired particle size distribution. This system would operate in real-time, and use the output of sensors in a process feedback loop, based on an expert system, in order to optimize the process. The approach taken to realize this goal is to develop: (1) a fundamental understanding of liquid jet break-up processes that lead to droplet formation, (2) real time sensing techniques for in situ measurement of droplet/particle size distribution and velocity, and (3) expert systems for control of the atomization process using process models developed in task (1) and particle sensing techniques in task (2). This year progress has been made in all three tasks.

(1) Process and Model Development -- The interaction between a series of supersonic gas jets and the molten liquid metal jet leads to liquid jet instabilities and break-up, followed by formation of droplets. Detailed characteristics of the supersonic gas flow are critical in determining the size of the metal droplets formed. A range of flow measurement techniques has been tested and evaluated for determining the salient features of the gas flow in the atomization process. Schlieren photography in conjunction with hot wire anemometry was used to measure the pressure and velocity fields in the gas jets. Figure 1 shows a Schlieren photograph of the gas-only flow field. The classic "bow-tie" shock patterns are visible delineating the gas expansion regions. When the liquid jet is present, the jet pattern is changed because of the interactions downstream of the gas-liquid contact point. Upstream of the contact point in the actual atomization process, the gas flow should closely resemble that occurring in Figure 1.

Hot wire anemometry is also being used with Schlieren photography in the gas-only flow field to measure the velocity and pressure distributions. In this way we are completing our understanding of the physical phenomena occurring in the actual atomization process. These results will indicate both the phenomena and the sensing techniques which will be useful for input to the expert control system being designed to optimize this process. A number of modifications have been made to the atomizer to enable high speed photography of the gas-liquid contact zone. These include four windows equipped with snorkel tubes to allow us to take high resolution, high speed photographs of the disruption phenomena.

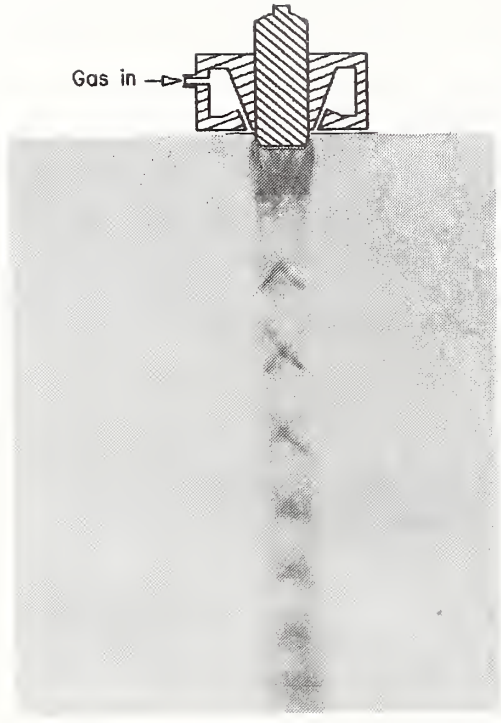


Figure 1. Schlieren photograph with die sketch showing supersonic gas flow (downward) from die operated at a pressure of 9.3 MPa (1350 psig).

In addition, atomization of Sn-5 wt % Sb under various gas and metal flow rates has shown an interesting phenomenon associated with the disruption mechanism involved in this process. For each atomization experiment, the particle distribution displayed distinct, multi-modal characteristics, regardless of operating conditions, as shown in Figure 2. These atomization studies also indicated that a gas:metal mass flow ratio of 4:1 produced the highest yield of sub 40 μm particles. Higher metal flow rates than this resulted in a large number of ill-formed particles due to incomplete atomization [2].

Future activities will include further studies of the gas jets using Schlieren photography, hot wire anemometry, and pitot tube surveys to map the gas velocities in the disruption region for various flow conditions leading to the realistic atomizer conditions. High speed (5-10 ns exposure) photography and holography will be used to determine the disruption mechanism under realistic atomizer conditions.

(2) Sensor Development -- Particle sizing techniques will be utilized for diagnostics of the jet break-up and droplet formation processes in the atomization chamber, by providing detailed measurements of droplet size, number density and velocity. A comparative evaluation of three point measurement techniques has been performed in liquid sprays; these include: a) polarization ratio, b) scattering intensity deconvolution, and c) phase/Doppler techniques. Results indicate that the first two are more sensitive to the smaller particle size range ($d < 5$ μm), whereas the third one provides more reliable measurements for larger droplets ($d > 3$ μm) [3,4]. Tests are now scheduled to perform similar measurements with metal powders.

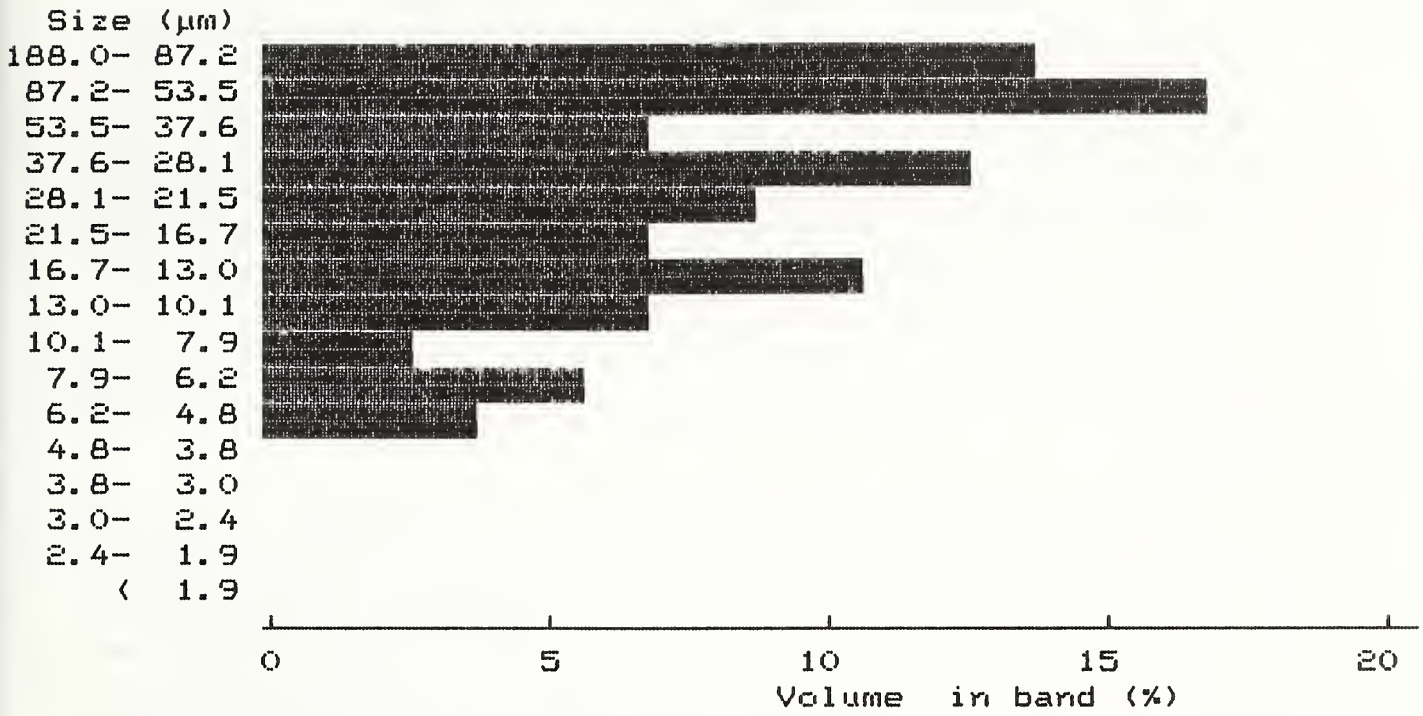


Figure 2. Particle size distribution histogram obtained with the particle size analyzer, indicating the presence of a multimodel distribution.

A second set of instruments, which is more suitable for measurements at the exit section of the inert gas atomizer facility, has also been evaluated. These techniques involve laser diffraction measurements, providing particle size distribution measurements along a line of sight. This particle size distribution, averaged along a cross section of the process stream, can then be used for process feedback and control. Because of the high flow rates and the short period of time (about one minute) available for atomization, it is critical to use a technique with good time resolution. Two instruments were evaluated in laboratory tests with metal powders. It was concluded that an instrument with a multi-element ring diode array could provide more flexibility and much higher data acquisition rates (30 scans/sec repetition rate, 10 msec/reading resolution). This instrument can be used for on-line as well as off-line measurements. A typical set of results obtained with this instrument is shown in Figure 2. This figure shows the corresponding size distribution data in the form of a histogram, which also indicates that even a multimodal size distribution can be resolved by this technique.

The laser diffraction system can be utilized at high particle number densities, up to an obscuration of about 60%. Mie theory calculations indicate that for 5 μm particles, with a density of 10^5 particles/cc, the transmittance should be about 43%. To evaluate this problem under realistic conditions, the exit section of the atomizer chamber was modified to install two windows, and transmission of a laser beam was measured during an atomization run. It was found that transmission decreased to less than 10% within 25 sec after the start of atomization. However, this was found to be primarily due to particle deposition on the windows. The window design is currently being modified to

allow for purging of the window to reduce deposition and obscuration of the laser beam.

During the coming year, installation of the laser diffraction system in the exit section of the atomization chamber will be completed. Tests will also be conducted off-line to allow evaluation of the system capabilities and comparison with other particle sizing techniques. Experiments will also be performed with a laser sheet beam illuminating the spray jet, and high speed cinematography. These tests will provide qualitative data on the general features of the liquid jet and droplets. Two point measurement systems will also be evaluated on a comparative basis to select one which would be utilized for diagnostics in the atomization chamber and to provide data for development of process models for input into expert control systems and for process optimization.

(3) Intelligent Control System Development -- In the area of process control, our work has focused on the design of an expert control system for the atomizer that would incorporate rule-based, heuristic control strategies while still supporting the more traditional process feedback loops currently being used in the system. The atomizing system has been analyzed in terms of the sensory data, decision functions, and process parameters used to control particle production. A preliminary control model has been developed based on this analysis that includes tasks for the various subsystems of the atomizer, including molten metal, pressure chamber, die, and particle size measurement. In addition, tasks were specified for overall atomizer control and for a user interface that would allow interaction with the atomizer and would provide a graphic presentation of the atomizer status and activity. Work has begun on implementing a control system based on the above design. An Amiga computer was chosen for the control system because of its graphics and multi-tasking capabilities. Development has begun on a software environment for the Amiga that allows multiple, rule-based, control processes to communicate between one another. This environment also allows communication between the Amiga and external sensory and control devices and computers.

Future activities will include completion of the Amiga environment, implementation of a rule-based control system running in this environment, and integration of the Amiga-based control system with the existing atomizer system. Presentation graphics will be developed that will display the operational status of the atomizer based on the sensory data and control decisions.

References:

1. S. D. Ridder and D. Shechtman, "Microstructure of Supercooled Submicrometer Aluminum-Copper Alloy Powder," Rapidly Solidified Powder Aluminum Alloys, ASTM STP 890, Philadelphia, 1985.

2. S. D. Ridder and F. S. Biancaniello, "Process Control During High Pressure Atomization," Proc. 6th Int. Conf. on Rapidly Quenched Metals (RQ6), Montreal, Canada, August 3-7, 1987.

3. C. Presser, A. K. Gupta, R. J. Santoro, and H. G. Semerjian, "Laser Diagnostics for Characterization of Fuel Sprays," Proc. 5th Int. Congress on Appl. of Lasers and Electro-Optics (ICALEO 1986), Vol. 58, pp. 160-167, Arlington, VA (1986).

4. C. Presser, A. K. Gupta, H. G. Semerjian, and R. J. Santoro, "Droplet/Air Interaction in a Swirl-Stabilized Spray Flame," Proc. 2nd ASME/JSME Therm. Eng. Joint Conf., Vol. 1, pp. 73-83, ASME, New York, NY (1987).

II. NDE FOR FORMABILITY OF METALS

The goal of this activity in the Nondestructive Evaluation Program is to develop generic approaches, sensors, and procedures for quantitative NDE of metals during the forming process. The emphasis is on measurements that can be made on the production line to improve process control rather than developing inspection techniques for post-manufacturing inspection.

Current efforts in this activity include: the development of NDE temperature sensors based on ultrasonic and eddy current techniques for determining the internal temperature distribution in hot metal objects, developing an in-process ultrasonic monitor for metal grain texture in manufacturing of aluminum products, and utilizing ultrasonics for on-line monitoring of both metal surface roughness and cutting tool wear during machining. Accomplishments in this activity during the past year include:

- During manufacture, the determination of internal temperature distribution in thin extruded aluminum sections for process control has been a troublesome problem for the aluminum industry. In a joint effort between NBS and the Aluminum Association, last year's theoretical and experimental results showed that a multifrequency eddy current measurement approach in the 100 kHz to 1 MHz range appears likely to provide an NDE temperature sensor for process control. This year the eddy current sensor was successfully tested in an aluminum processing plant and the temperature of an aluminum rod was determined.

- The determination of surface roughness during machining of bulk metal parts is desirable for both quality control of the finished product and for control of the cutting or grinding process. An ultrasonic NDE sensor was developed last year for on-line monitoring of surface finish during machining of metal parts. This year's research successfully demonstrated that the same ultrasonic sensor could be utilized as a smart sensor to detect worn or damaged cutting tools on-line. The sensor is noncontacting and utilizes the liquid stream of cutting-cooling solution as a means to couple the ultrasonic signal to the metal.

In-Process Ultrasonic Monitoring of Texture in Manufacturing of Aluminum Product

A. V. Clark
Fracture and Deformation Division
Institute for Materials Science and Engineering

G. V. Blessing
Automated Production Technology Division
Center for Manufacturing Engineering

R. B. Thompson and J. F. Smith
Ames Laboratory
Iowa State University

The presence of texture (preferred orientation) in rolled aluminum alloy and steel sheets has a significant influence on the formability of the material

during processes such as deep-drawing. Texture is created when the rolling process causes a preferred orientation of the single crystals which comprise the material. Improved control of texture during processing is desired and will lead to improved product quality and reduced rejection rates.

Texture can be characterized by the orientation distribution function (ODF), which gives the probability that a single crystal, or grain, will have its axes at a certain solid angle to the rolling and thickness directions of the sheet. The ODF can be expressed mathematically by a generalized series representation; the coefficient multipliers in the series are called the orientation distribution coefficients (ODC). For the symmetries associated with rolling of cubic metals (e.g., aluminum and iron), the formability will be related to only three ODC for moderate amounts of texture.

This research is directed to developing an on-line ultrasonic technique for monitoring texture and is a continuation of research reported here last year. Recently, theories have been developed which model the effect of texture on the velocity of ultrasonic waves. These theories assume that the effective macroscopic elastic moduli (or compliances) can be obtained by averaging the single-crystal moduli (or compliances), suitably weighted by the ODF. For bulk, surface, and guided waves, the theories predict that the velocities are influenced by the same ODC which affect formability. Consequently, the possibility exists that texture can be monitored on-line (during production) by ultrasonic techniques.

To test this hypothesis, specimens of a commercial grade aluminum alloy used as stock for can manufacture have been obtained. The specimens (courtesy of the Aluminum Company of America) were from actual production runs. The ultrasonic velocities of both bulk and guided waves were measured on these plates. Measurements were made at both NBS and at the Ames Laboratory of the Iowa State University. Different transducers and velocity measurement systems were used, so these inter-laboratory measurements could be compared for an internal consistency check both on the measurements and also on the validity of the assumptions made in the mathematical modeling of the effect of texture on ultrasound. For example, the ODC ω_{420} can be obtained by two different methods: (a) difference of velocity of shear waves propagating through sheet thickness with their polarizations parallel and perpendicular to rolling direction and b) difference of velocities of a guided-wave (Lamb-wave) propagating parallel and perpendicular to the rolling direction.

An independent check utilizing a neutron diffraction reference technique was also made to verify the validity of the predictions of the ultrasonic theories. This was done by measuring the ODC with neutron diffraction pole figures obtained with the NBS reactor. Diffraction of neutrons incident on the sheet will occur in certain directions due to texture. This diffraction can be measured and related quantitatively to the ODC.

Ultrasonic measurements of two ODC made at NBS and at Ames Laboratory were in good overall agreement. Furthermore, the neutron diffraction and ultrasonic measurements agreed within 20%. Those comparisons indicate that the mathematical theories accurately model the dominant features of the interaction of ultrasound with a moderately textured material.

Of equal significance from a process control standpoint is establishing a correlation between formability and ultrasonic velocities. This correlation was accomplished by comparing a measure of formability with the ODC. Formability was measured on rolled aluminum sheets obtained from different production runs. As part of the production process, blanks were cut from the rolled sheet and deep-drawn to can shape. Because of texture, some anistropy in plastic deformation occurs, so that some regions of the blank are deformed more than others. This results in "earing"; the top of the can is not circular, but has a lobe structure which must be trimmed off before a lid may be placed on the can. If this earing is excessive, then the appropriate volume of the can may not be obtained. In some cases, this causes rejection of the entire rolled sheet.

A comparison of formability was made with ultrasonic measurement of ODC. One of the ODC displayed a good correlation with formability as shown in Figure 1. This ODC is theoretically related (by formability theory) to the tendency of material to form two "ears" on deep drawing; typically two ears do, in fact, result from drawing the material tested. The other ODC, which relates to the tendency for four ears to form, did not correlate as well. A simple error propagation analysis was done on the data shown in Figure 1. The result is that uncertainty in prediction of formability from ultrasonic measurement is less than the uncertainty in (destructive) earing measurements.

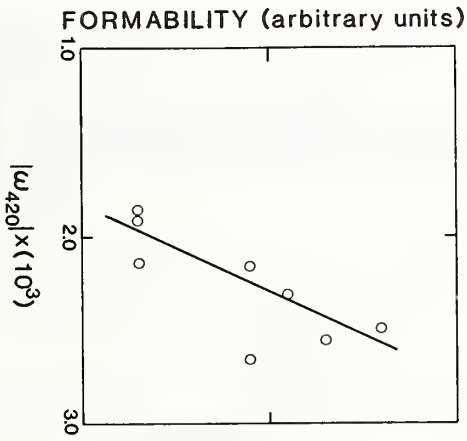


Figure 1. Comparison of orientation distribution coefficient, ω_{420}, and percent earing. Formability values are presented in arbitrary units. Solid line is linear regression fit to data.

All the ultrasonic measurements were done off-line, i.e., in a laboratory environment with the sheet stationary. Possible production application of ultrasonic texture monitoring may require measurements to be made on rapidly moving sheet in a rolling mill. For conventional ultrasonic transducers, the requirement for a medium to couple sound from the transducer to the sheet is a severe limitation. However, there is a class of ultrasonic transducers which is noncontacting (requires no couplant) and can operate at a standoff of typically a few mm from the plate. These devices are called electromagnetic-acoustic transducers or EMATs. NBS is actively engaged in research and development of EMATs for texture application.

The price to be paid for use of EMATs is their relatively inefficient transduction mechanism (typically 60 db below that of a piezoelectric transducer). Overcoming these limitations requires a high-current pulser for the transmitting EMAT and a low-noise high-gain amplifier for the receiving EMAT. An EMAT system has recently been developed at NBS which has a signal/noise (S/N) ratio as large as that typically found with conventional transducers. This system has good tolerance of liftoff; it has S/N greater than 10 for a liftoff of 1 mm and can measure changes in arrival times of ultrasonic waves with a precision of ± 1 ns. With this precision, the velocity measurements can be made with a resolution of about 10 parts per million, which is two orders of magnitude smaller than velocity changes due to texture (about one part per thousand). The system can output data from measurements having this precision approximately every second. Consequently, this type of system shows great promise for technology transfer of texture monitoring from the laboratory to a production line.

References:

1. A. V. Clark, A. Govada, R. B. Thompson, J. F. Smith, G. V. Blessing, P. P. Delsanto and R. B. Mignogna, "The Use of Ultrasonics for Texture Monitoring in Aluminum Alloys," Rev. of Progress in Quant. NDE, D. O. Thompson and D. E. Chimenti, eds., pp. 1515-1524 (Plenum, 1986).

2. R. C. Reno, A. V. Clark, G. V. Blessing, R. J. Fields, A. Govada, R. B. Thompson, P. P. Delsanto, R. B. Mignogna, and J. F. Smith, "Use of Neutron Pole Figures to Calibrate Ultrasonic Techniques for On-Line Texture Control of Aluminum Plates," Proc. of Conf. on Intelligent Processing of Materials and Advanced Sensors II, ed. by H. N. G. Wadley, B. B. Rath, S. M. Wolf and P. A. Parrish, TMS, Oct. 1986.

3. A. V. Clark, G. V. Blessing, R. B. Thompson, and J. F. Smith, "Ultrasonic Methods of Texture Monitoring for Characterization of Rolled Aluminum Sheet," to be published in Rev. of Progress in Quant. NDE (1987).

4. R. C. Reno, R. J. Fields, and A. V. Clark, "Crystallographic Texture in Rolled Aluminum Plates: Neutron Pole Figure Measurements," to be published in Rev. of Progress in Quant. NDE (1987).

5. A. V. Clark, R. C. Reno, A. Govada, R. B. Thompson, J. F. Smith, G. V. Blessing, R. J. Fields, P. P. Delsanto, and R. B. Mignogna, "Texture Monitoring in Aluminum Alloys: A Comparison of Ultrasonic and Neutron Diffraction Measurement," in preparation.

Eddy Current Temperature Sensing

H. N. G. Wadley, A. H. Kahn, and M. L. Mester[1]
Metallurgy Division
Institute for Materials Science and Engineering

[1] Research Associate, Aluminum Association

During manufacture, the determination of internal temperature distribution in thin extruded aluminum sections is an important key to controlling the mechan-

ical properties of the final product. However, the on-line measurement of internal temperature has been a troublesome problem for the aluminum industry. This project, to develop a suitable on-line temperature sensor, is a joint effort between the NBS Metallurgy Division and the Aluminum Association (a consortium of manufacturing and processing companies) [1]. The impetus for its undertaking has been the objective of improved product quality and reduction of rejected output.

Last year we reported that both theoretical and experimental results indicated that a multifrequency eddy current measurement approach in the 100 kHz to 1 MHz range appeared promising. During this year, the design and construction of a prototype model of an eddy current sensor for measuring the diameter, electrical conductivity, and the temperature of aluminum rod during extrusion processing has been completed. A plant demonstration and test of the eddy current temperature sensor was successfully conducted in July 1987.

The envisioned process control system will use acquired eddy current temperature measurements in a feedback loop, controlling the initial temperature of the aluminum billets and controlling the speed of extrusion (itself a heat-generating process). The function of the temperature measurement in the control loop is shown in Figure 1.

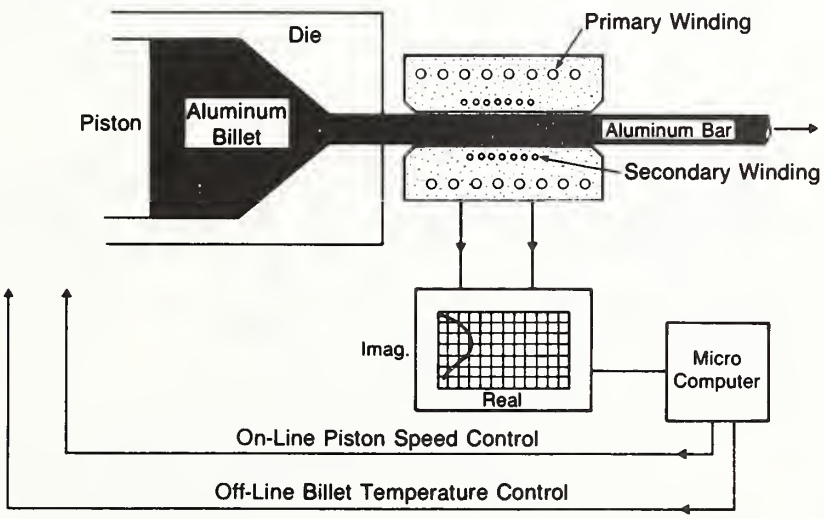


Figure 1. Proposed application of eddy current temperature sensor for controlling extrusion processing.

The prototype model is based on the use of a commercially available impedance/gain-phase analyzer, with control and recording of data performed by a personal computer [2]. Later, an application specific design, requiring less versatility than needed for the development phase, could lead to a smaller, more economical device capable of more rapid measurement. A block diagram of the prototype circuit is shown in Figure 2. It is seen from the figure that the measurement of the properties of the test sample is obtained from the transfer impedance between the primary and secondary circuits. An audio power amplifier is placed in the primary circuit to enhance sensitivity, particularly at low frequencies. From the impedance measurements we obtain

the diameter and conductivity of the aluminum rod. Since the electrical conductivity of the test material is an already known continuously decreasing function of temperature, the impedance measurement provides the required data.

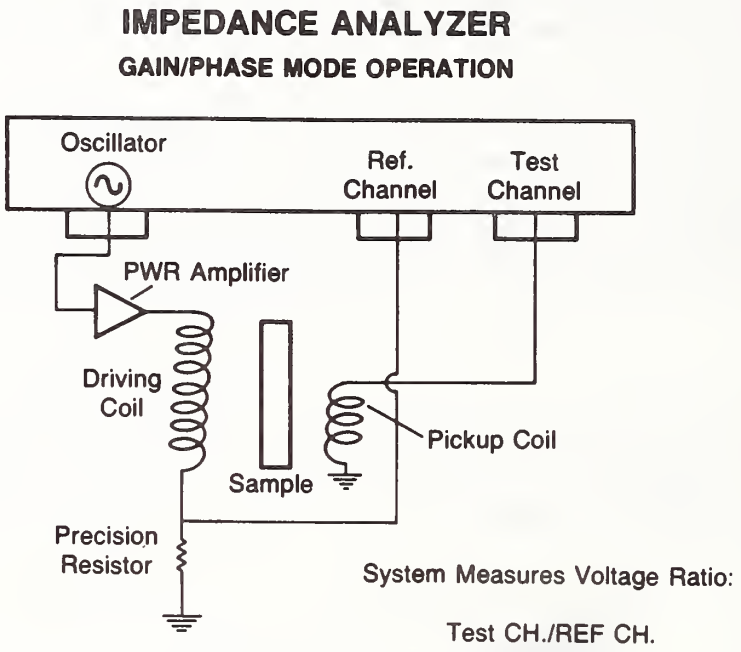


Figure 2. Schematic diagram of sensor system showing primary and secondary coils and power amplifier. Measurement of voltage gain and phase values with test sample present relative to the values for the empty coil allows a determination of the resistivity and cross-sectional area of the test sample.

In laboratory tests, the following measurement accuracies were attained:

Time of measurement: 1 sec
Diameter: +/-0.001" from 0.750" to 1.375"
Conductivity: +/-0.1% IACS from 16% to 22% IACS (+/-0.5% relative error for aluminum at temperatures from 875 to 950 deg F)
Temperature: Above values imply a precision of +/-5.0 deg F

References:

1. A. H. Kahn and H. N. G. Wadley, "Application of Eddy Current Methods to the In-Process Measurement of Temperature Distributions," Proceedings of Aluminum Association Workshop on Sensors, May 13-14, 1986, Atlanta, GA, pp. 129-151 (Aluminum Association, Washington, D.C.).

2. A. H. Kahn and M. L. Mester, "An Eddy Current System for the Measurement of Resistivity and Temperature of Aluminum Rod During Extrusion Processing," Review of Progress in Quantitative NDE, June 21-16, 1987, Williamsburg, VA.

Metal Processing Sensors

H. N. G. Wadley, S. J. Norton, and F. A. Mauer
Metallurgy Division
Institute for Materials Science and Engineering

In operations such as continuous casting, slab reheating and annealing, internal temperature distribution is of fundamental importance. However, no adequate method for measuring this distribution exists. The benefit of an internal temperature sensor has been estimated as equivalent to over $200M annually for the domestic steel industry. The research reported here is aimed at establishing a sensor for determining internal temperature distributions in solid and solidifying bodies. Progress in several areas is discussed below.

The fully automated ten-channel ultrasonic temperature sensor built and reported on last year is shown schematically in Figure 1. Although the figure anticipates the presence of a liquid domain within the body being scanned, evaluation of the system this year was carried out using a 15 cm square block of solid AISI 304 stainless steel. Thermocouples were embedded in the block at the locations shown in Figure 2 to provide an independent record of the temperature distribution. Figure 2 also indicates the location of the two arrays of ultrasonic receivers R1 - R5. The receivers presently in use consist of piezoelectric transducers attached to buffer cones made of AISI 304 stainless steel, but these are to be replaced with noncontacting EMAT receivers (electromagnetic acoustic transducers) now under development.

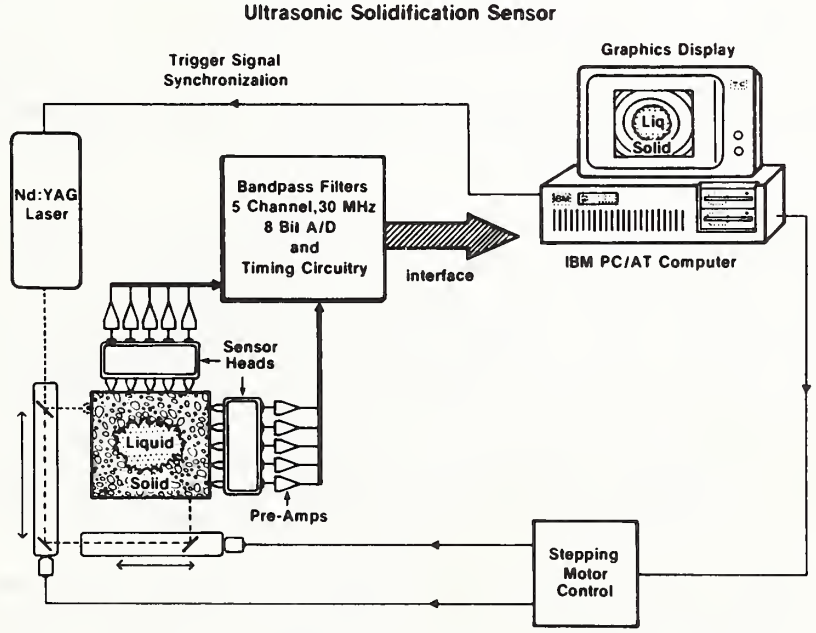


Figure 1. Schematic diagram of the fully automated ten-channel ultrasonic sensor used to obtain the internal temperature distribution in a 6-inch square block of stainless steel.

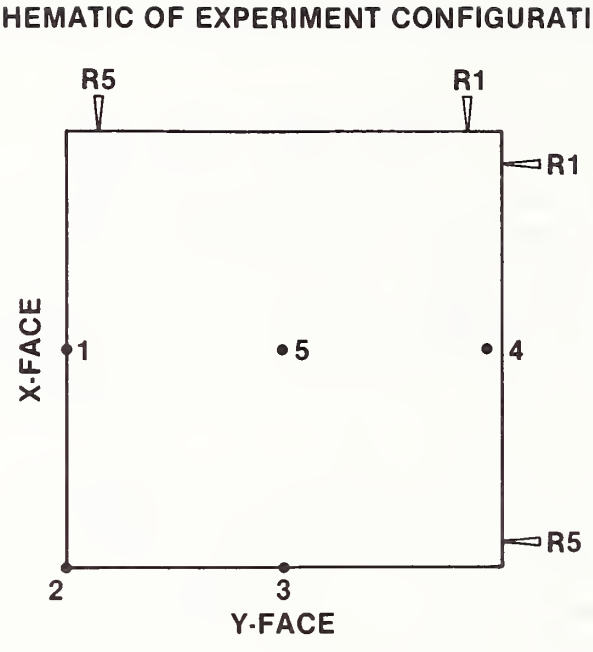


Figure 2. The location of thermocouples and ultrasonic receiver arrays for measuring temperature profiles in the 6-inch square block.

An 800 mJ laser pulse is used as a non-contact generator of ultrasonic waves. As shown in Figure 1, moving mirrors are used to direct the laser beam and generate ultrasonic pulses at points opposite each of the receivers. Thus the time of flight is measured along five equally spaced paths perpendicular to the x face and five perpendicular to the y face. The complete waveform is recorded in each case using transient recorders with a sampling interval of 32 ns.

One of the accomplishments this year was the development of a new method for extracting the time of flight (TOF) from these waveforms using a cross correlation technique. Each high temperature waveform is cross correlated with the one obtained on the corresponding path at room temperature. The maximum value of the cross correlation coefficient occurs at the point corresponding the shift on the time axis. This shift, applied to the room temperature TOF gives the high temperature value, corrected for the delay introduced by the buffer cone and other components. The cross correlation, based on 20 to 40 points, has an averaging effect that improves the signal-to-noise ratio and the accuracy of the TOF data.

A graphics program, also developed this year, displays each waveform, performs the cross correlation, and plots the coefficients. Figure 3 shows the results for a set of waveforms obtained at 720 °C. Plots 1-5 and 6-10 are for waves perpendicular to the x and y faces respectively. The shifts in TOF, multiplied by eight, are shown on the right, where each division corresponds to 32 ns.

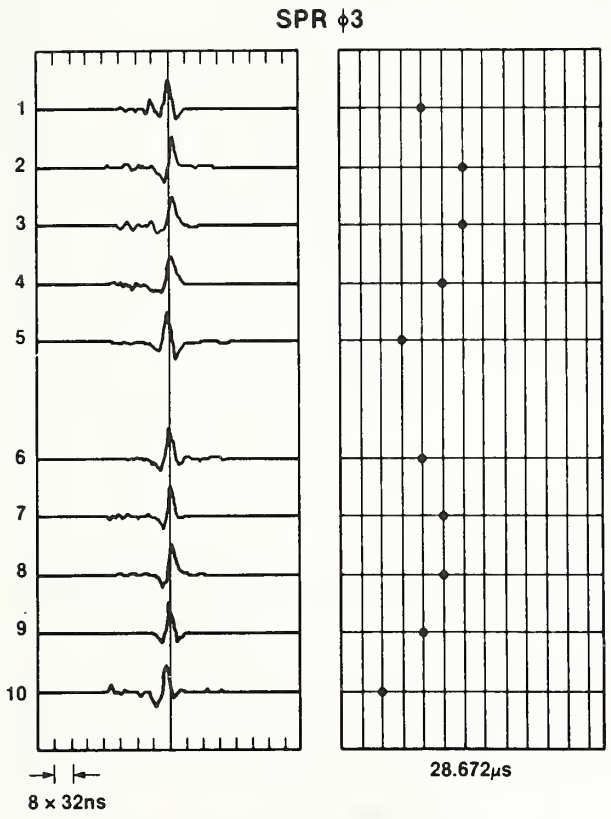


Figure 3. Plot of cross correlation coefficients for waveforms perpendicular to the x face (1-5) and perpendicular to the y face (6-10). The time of flight corresponding to the maximum is plotted on an expanded scale (32 nanoseconds per division) at the right.

To obtain the temperature distribution, the TOF data are compared with values computed from a model based on a priori heat flow information. The parameters of the model are adjusted by a least squares routine to minimize the differences. This routine is now running on the laboratory PC. Using the parameters obtained, temperature profiles are computed from the model for any line in the cross section sampled. We have computed the profile for a section through the block that includes thermocouples 1, 4, and 5 in Figure 2. The measured and computed TOF's, the reconstructed profile, and the thermocouple readings are shown in Figure 4. The center temperatures agree exactly. There is a 15 °C discrepancy at the surface of the block which may in part be due to difficulty in getting accurate thermocouple readings of surface temperature.

Application of the tomographic approach to solidifying bodies, as depicted in Figure 1, has been delayed because of difficulty in producing a body having a liquid center. Presently, molten aluminum (1100 series) is poured into a preheated container of the same material and TOF measurements are carried out during solidification. Problems have been encountered in getting the liquid to wet the solid and form a continuous path across the interface. We have formulated a new design for containment of the molten material which should overcome these difficulties.

In preparation for eventual field tests of the ultrasonic temperature sensor in a plant environment, the American Iron and Steel Institute has arranged for access to a horizontal strand caster at the ARMCO plant in Baltimore.

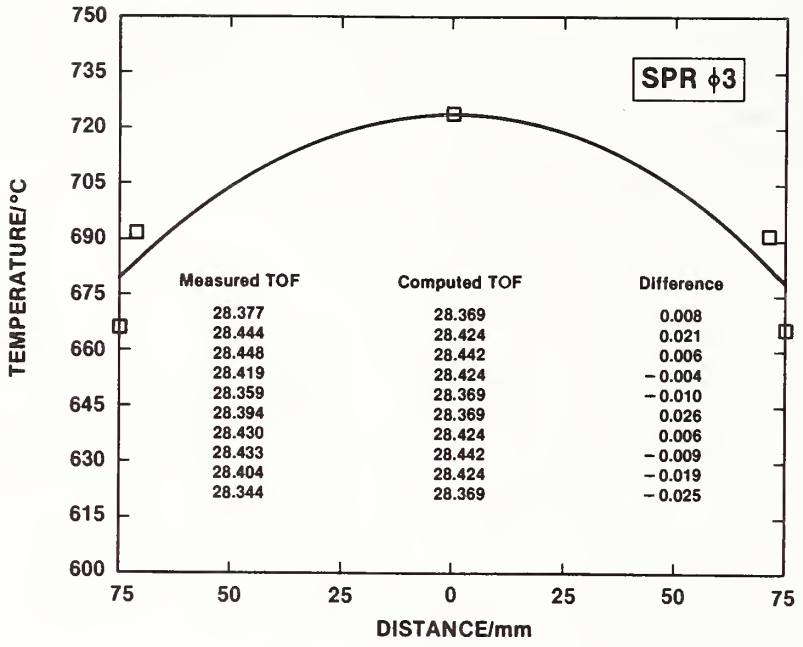


Figure 4. Reconstructed temperature profile along a line through the block that includes thermocouples 1, 5, and 4.

Dr. James Cook and Dr. Bernie Droney, who are NBS Research Associates, carried out tests at this site in April 1987, on a hot strand of stainless steel, to demonstrate the feasibility of making measurements under operating conditions. NBS ultrasonic systems were subsequently used in some of these tests. Sections of two strands were brought to NBS for characterization by metallographic and ultrasonic techniques. The field tests will eventually involve collaboration with Battelle Pacific Northwest Laboratories and a manufacturer of EMAT receivers.

Ultrasonic Surface Characterization

G. V. Blessing and D. G. Eitzen
Automated Production Technology Division
Center for Manufacturing Engineering

The measurement of surface topography is important in many areas of part manufacturing, especially for the finished product. In order to provide improved NDE sensing capability, we have been applying ultrasonic scattering and reflection techniques to the evaluation of both average surface roughness and detailed surface profilometry on parts in situ during the manufacturing process [1].

Last year we studied the application of an ultrasonic sensor to rapidly rotating cylindrical parts with varying surface roughnesses. The purpose was to simulate the application of monitoring a part surface during cutting in a turning center. This year we studied the use of an ultrasonic sensor scanning stationary parts in a milling center.

Of special interest to the manufacturing community is the ability to detect worn or damaged tools on-line with a smart sensor scheme. Examples of the application of an ultrasonic sensor system [2], illustrated in Figure 1, are given here. Pulses of ultrasonic waves (MHz frequencies) are coupled to the part surface using the machine center cutting fluid which is pumped to a chamber housing the ultrasonic transducer, and then through attached tubing to form a liquid pipe acting as a waveguide for the ultrasound.

A one-half inch diameter end mill with a chipped radial cutting edge barely visible to the naked eye was used to side-mill a one inch thick 4150 steel plate. The contrasting ultrasonic signatures created by a new and the chipped cutting tool are shown respectively in the left and right hand portions of Figure 2, generated by the ultrasonic sensor scanning across the side-milled part face. Both the mean value and the variation σ (standard deviation) of the reflected echo amplitude of the 6 MHz ultrasound were sensitive to the part surface affected by the damaged tool. The average surface roughness values measured with a contacting profilometer were respectively 1 and 7 μm.

Similar tests were also performed to contrast the effect of new and worn tools on the ultrasonic signatures obtained from the milled surface. On the same steel plate used above, more than a factor of two increase in signal variation σ was observed on a surface cut with a worn tool as compared with a new tool. The increased variation resulting from the worn tool was believed principally due to an increased waviness in the milled surface.

The authors are especially appreciative of the laboratory help provided by R. Tregoning and H. Ryan of the University of Maryland, and D. Neal of NBS.

References:

1. G. V. Blessing and D. G. Eitzen, "Ultrasonic Sensing of Surface Roughness," abstract in IEEE Trans. on Ultrasonics, Ferroelectrics, and Frequency Control, UFFC-34, No. 3 (May 1987) p. 40.

2. U.S. patent applied for by authors (Jan. 1987).

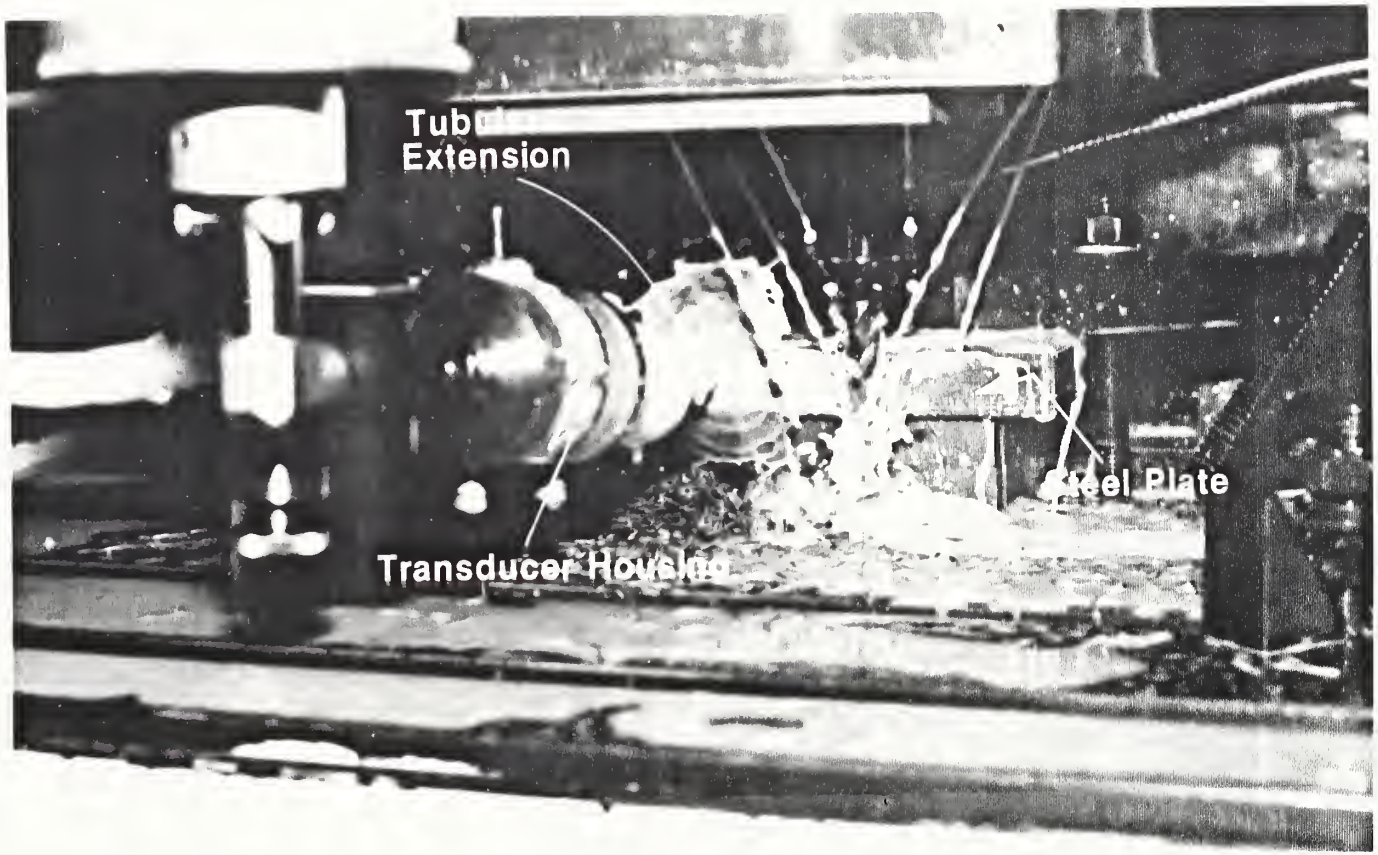


Figure 1. The ultrasonic sensor on-line in a milling center, interrogating the surface feature of a side-milled steel plate. The sensor includes a liquid-fitted chamber housing the transducer, and a tubular extension to couple the ultrasound to the part surface.

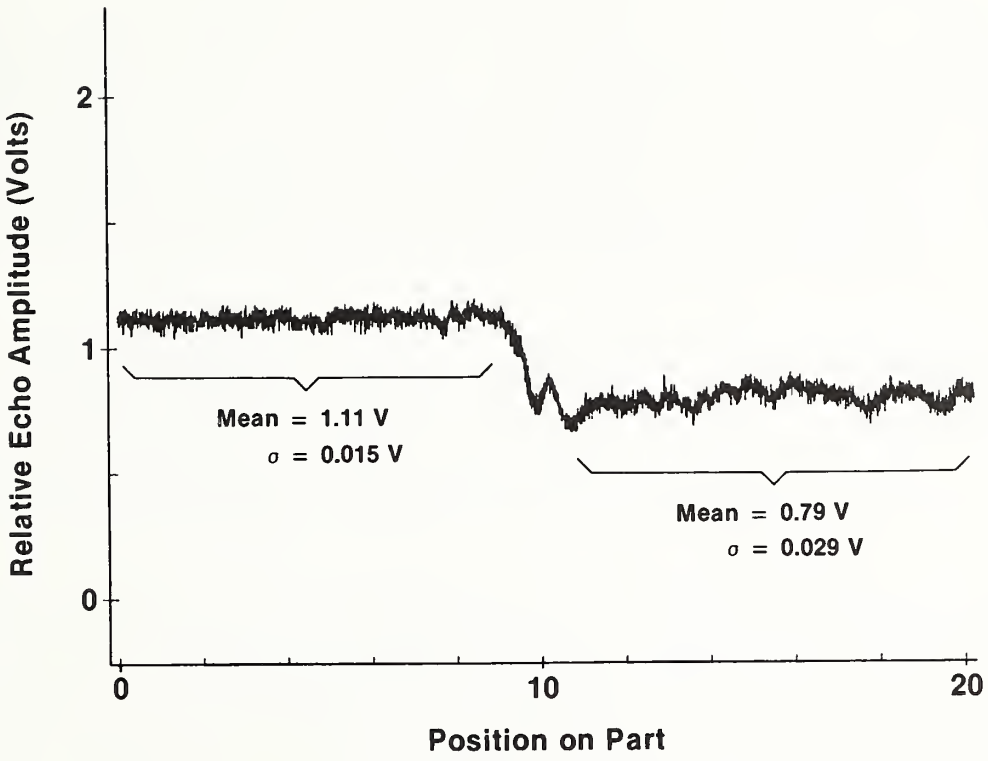


Figure 2. An ultrasonic echo amplitude signature obtained using the system depicted in Figure 1. The left and right half portions of the signature were generated respectively by a good and a defective (chipped) tool bit. The position on the machined part, plotted on the abscissa, has units of approximately one-half inch.

III. NDE FOR COMPOSITES PROCESSING AND INTERFACES

The goal of this activity is to develop generic approaches, sensors, and procedures for quantitative NDE of composites and interfaces. As in the two previous activities, the emphasis is on measurements that can be made during the manufacturing process to sense the properties of the product during critical stages of its formation and to provide the data required to control the process to optimize quality and productivity. Since the knowledge base on composites characterization is far from complete, we expect that a portion of this activity will be concerned with relating important composite characteristics with performance and then developing NDE monitoring methods.

This activity includes: research on utilizing fluorescent spectroscopy and dielectric measurements to monitor the processing of polymer matrix composites; applying ultrasonic techniques to improve the understanding of, and the ability to monitor, interfaces; utilizing photothermal radiometry (thermal wave NDE) to monitor the quality of ceramic coatings on metals; and utilizing infrared spectroscopy to monitor the quality of organic coatings on metals. Noteworthy accomplishments in this activity include:

- A major barrier to the implementation of composite materials in many applications is that the processing lacks the desired reliability. To improve the reliability, nondestructive evaluation measurement techniques need to be developed to improve process monitoring and control. Research has shown that fluorescence spectroscopy appears to be feasible to monitor the non-Newtonian shear viscosity of polymer melts during processing. This is an important processing parameter which reflects the molecular orientation of the polymer system and determines the flow volume of the processing line.

- Ceramic coatings are increasingly used in high temperature and high friction environments such as those found in internal combustion engines, rockets, turbines, and cutting tools. Their thermal and mechanical integrity is essential for proper performance and needs to be monitored on-line during production to assure quality and economic production. This research has demonstrated that photothermal radiometry (thermal wave NDE) can determine thermal conductivity and thermal diffusivity of thin ceramic films and has the potential to be a noncontacting on-line monitoring technique for plasma sprayed films. The thermal conductivity of plasma sprayed zirconia and chromia appears to be from two to five times smaller than the thermal conductivity of the bulk materials.

Process Monitoring for Polymer Matrix Composites

A. J. Bur, S. S. Chang, B. M. Fanconi, D. L. Hunston, F. I. Mopsik,
F. W. Wang, and W. L. Wu
Polymers Division
Institute of Materials Science and Engineering

A major barrier to the implementation of composite materials in many applications is that the processing lacks the desired reliability. This creates a need to develop nondestructive measurement techniques to improve

processing through process monitoring and control. The research reported here includes two areas: the first studies the feasibility of applying time-domain dielectric spectroscopy, optical fiber sensors, and ultrasonic techniques as NDE methods for process monitoring of thermoplastic, polymer-matrix composites; the second investigates the use of fluorescence spectroscopy to measure the shear viscosity of polymer melts during processing.

NDE for Process Control in Thermoplastic Composites:

There are two pressing problems in the area of thermoplastic composites; one is to insure the proper consolidation of the resin-fiber layers and the other is to control the crystallinity in composites based on partially crystalline matrix resins. On-line measurement techniques are sought in both areas. Crystallinity is particularly difficult to measure on-line and, consequently, the work here represents preliminary tests using two techniques that were thought to have the most potential: dielectric spectroscopy and ultrasonics. The recently developed polymer, poly(aryl-ether-ether ketone), known as PEEK, was chosen as the model resin for the first part of this study. PEEK shows promise as an engineering material for the matrix component in composites and has generated wide interest in composite industries. To measure consolidation, the application of fiber sensor techniques was investigated.

The approach undertaken on measurement of crystallinity, as the first step in this feasibility study, is to prepare PEEK films with different crystallinities, followed by investigations into the sensitivity of the two measurement methods in detecting these differences. To prepare such samples, a special mold has been constructed. This mold can be cooled very rapidly from the processing temperature to prevent or minimize crystallinity. These samples can then be annealed to induce various degrees of crystallinity. Construction of the mold has been completed and tests are currently underway to evaluate its performance.

In order to initiate studies before the mold was completed, PEEK film with a thickness of 65 μm was obtained from the manufacturer and analyzed for crystallinity using differential scanning calorimetry (DSC). The results show that the as-received film is amorphous with a glass transition temperature (Tg) at 141 °C. The DSC results further demonstrate that by annealing the amorphous PEEK at temperatures above 165 °C, crystallinities up to 38% can be obtained. The observed heat of fusion was 50 J/g. An annealing furnace with the appropriate temperature range was constructed to prepare samples with varying degrees of crystallinity. The sample chamber of the annealing furnaces was kept under dry nitrogen atmosphere to avoid polymer degradation. Films annealed at 250 °C for four hours had a crystallinity of 30% according to DSC results. Two samples with crystallinities of 0% and 30% were characterized by time-domain spectroscopy, and the result is summarized as follows.

Clear differences in the dielectric spectra were detected with the time-domain spectroscopy. However, the result is complicated by the presence of some conductive-like effect, probably due to some additive or contaminant inside the film. One possibility is release agent used during the molding of the film. Future work will be conducted using films prepared in our laboratory where additives and contaminant can be controlled.

The second technique to be investigated is ultrasonics. Unfortunately, the 65 μm film is too thin for such measurements. Efforts are now underway to prepare thicker samples so these tests can be conducted.

The other part of the effort on thermoplastics deals with utilizing optic fiber sensors as monitors of consolidation of plies during processing of polymer composite laminates. Consolidation requires resin flow between plies to eliminate entrapped air and promote fiber wetting. Temperature and pressure may be varied to promote consolidation, and the function of the sensor is to indicate that resin has flowed across the interface between plies and air and other volatiles have been eliminated.

Three approaches to consolidation monitoring based on optic fiber sensors have been analyzed in the current work. For each approach, the optic fiber sensors are placed between the plies of the laminate and the fraction of the total sensing fiber surface to contact with the resin is taken to be a measure of consolidation. One method is based on fluorescent probes such as those used in our previous studies [1, 2] to measure resin viscosity, and the other two utilize the refractive index difference between the resin and air (or other volatiles) as a measure of the fractional surface coverage. From the analyses, we conclude that the technique involving fluorescent probes in combination with optic fiber sensors has advantages over the other options in terms of sensitivity over a wide range of surface coverage and its measurement simplicity.

Fluorescence Monitoring of Polymer Melts:

The objective of this project is to use fluorescence spectroscopy as a tool to measure the non-Newtonian shear viscosity of a polymer melt which is undergoing shear induced flow. This is an important processing parameter which reflects the molecular orientation of the polymeric system and determines the flow volume of the polymer processing line. By measuring fluorescence anisotropy it is possible to calculate molecular orientation and to correlate the increase in anisotropy with the decrease in non-Newtonian viscosity. Since most polymer molecules are not fluorescently active, it is necessary to dope the polymer system with a low concentration of fluorescence chromophores. For the measurement of anisotropy, the dopant chromophore, which is usually a small low molecular weight molecule, must be chemically bound to a polymer molecule in order that the shear stresses are translated into orientation of the chromophore.

Our efforts during FY87 have focused on the design and development of measurement equipment and on the identification and chemical synthesis of the fluorescent chromophore which we will use to measure fluorescence anisotropy of a polymer solution. We have carried out preliminary experiments using concentrated solutions of polybutadiene in toluene. The feasibility of the technique was established by observing the fluorescence from an impurity in the polybutadiene. Changes in anisotropy were observed with changes in applied pressure. These experiments will continue when the chemical synthesis of the polymer chromophore is complete.

Our plan is to correlate the measurements of anisotropy and viscosity for a polymer solution and for a polymer melt. We intend to demonstrate this monitoring technique using an industrial processing machine.

References:

1. B. M. Fanconi, F. W. Wang, and D. L. Hunston, "Comparisons Among Process Monitoring Techniques," Proc. ANTEC87, SPE, Los Angeles, May, 1987.

2. S. Chang, D. Hunston, and F. Mopsik, "Correlation of Cure Monitoring Techniques," Proc. SAMPE Tech. Conf., Wash., D.C., August, 1987.

Guided Interface Waves

H. N. G. Wadley, J. A. Simmons, E. Drescher-Krasicka[1], M. Rosen[2], T. Hsieh[2], and K. Hirshmann[2]
Metallurgy Division
Institute for Materials Science and Engineering

[1] Research Scientist on contract, Johns Hopkins University, Baltimore, MD.
[2] Guest Scientists, Johns Hopkins University

A new generation of advanced composites with metal and even ceramic matrices shows the greatest promise for satisfying many future materials needs. Early attempts at processing these materials have met with mixed success with wide variations from batch to batch. The bulk behavior of these composites has been predicted to be strongly influenced by the local elastic properties, residual stresses and adhesion of the interface. Techniques to nondestructively measure these newly perceived quantities of importance do not exist. Thus, it is not possible experimentally to (a) confirm the micromechanical model predictions, (b) explore the relationships between interface properties and processing variables, and (c) ensure acceptable interface properties in materials destined for aerospace and other systems.

This research program is directed at developing experimental techniques for characterizing interfaces in composite materials and coupling this expertise to other composite research programs to enable optimum interfaces to be designed for the next generation of advanced composites. We have explored guided interface waves, a family of ultrasonic techniques with potential for characterizing interface elastic and anelastic properties.

Last year we reported a theoretical and experimental study of guided interface waves in a model system consisting of a stainless steel rod shrink-fitted into an aluminum matrix. We obtained good agreement between measured and theoretically predicted interface wave velocities. This year we have achieved the formulation of the ultrasonic scattering/interface wave propagation problem for a general interface with interface zones. A general theory encompassing planar, cylindrical, and spherical interface zones was developed. The constitutive relations for the interface zone permit viscoelastic effects to be modeled. Conservation relations, following from the geometry of the model and the equations of motion, allow the result of any scattering or interface wave experiment to be expressed in terms of the superposition of monochromatic eigenwaves characteristic of the composite structure and interface zone. Each of these eigenwaves is shown to be determined everywhere by a six component vector which is related to simple waves in the matrix and reinforcement. Distinguishing the incoming from the outgoing components in an eigenwave allowed

us to write down the scattering matrix, and interface waves are shown to arise under conditions where the scattering matrix fails to exist. This general approach shows that there is a rich variety of interface waves other than non-dissipative Stoneley type waves. These include 'leaky' or 'evanescent' waves which might be experimentally detectable and, therein, provide a means for characterizing the interface zone [1].

Extensive detailed calculations, including particle displacement trajectories and energy flow diagrams, have been carried out for the isotropic fiber-reinforced and laminate geometries without interface zone [2]. New search algorithms for finding roots in the complex plane have permitted systematic mapping of leaky interface waves in many composite systems, including Al-Fe, Al-SiC, Al-B, Fe-Ti. Near-term future work in this area will extend these calculations to finite thickness interface zones and to the incorporation of anisotropy and scattering.

Experiments were carried out this year in the planar geometry and related to the theory of interface waves as a function of the elastic constants of the matrix and reinforcement. Here, a steel-titanium system was used. Elastic properties of the steel were varied by heat treatments. The titanium exhibited an anisotropy due to rolling texture. This anisotropy allowed exploration of quite a wide range of material pairs.

Four types of interface waves were found to be present over the range of microstructures (elastic properties) available: Stoneley waves, two types of leaky waves (those leaking into the steel and those leaking into the titanium) and one type of divergent wave (one that transports energy parallel to the interface but whose displacement increases exponentially away from the interface into the steel). Table I summarizes these results.

Table I. Interface wave velocities for four different microstructures in 4340 steel and two different orientations of a block of rolled Ti-6A1-4V.

4340 STEEL PROPERTIES				STEEL/TITANIUM* DUPLEX PLATE				
Microstructure	Density (kg/m^3)	Long Velocity (m/s)	Shear Velocity (m/s)	Case No.	Rolling Direction of Ti Plate	Predicted Wave Type	Interface Wave Velocity	
							Measured (m/s)	Predicted (m/s)
Fine Pearlite (Held at 1200F)	7340	5952	3250	1A	⊥	Stoneley	3217 ± 15	3247
				1B	∥	Leaky in Ti	3217 ± 15	3232
Tempered Martensite (Quenched and Temp.)	7805	5868	3170	2A	⊥	Divergent in Steel	3216 ± 55	3170
				2B	∥	Stoneley	3166 ± 15	3162
Bainite (Air cooled)	7317	5869	3175	3A	⊥	Divergent in Steel	3221 ± 55	3175
				3B	∥	Stoneley	3162 ± 15	3166
Fine Pearlite (As Received-Hot Rolled)	7839	5934	3235	4A	⊥	Stoneley	3223 ± 15	3233
				4B	∥	Leaky in Ti	3206 ± 15	3218

*Density of Ti-6Al-4V = 4430 kg/m^3,
Longitudinal velocity of Ti-6Al-4V = 6287 m/s in both directions
Shear velocity of Ti-6Al-4V: { Perpendicular to rolling direction (⊥) = 3287 m/s, Parallel to rolling direction (∥) = 3171 m/s.

Leaky interface waves were experimentally generated and detected [3] in the fiber reinforced geometry for the steel in Al (Figure 1) and for SiC in Al (Figure 2). Figure 1 shows the experimental results (points) obtained at the surfaces of the aluminum matrix parallel and perpendicular to the cylindrical Al-steel interface [2]. There are pronounced maxima visible for three different leaky modes (a, b, c). These maxima are connected to an angle of maximal energy flow associated to the Poynting vector integrated from the point of origin of the mode at the interface. The theoretical results (the displacement fields calculated for the Al-steel cylindrical sample) were compared with the experiment for the three leaky modes a, b, and c. Mode a exhibits a maximum angle of leakage at about 50°. The high leakage angle is associated, in this case, to a high attenuation along the interface. Measurements carried out at the end of the sample on the face perpendicular to the interface are also shown in Figure 1 (b, c). At 2.5 MHz, the maximum energy flow for mode b is 13-14°. The small maximum occurring near the interface at about 3° could be due to the third leaky mode c [2]. Both the leakage angles and velocities were compared with theory in the Al-Fe system by careful mapping of interface displacements, while the velocity dispersions curve for Al-SiC was checked over a range of velocities.

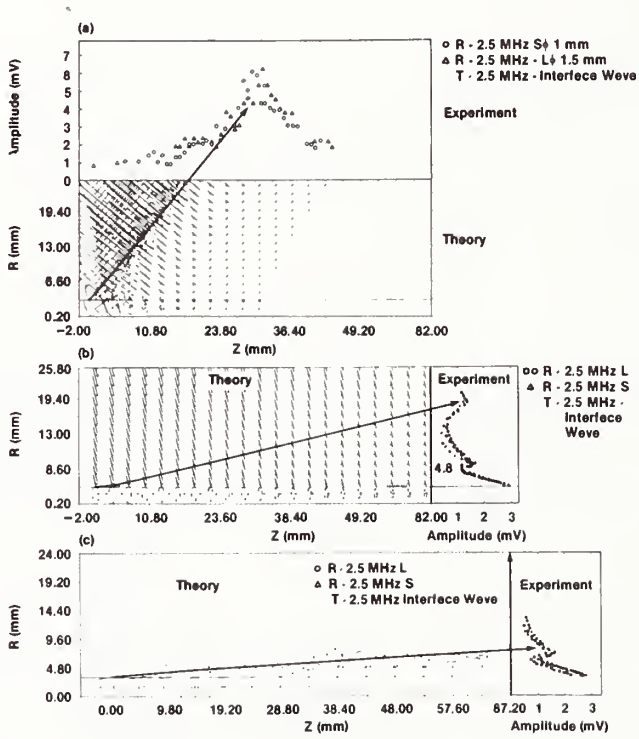


Figure 1. Experimental identification of radial displacement modes created in a cylindrical sample of steel in aluminum.

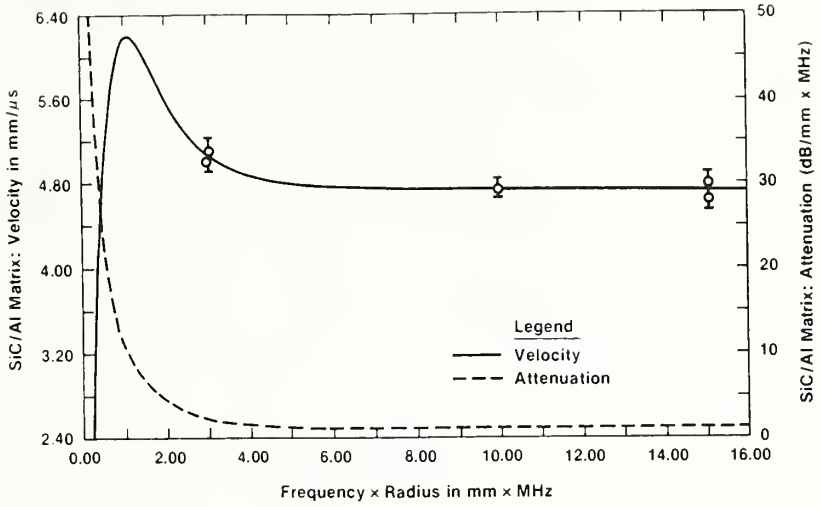


Figure 2. Measured velocities at the Al-SiC interface as a function of frequency (points) compared with the theory (continuous line).

We have begun to devise schemes for implementing these approaches on actual composites. Near term future work will concentrate on measuring local velocities along the fiber and developing scattering methods which can be scaled down for use in acoustic microscopy. The work in acoustic microscopy will be carried out in collaboration with Professor B. T. Khuri-Yakub's group at Stanford University.

References:

1. H. N. G. Wadley, J. A. Simmons, E. Drescher-Krasicka, M. Rosen, R. B. Clough, T. Hsieh, K. Hirschman, and F. Biancaniello, "Composite Materials Interface Characterization," NBS Internal Report 87-3630, 1987.

2. J. A. Simmons, H. N. G. Wadley, E. Drescher-Krasicka, M. Rosen, and T. Hsieh, "Ultrasonic Methods for Characterizing the Interface Zone in Composites," Review of Progress in ONDE Conference, Williamsburg, VA, 1987, in press.

3. E. Drescher-Krasicka, J. A. Simmons, and H. N. G. Wadley, "Guided Interface Waves," Review of Progress in Quantitative Nondestructive Evaluation, 6A, eds. D. O. Thompson and D. E. Chimenti, pp. 1129-1136 (Plenum Press, 1987).

Photothermal Radiometry for the Evaluation of Coatings

H. Frederikse and A. Feldman
Ceramics Division
Institute for Materials Science and Engineering

The purpose of this project is to assess thermal wave techniques as non-contact methods to monitor the quality of ceramic coatings. Such coatings are increasingly used in high temperature and high-friction environments: engines, turbines, rockets, cutting tools, etc. Their thermal and mechanical integrity is essential and should be determined and controlled on-line. Photothermal radiometry, which opens the possibility to do this, depends on the propagation of a thermal wave into the coating and reflection by the metal substrate. Heat is generated by a modulated laser beam at a spot on the ceramic surface and the surface temperature is recorded with an infrared detector aimed at the same spot. The phase angle difference $\Delta\phi$ is measured as a function of the modulation frequency f. Analysis of the relation between $\Delta\phi$ and $f^{1/2}$ will yield the desired information: thermal diffusivity α and reflectivity R.

Last year we assembled the experimental equipment, and this year we carried out a number of experiments. The photothermal radiometry signal vs chopping frequency from plasma sprayed films of zirconia and chromia has been fit to a mathematical model and values for thermal diffusivity and thermal conductivity have been obtained as a means for monitoring the quality of ceramic coatings. The bulk of the measurements were performed with the set-up shown in Figure 1. During the fall of 1986, we received an A-O modulator for the visible range of the spectrum. In principle, this device allows modulation at low MHz frequencies. However, the lock-in amplifier limits the frequency range to 200 kHz. The A-O modulator was used in conjunction with an argon laser in some experiments.

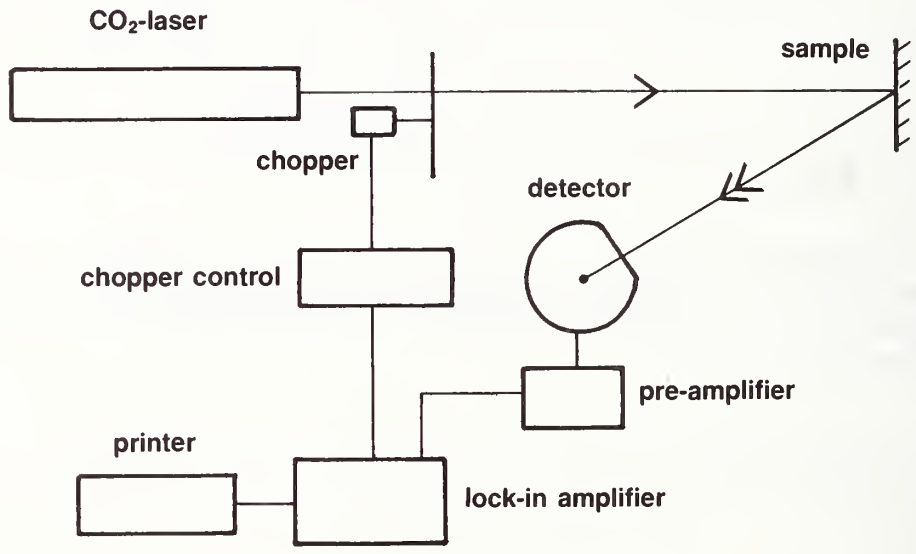


Figure 1. Experimental arrangement for photothermal radiometry.

In most of the measurements, the hot spot at the surface of the sample was produced by a CO_2 laser tunable from 9.4-10.6 μm. The oxide coatings used in the present experiments, chromia and partially stabilized zirconia, are fully opaque in this infrared spectral range. The detector is a liquid-nitrogen-cooled InSb-PV cell which has its maximum detectivity at 5.5 μm just before the cut off at 6.5 μm. The emission from the hot spot is focused on the cell by means of a CaF_2 lens while a thin Si window is used for rejection of visible and near infrared radiation. The temperature signal S and phase angle $\Delta\phi$ are read on a lock-in amplifier using the frequency f from the chopper control box as the reference signal. For oxide samples studied during this period the optimum frequency range is 5-1000 Hz which is covered with two chopper blades (2 and 6 open sectors, respectively).

The relation between $\Delta\phi$ and f is as follows:

$$\Delta\phi = \phi - \phi_{ref} = \arctan\left(\frac{2R\sin 2x}{e^{2x} - R^2 e^{-2x}}\right)$$

where: $x = L/\mu = L\sqrt{\frac{\pi}{\alpha}} \cdot f^{1/2} = F \cdot f^{1/2}$

L = coating thickness
μ = thermal diffusion length = $\sqrt{\alpha/\pi f}$
R = reflection coefficient = (1-b)/(1+b)

$$b = \sqrt{\frac{\kappa_s \rho_s C_s}{\kappa_c \rho_c C_c}}$$ (s → substrate and c → coating)

α = thermal diffusivity
κ = thermal conductivity
ρ = density
C = specific heat

Experimental results of $\Delta\phi$ vs $f^{1/2}$ are shown in Figure 2 for a 100-μm chromia coating on 1/16-inch-thick Al. Curve fitting yields values of F and R which in turn permit determination of α and κ. Table I is a summary of the results obtained on four chromia and two zirconia samples. The thermal conductivities of the coatings appear to be two to five times smaller than the thermal conductivities of bulk materials.

In the future, we plan to extend the thermal evaluation of oxide coatings to higher temperatures. We will also test a number of ceramic coatings (ZrO_2, MgO, SiC, etc) of various densities, grain size, substrate temperature (during deposition), etc. In addition, we plan to evaluate the mechanical perfection (or imperfection) of ceramic coatings in terms of cracks and non-adherence.

Reference:

1. A. Lachaîne, J. Appl. Phys. 57, 5075 (1985).

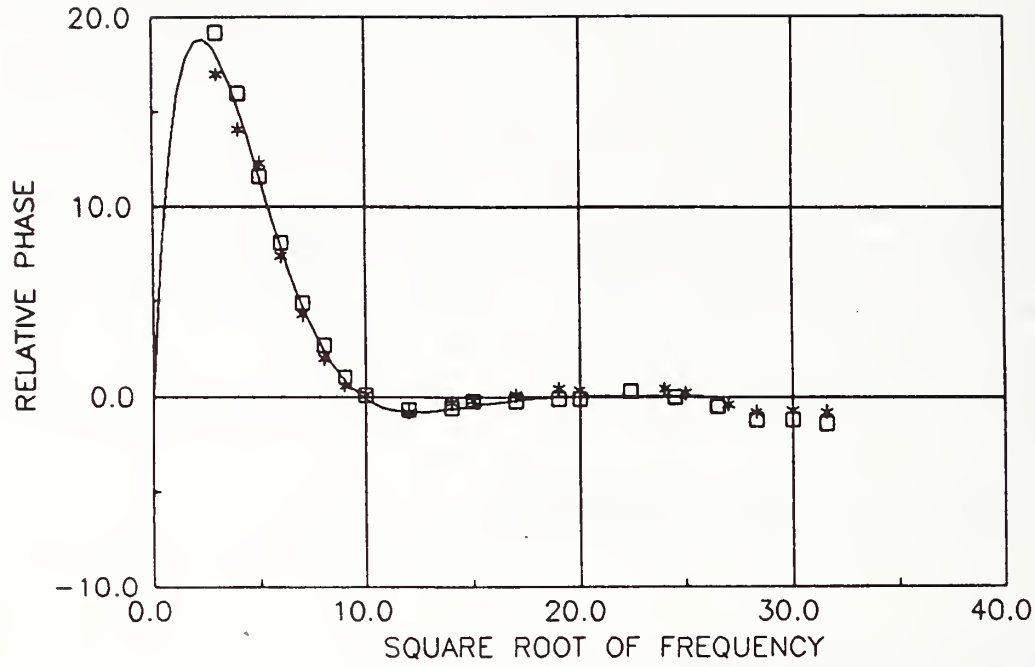


Figure 2. Relative phase angle as a function of (frequency)½

Table I. Summary of Thermal Diffusivities (α) and Thermal Conductivities (κ)

Sample	Thickness L (μm)	Reflec. Coeff. R	Thermal Diff. α (cm^2/sec)	Thermal Cond. κ (W/cm deg)	Thermal Cond. κ [Handbook*]
Cr_2O_3 - #1	100	0.5	0.0129	0.0835	0.10 - 0.33
Cr_2O_3 - #2	150	0.58	0.0140	0.0695	
Cr_2O_3 III	90	0.62	0.025	0.082	
Cr_2O_3 IV	125	0.62	0.015	0.063	
ZrO_2 A	60	.95	0.017	0.0074	0.018 - 0.022 (part.stab.)
ZrO_2 B	97	.92	0.017	0.0120	0.0069 - 0.024 (plasma spr.)

* "Ceramic Source 1986" (Am. Ceramic Soc.), pp. 350-351.

Interface Bond Strength of Organic Films on Metals Using Reflection/Absorption Fourier Transform Infrared Spectroscopy

Tinh Nguyen, M. E. McKnight, E. W. Byrd, and J. Seiler
Building Materials Division
Center for Building Technology

The performance of a film on a metal substrate depends on the cohesive and adhesive properties of the film. For polar organic films, adhesion is governed mostly by the polar interactions between the film and substrate, while one of the factors controlling the cohesive strength is the degree and extent of hydrogen bonding within the film. In service, both the adhesive and cohesive properties are likely to change with time and affect the durability of the film. A technique that is in rapid development as a nondestructive method for studies of thin and thick films on bulk metals is reflection/absorption infrared spectroscopy (RAS). RAS, which measures the fractional reflective changes of the substrate due to the absorption by the film, was developed for very thin films on highly polished substrates [1] and extended to thick films (>100 nm) recently [2]. Since transmission infrared spectroscopy is known as the most effective method for studying hydrogen bonding in solutions [3], it was appropriate to investigate RAS as a nondestructive method to monitor the degree of H-bonding in films on metals and to relate these changes to mechanical adhesion.

The aim of our research is to develop nondestructive evaluation techniques to monitor interface bond strength of organic films on metals. This year our research had two objectives; to monitor the molecular changes resulting from change in the degree of H-bonding, and to determine the relationship between Fourier transform infrared reflection/absorption (FTIR-RAS) spectral changes and mechanical adhesion.

In the first phase of the research, to monitor molecular changes resulting from changes in the degree of H-bonding, highly cross-linked epoxy and polybutadiene (PB) films about 2 μm thick were spin coated on mechanically-polished, rolled steel and cured at ambient conditions for four weeks. RAS spectra were taken before and during exposure to 40 °C and 80% relative humidity (RH). This was done using a variable angle external reflection accessory connected to a FTIR spectrometer, as shown in Figure 1.

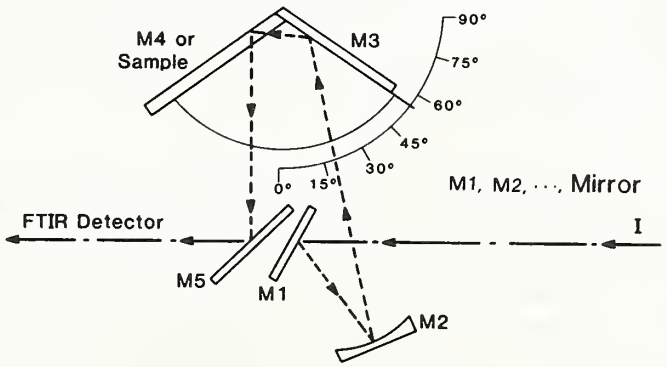


Figure 1. Optical layout of the reflection/absorption FTIR experiment.

Figures 2a and 2b are the expanded and normalized spectra (intensity versus wavenumber, cm^{-1}) of the OH stretchings of epoxy and PB films on steel at several exposure periods.

The most interesting changes in the RAS spectra of both epoxy and PB films on steel during service are the shifts of the peak maxima in the 3150-3650 cm^{-1} region. A shift to lower frequency indicates an increase in the strength of the H-bonding in the system, and vice versa [3].

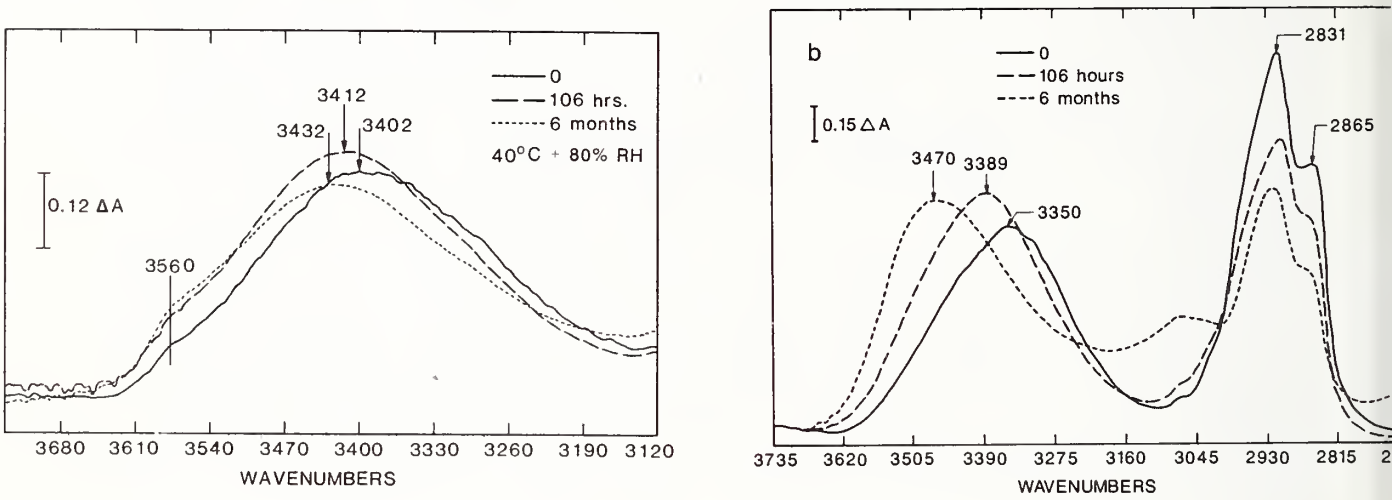


Figure 2. Expanded and normalized FTIR-RA spectra in the 3150-3650 cm^{-1} region of epoxy and polybutadiene films on steel exposed for different periods at 40 °C and 80% RH. (a) epoxy and (b) polybutadiene.

For the epoxy sample, a shift of about 30 cm^{-1} to higher frequency, which translates to about 1 kcal/mole, indicates a reduction in the degree of H-bonding in the system. Since our preliminary experiments showed that there is no strong interaction between an epoxy film and a steel substrate, these results suggest that the reduction of H-bonding in the epoxy during exposure occurred within the film and not at the film/steel interface.

For PB films on steel, the 120 cm^{-1} shift (ca. 4 kcal/mole) to higher frequency of the PB coated film during service is attributed to the reduction of the H-bonding of the system (both in the bulk polymer and at the metal/polymer interface). The results obtained indicate that FTIR-RAS can be used to follow molecular changes resulting from changes in the strength of H-bonding of films on metals.

In the second phase of our research, concerned with relating FTIR-RAS spectral changes with adhesion, thicker PB films were spin coated on mechanically-polished steel and copper samples. A pneumatic pull-off adhesion test was employed. FTIR-RAS spectra and adhesion measurements were taken after exposure in a more severe environment, 80% RH at 75 °C, to accelerate molecular changes of the thicker films. Figure 3, which plots the OH maximum frequency vs. the adhesion, summarizes the results obtained after five exposure periods. From Figure 3, it appears that there is a good correlation between the bonding strength and the OH peak frequency; the higher the peak frequency the higher

the bonding strength. However, this is contrary to our hypothesis that as the OH peak frequency increases the bonding should decrease. This may be due to the thick films and the higher exposure temperature employed. Results from our previous work and from Dickie's [4] indicate that in a corrosive environment, PB on steel debonds cohesively, but the locus of failure is very close to the interface. Spectroscopic changes associated with this interfacial failure may be masked by larger ones caused by the continued curing and oxidation of the bulk polymer. Additional experiments using relatively thin films are being carried out to verify this explanation and to validate the original hypothesis.

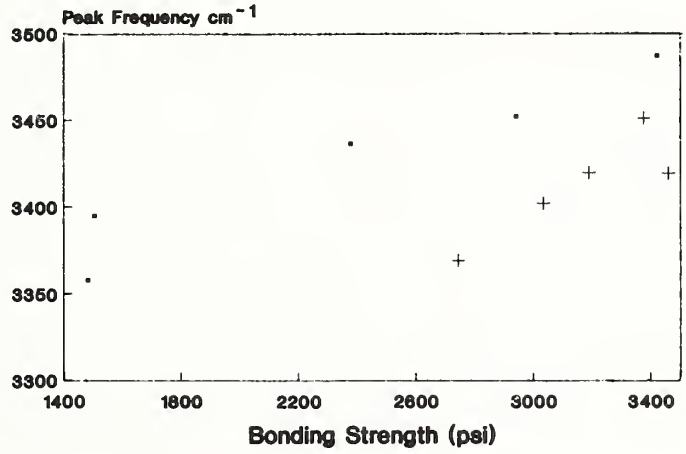


Figure 3. Correlation between the OH maxima frequency and bonding strength of polybutadiene films on steel and copper. ■ - steel; + - copper

In carrying out this research in 1987, we observed that the technique may be useful in monitoring the growth and depletion of many functional groups of polymer films on metals during curing. Future efforts will explore RAS as a nondestructive method for monitoring the degree of cure of coatings on metals.

References:

1. R. G. Greenler, J. Chem. Phys. 44, 310 (1966).

2. Tinh Nguyen and E. W. Byrd, Proc. Polym. Mat. Sci. Eng. 55, 665 (Am. Chem. Soc., Washington, D.C., 1986).

3. G. C. Pimentel and A. L. McClellan, Ann. Rev. Phys. Chem. 22, 347 (1971).

4. R. A. Dickie, et al., Ind. Eng. Chem. Prod. Res. Dev. 20 (1981).

IV. NDE STANDARDS AND METHODS

The objective of the Standards and Methods activity in the Nondestructive Evaluation Program is to provide the scientific understanding of NDE measurement methods and to develop, maintain, and disseminate effective standards for NDE measurements which are traceable to national standards.

In the early years of the Program (i.e., 1975-1980) there was some thought that the standardization of NDE measurements was a finite task which could be completed in a few years, after which a comparatively small maintenance effort would suffice. It soon became clear, however, that this was not to be the case. New methods and techniques for NDE continue to demand new standards while, simultaneously, industrial acceptance of the Bureau's work on NDE standards has created ever-increasing calls for more of the same. During FY 1987, for example, the Bureau's method for calibrating acoustic emission transducers was adopted by ASTM in record time and research is progressing rapidly on secondary calibration methods that can disseminate the standard more efficiently. The Department of Defense is evaluating a new NBS test method for characterizing eddy current probes and a new approach to electromagnetic NDE, using capacitive probes, is being developed. Standard methods for evaluating radiographic image quality at low and high voltage levels are nearing completion as plans are being formulated for new standardization efforts in real time radiology. Acoustic emission, capacitive NDE, real time radiology, and thermography were unknown or in their infancy ten years ago; today these methods are in use and in need of standardization.

Nine project reports from the NDE Standards and Methods activity follow. Without exception, they document important progress. A few brief examples of this progress are summarized here.

- Three workshops were convened at NBS during FY 1987 in order to maintain active awareness of industry's needs for NDE standards and to facilitate the dissemination of these standards. A specialists' workshop on laser-based optical methods, used for measurement of droplet/particle size distribution in laboratories and industrial systems, was held in Nov. 1986. The capabilities and limitations of advanced sizing techniques were discussed and the needs for standards were explored. Another specialists' workshop, this one on real time x-ray radiology, was convened in April 1987. This workshop critically evaluated the standards requirements of this rapidly emerging technology. The proceedings of this intensive two-day meeting are expected to guide the NBS effort in this field for the next few years. The Bureau's second biannual workshop on the calibration of standard leaks was held in September 1987 in order to exchange information on leak calibration systems, primary standards, leak artifact stability, and leak measurement requirements and to present the latest progress and measurement services which are available from NBS in this field.

- A new NDE sensor, based on a planar array of capacitive elements, was developed in collaboration with Stanford University. The sensor has demonstrated exceptional sensitivity to flaws in ceramics and other nonconductive materials. Using printed circuit board technology, a probe with a sensing area of approximately 2 mm^2 was fabricated, which has detected 4-μm wide surface cracks in glass and artificial notches 4 mm beneath the surface of a dielectric sample, with a signal-to-noise ratio exceeding 10.

- o NBS work on infrared thermography in the NDE Program has led to the identification of meaningful performance parameters for thermographic equipment, and to the formulation of methods for evaluating the parameters. The first of these methods, "Standard Test Method for Minumum Resolvable Temperature Difference for Thermal Imaging Systems" has been adopted by ASTM (E 1213) and a second standard, dealing with minimum detectable temperature difference, is nearing completion. These standards will facilitate the specification and evaluation of thermographic equipment for specific NDE applications.

Standards and Methods for Ultrasonics and Acoustic Emission

D. G. Eitzen and the Ultrasonic Standards Group
Automated Production Technology Division
Center for Manufacturing Engineering

The objective of this project is to develop and disseminate artifact and documentary standards, develop and maintain measurement services, and develop new or improved methods for acoustic emission (AE) and ultrasonic NDE techniques.

Our documentary standards activities in the last year have been extremely extensive and fruitful. Our strategy is to perform the necessary measurements and analysis, design and pilot the necessary round robins, assist in the adoption of U.S. standards (mainly through ASTM) and then use these U.S. standards as the technical basis for ISO standards. Arguably, the biggest payoff is in this last step which promotes U.S. industrial competitiveness.

ASTM standard E 127 on procedures for measuring the response of ultrasonic reference blocks has been significantly modified. Response curves and tolerances were changed and a new procedure for measuring area-amplitude blocks (based on NBS-developed procedures) has been included. The modified document will be balloted this fall. A basis for more fundamental changes has been established, but implementation awaits further industrial support.

With partial support from the Army Materials Technology Laboratory, the technical work on a MIL Standard ultrasonics glossary has been completed and a proposed MIL Standard on "Field Assurance of Acoustic Emission System Operation Using Simulated Acoustic Emission Events" (in collaboration with Physical Acoustics Corporation) is progressing. The method relies on the NBS-developed Hsu pencil source.

An article on "AE Sensors and Their Calibration" [1] was completed for the American Society for Nondestructive Testing Handbook on Acoustic Emission Techniques. In addition to background information on AE sensors, the article disseminates information on the NBS primary calibration service [2], and provides information on the NBS-developed tools for secondary calibration [3,4].

Activities related to ISO Technical Committee (TC) 135 on nondestructive testing have increased considerably, especially those relating to Subcommittee (SC) 3 on acoustic methods. Plenary meetings of TC 135 and SC 3 were held in

May 1987 in Yokohoma, Japan. All of the US/NBS agenda items were completed including the election of one of the authors (D.G.E.) as chairman of SC 3 [5]. ASTM standard E 1106 has been rewritten for consideration as a draft ISO standard on the primary calibration of AE transducers.

Secondary methods of AE sensor calibration are also under development. These are needed so that at least transducer houses and the larger laboratories can calibrate "working" transducers with traceability to national standards. This traceability can be most easily obtained by using the NBS conical transducer [3] (Standard Reference Material No. 1856) as a transfer standard. A prototype secondary calibration system is being constructed. One NBS conical transducer will be used as a source and another as a receiver; the output of the latter will be compared with the output of the AE sensor under test. The transfer medium in the prototype is a large (but inexpensive) steel plate. The steel plate has been tested in several ways. The sound speeds of the plate have been accurately measured and used in a computer code [4] to calculate the theoretical dynamic displacement of the plate. This predicted displacement is compared with the measured displacement due to a breaking capillary event [6]. The results of one such test are shown in Figure 1. Several modules of the required signal processing have also been constructed. The method(s) will be promulgated through ASTM and ISO.

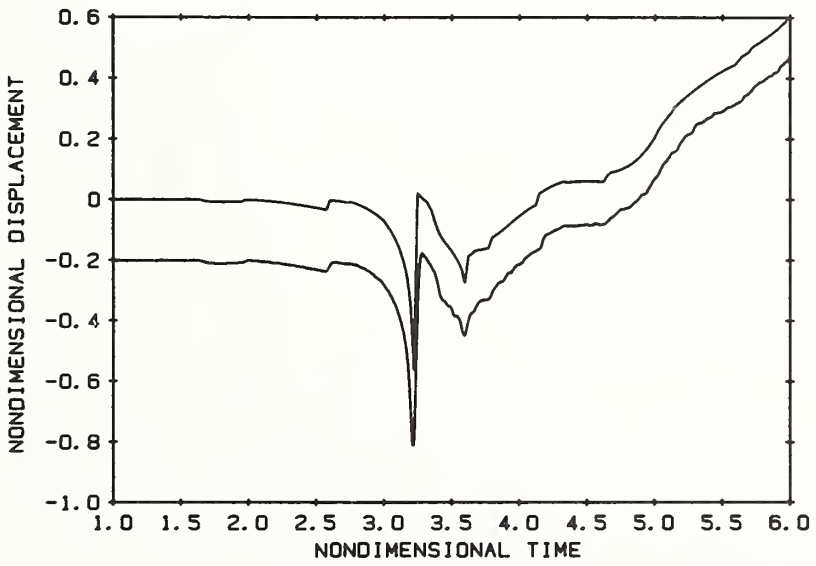


Figure 1. Comparison of experimental waveform (lower trace) and calculated waveform for a steel plate. Conditions: source, capillary break; receiver, NBS conical transducer; plate thickness, 33 mm; source and receiver on the same side, separated by three plate thicknesses. Calculations are performed using the program of reference 4.

The AE transducer calibration facility is indispensible as an NDE national standard, as a tool for the development of AE transducers, and for analyzing the performance parameters of AE transducers. The facility was upgraded substantially in the last year. New transient recorders have been integrated into the calibration system. They have an additional two bits of accuracy (10 bit), better linearity, and much better performance at high slew rates than

the old transient recorders. A dedicated IBM-compatible computer has also been interfaced and programs rewritten. The previously shared-use mainframe became obsolete. The calibration process has been speeded up and the calibration data are transferable to other IBM-compatible computers. Improvements in the electronics processing of the calibration signals have also resulted in a lower noise floor.

Several aspects of transducer performance have resulted in important conclusions. By constructing an extended capacitance transducer with a circular element approximately 2 cm in diameter and calibrating it with the surface pulse, the aperture effect was accurately measured. The aperture effect can also be calculated theoretically by integrating the surface pulse waveform under the transducer to obtain a theoretical time waveform response. These are compared in Figure 2. The experiment and theory also compare very well in the frequency domain; the frequency response is nearly a Bessel function [7].

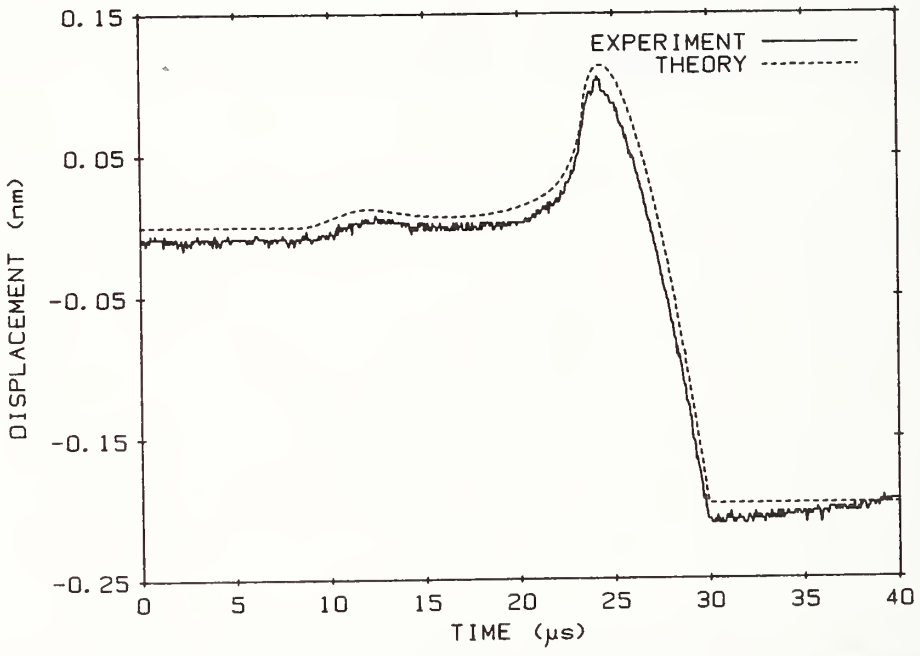


Figure 2. Results of the calculation of the aperture effect compared with an experimental waveform from a capacitive disk transducer. Conditions: source to receiver distance, d = 0.101 m; transducer radius, a = 9.8 mm; surface pulse generated by a glass capillary break on the NBS standard steel block for calibrating AE transducers.

Another aspect of transducer behavior which is very important is the transducer loading effect. An AE sensor alters the motion of the surface to which it is attached. The motion under the transducer depends on the interaction of the mechanical impedance of the test medium with that of the transducer. However, in calibrating a transducer the motional input is considered to be the motion of the surface with no transducer present. For different media the motion for the same input and same transducer will, in

general, be different. That is, the calibration is fundamentally dependent on the test medium. But, for example, if a transducer is calibrated on a steel block, how badly in error will it be if used on a glass block?

In order to more quantitatively assess this effect, transducers were calibrated on the NBS steel block and also subjected to surface-pulse waveforms on aluminum, glass, and plexiglass, and analyzed in a way to generate an approximate calibration [7]. The results for the NBS conical transducer are shown in Figure 3. Analysis of the NBS conical transducer [8] on these same media are shown in Figure 4. The results presented in the two figures agree very well considering the necessary approximations in both, and it is clear that the loading effect is more than an order of magnitude for plastic. This is a major reason for concern about the lack of calibration capability for polymer-based composites and a reason for development of a conical transducer for lower impedance materials.

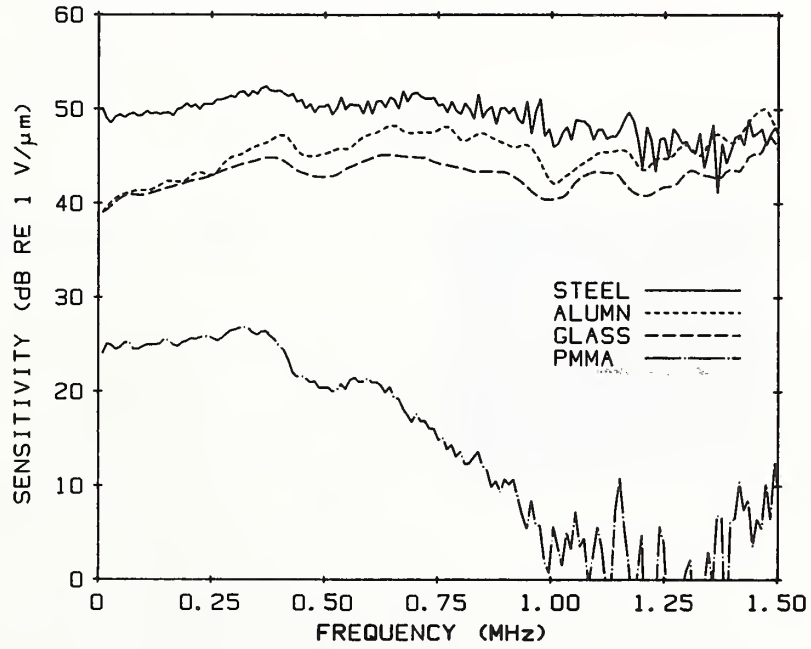


Figure 3. Approximate calibration of an NBS conical transducer on blocks of different materials. A pencil break was the source for all except the steel block calibration. The results were compensated for the pencil-break waveform.

Resources have also been devoted to work on new and/or improved acoustic emission and ultrasonic methods. This work resulted in several major developments in the last year. Generally, it is no longer possible to logically separate the basic methods work on AE and ultrasonics except through specific applications. For example, a dropping-ball source and point detector was motivated by interest in simulated AE sources but was applied to laboratory inspection of concrete by the "impact/echo" method (essentially a pitch/catch ultrasonic method) and is now being introduced to industry through other NBS programs.

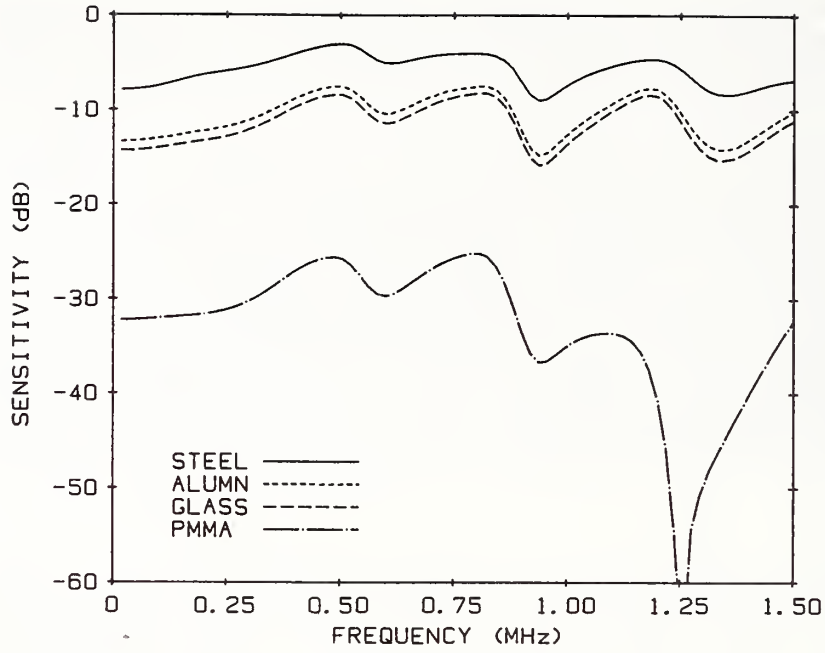


Figure 4. Calculated sensitivity of the NBS conical transducer of Figure 3 on four different materials. Calculations are based on the theory of reference 8.

A new ultrasonic wave speed measurement technique has also been developed [9]. In this technique the echo waveforms generated by a point impulse and detected by a point normal displacement (NBS conical) transducer can be used to determine both the longitudinal and shear wave speeds of a sample. No special shear couplant or shear transducer is needed. Both wave speeds are determined simultaneously from a single event or impact. This new technique contrasts with traditional ultrasonic velocity measurement methods in which the approximations of well defined modes of vibration and planar wave fronts are assumed, and without which the method would not be possible. The NBS conical transducer [3] was an essential tool for the experiments, and the computer program for predicting surface motion [4] was essential for optimizing the test configuration. It has been shown that from a measured waveform, the longitudinal and shear wave speeds can be explicitly calculated from the time intervals between the first and second, and the second and third echo arrivals, the thickness of the sample and the location of the detector [9]. The method has some important technological applications for on-line control of materials processing, which we expect to pursue in the coming year. Significant progress was recently reported on the laser generation of ultrasound [10]. This work showed that the ultrasound produced by a point normal force or a point dipole using a pulsed laser can be accurately predicted by convolving the appropriate Green's function with the force-time history of the source [11]. In a collaboration with the National Research Council of Canada, this work has been extended so that the long-sought-after goal of laser generated/laser detected ultrasonic testing can now be

developed, optimized and applied. The code for calculating exact surface displacements was extended so that the displacements from extended sources could be calculated. Experiments by NRC used a ring of light so that more ultrasonic energy could be created while still not exceeding the surface damage level and so that the displacements at the center of the ring had adequate amplitudes to be detected by a second laser. In Figure 5 the interferometrically measured ultrasound is (very favorably) compared with the displacement waveform predicted by the extended Green's function approach. The key point is that the computer code is an invaluable tool for further developing such techniques. Other or more complex light deposition patterns will be required; the code is the ideal tool for designing the patterns and for determining system performance.

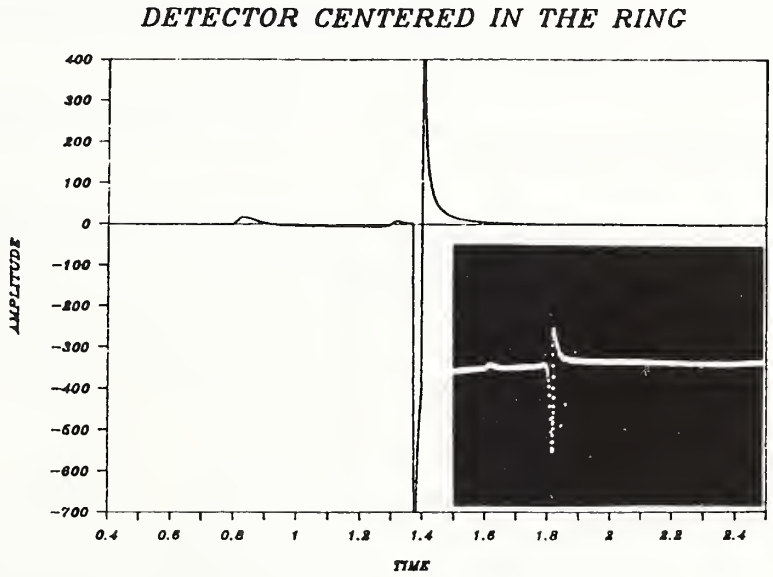


Figure 5. Comparison of experimental waveform (inset oscillograph) and calculated waveform for a plate which is subjected to laser-induced ultrasound. The laser energy is deposited as a ring of light. The laser detector is focused at the center of the ring.

References:

1. D. G. Eitzen and F. R. Breckenridge, "Acoustic Emission Sensors and Their Calibration," American Society for Nondestructive Testing Handbook (to be published).

2. F. R. Breckenridge, "Acoustic Emission Transducer Calibration by Means of the Seismic Surface Pulse," J. Acoustic Emission, Vol. 1, No. 2, pp. 87-94 (1982).

3. T. M. Proctor, "More Recent Improvements on the NBS Conical Transducer," J. Acoustic Emission, Vol. 5, No. 4, pp. 134-142 (1986).

4. N. N. Hsu, "Dynamic Green's Functions of an Infinite Plate -- A Computer Program", NBSIR 85-3234 (1985).

5. L. Mordfin, "International Standards for Nondestructive Testing," NBS J. Res. (to be published).

6. F. R. Breckenridge, C. E. Tschiegg, and M. Greenspan, "Acoustic Emission: Some Applications of Lamb's Problem," J. Acoust. Soc. Am., Vol. 57, No. 3, pp. 626-631 (1975).

7. F. R. Breckenridge, T. M. Proctor, N. N. Hsu, and D. G. Eitzen, "Some Notions Concerning the Behavior of Transducers," Progress in Acoustic Emission III, pp. 675-684 (The Japanese Society of NDI, 1986).

8. M. Greenspan, "The NBS Conical Transducer: Analysis," J. Acoust. Soc. Am. 81, No. 1, pp. 173-183 (1987).

9. N. N. Hsu and D. G. Eitzen, "Point Source/Point Receiver Ultrasonic Wave Speed Measurement," Proceedings of 1987 Ultrasonic International Conference, London, England (to be published).

10. H. T. Yolken, editor, "Nondestructive Evaluation Technical Activities 1986," NBSIR 86-3434 (1987).

11. D. A. Hutchins, K. Lundgren, R. P. Young, N. N. Hsu, "Laser Simulation of Buried AE Sources," J. Acoustic Emission, Vol. 5, No. 3, S29-S33 (July-Sept. 1986).

Standards for Industrial Radiography

R. C. Placious
Ionizing Radiation Division
Center for Radiation Research

Noteworthy progress has been achieved in the continuing effort to develop standards for quantitative assessment of x-ray image quality at very low and very high energies. The draft standard on image quality at high energies (4 to 24 MeV), which was disucssed in last year's report, has undergone two ballots in ASTM. The first ballot generated extensive revisions in the document in order to reconcile persuasive negative votes. Some additional revision is now under way in order to satisfy newly raised questions. The development of the procedure for assessing quality, the design of the image quality indicator, the establishment of the protocol for the round-robin test program, and the evaluation of the test results were all accomplished or arranged by NBS.

The draft standard on evaluating image quality at low energies, which is also primarily an NBS effort, is still under development. Revisions are being made to the test device and the protocol in order to correct inadequacies that surfaced in the last round-robin test series. This standard is geared towards laboratories involved in radiography of thin-walled metal structures and polymer-based composite materials.

NBS work on the characterization of x-ray sources for industrial radiography also advanced during the past year. Measurements made at NBS established

feasibility and formed the basis for a test protocol which was distributed to volunteers in other laboratories. Preliminary data from some of these laboratories revealed some discrepancies. NBS proposed and arranged a plan to redefine the responsibilities of each volunteer group. This revision is designed to isolate the cause of the discrepancies. These revised tests are presently under way.

The significance of this effort is considerable. The expertise to measure the x-ray spectrum, for each kilovoltage addressed in the protocol, exists at NBS. Therefore, once a standardized procedure is adopted, it will be a straightforward matter to completely characterize x-ray beams over the entire range from 50 to 300 kV at 50-kV intervals.

A new effort was initiated to develop a method to measure total (not just geometric) radiographic unsharpness.

Standards for Real Time Radiology

T. A. Siewert
Fracture and Deformation Division
Institute for Materials Science and Engineering

R. C. Placious
Ionizing Radiation Division
Center for Radiation Research

A two-day workshop (April 2-3, 1987) was held at the NBS Boulder Laboratories to review NBS activities in real time radiology, obtain the opinions of producers and users on standards needed for this rapidly developing technology, and develop a prioritized list of these needs. This list of needs will guide the NBS standardization activities by identifying the topics which provide the most benefit to this industry.

The workshop consisted of several overview presentations to establish a background on standardization activities, presentations by various experts in the field that described their perceptions of the needs, and several discussion sessions where the attendees developed a list of needs and ranked the national needs for this industry. The 27 experts who were invited to this workshop identified: (1) radiation transfer standards, (2) source characteristics, and (3) quantification of image quality as the three most important topics for standardization efforts, in descending order of priority. The attendees showed a desire to assist in the standardization activities by volunteering to join a cooperative program. Copies of the workshop report are available from T. A. Siewert, National Bureau of Standards, Boulder, CO 80303; (303) 497-3523. Details of the workshop were also presented as an invited talk at the annual ASNT summer topical conference this year.*

*"A Look at the Long Term Role of NBS in Real Time Radiology Standards," R. C. Placious and T. A. Siewert, ASNT Topical Conference, August 13, 1987, Wilmington, Delaware.

Eddy Current Coil Characterization

T. E. Capobianco and F. R. Fickett
Electromagnetic Technology Division
Center for Electronics and Electrical Engineering

The purpose of this experiment is to investigate the causes of variations in eddy current probe sensitivity related to probe coil construction. Quantifying the effects of construction variations will help move eddy current probe design from a "black art" to a predictable engineering practice.

In the course of developing a draft standard for characterizing eddy current probes [1] we have devised a test method for predicting probe sensitivity. This test can determine sensitivity, but it provides no information as to the causes of sensitivity variations. The construction and characterization of a precision-wound, controlled-variation coil set has been undertaken to provide these answers. Some of the coil construction variables being investigated include: wire gauge; number of turns; coil winding cross section; ferrite permeability and diameter; and scatter winding compared to precise layup. We report the results to date from this ongoing program. More detailed information can be obtained in other publications [2].

Past attempts to specify probe performance have usually involved measure of a single electrical parameter such as the inductance. An example of the confusion that can arise from relying on such data for probe characterization is shown in Table I. Two coils with similar inductances (AR4, AR2) show very different ΔZ responses (the parameter ΔZ_{Ti-Al} is described shortly). Conversely, coils with similar ΔZ responses (WG2, AR2) may have very different inductances.

The magnitude of the impedance change observed when the probes are in contact first with titanium and then aluminum (ΔZ_{Ti-Al}) is the test selected as the sensitivity criterion of the coils. When compared to used probes taken from service, this measurement, as well as ΔZ measurements on electric-discharge-machined (EDM) notches, corresponded very well to reported sensitivities. ΔZ_{Ti-Al} was chosen because it avoids the questions and uncertainties associated with the notched artifact standards such as notch size certification and the effects of machining damage to the bulk material when the notch is made. More importantly, in our earlier work we found that repeat ΔZ_{Ti-Al} measurements have an order of magnitude less variability than repeat ΔZ measurements made on EDM notches.

Table I. Coil Inductance on Al compared to ΔZ_{Ti-Al}

Coil Name	L_{Al}	ΔZ_{Ti-Al}
AR4	75.3	7.73
AR2	75.6	4.66
WG3	64.1	5.34
NT3	81.1	5.90
WG2	66.9	4.62
AR2	75.6	4.66

To date, three parts of the set have been completed. The construction variables represented are number of turns (NT1 to NT4), aspect ratio (AR1 to AR4), and wire gauge (WG1 to WG4). Thus, twelve coils have been wound and each coil has been characterized. Construction parameters of the coils are given in Table II.

Table II. Coil Construction Parameters

Coil Subset	Variation	Constants
NT1-4	Total number of turns	Ferrite permeability and dimensions, wire gauge.
AR1-4	Aspect ratio or turns/layer and number of layers	Ferrite permeability and dimensions, wire gauge, total number of turns.
WG1-4	Wire gauge	Ferrite permeability and dimensions, total number of turns.

The ΔZ_{Ti-Al} sensitivity of the coils was plotted as a function of each of the construction variables. With the help of Dom Vecchia of the Statistical Engineering Division, software employing a least squares method was used to find the best functional fit to the data. Figure 1 shows an example of an empirical fit of ΔZ_{Ti-Al} versus the aspect ratio (AR coil set). This process was repeated for the NT (number of turns) and the WG (wire gauge) coil sets. Several different functional forms, including linear, logarithmic, exponential, power, and quadratic fits, were evaluated for closeness of fit.

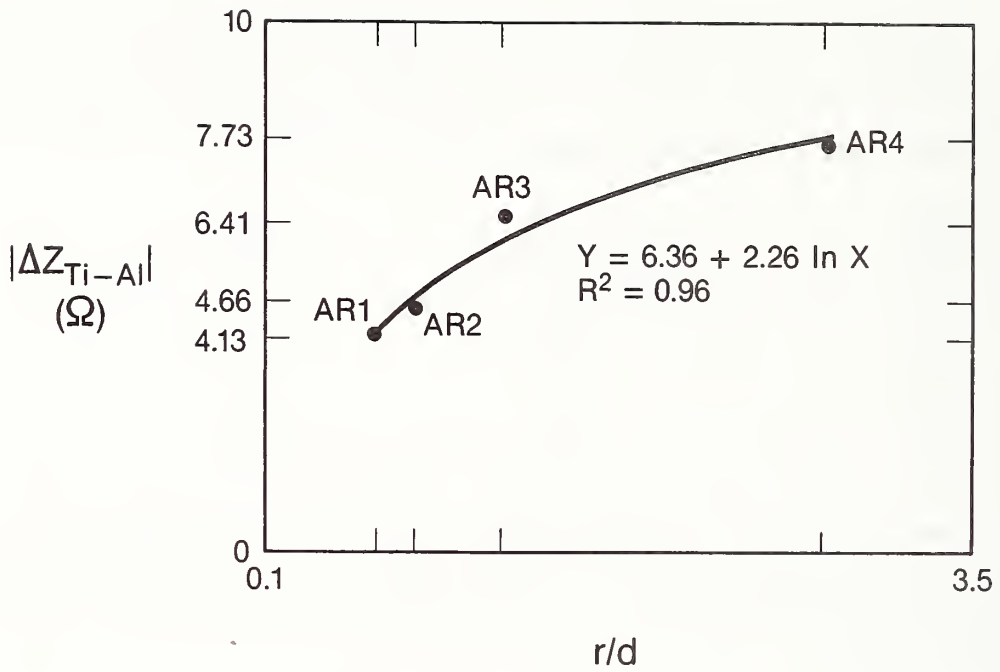


Figure 1. Sensitivity versus aspect ratio and the empirically determined function.

A common statistical model was derived from the functional form chosen for each individual parameter. One basic assumption of this model is that the effects of the construction parameters are additive; that is, they do not interact, which means there are no product terms in the combined model. Other elements of the model are based on observations of unpredictable shifts in the sensitivity data, which appear as offsets of some data points from the fitted curve (Figure 1).

Assuming that the effects of AR, NT, and WG are additive, ΔZ may be considered to be a composite of the one-at-a-time relationships and the effects of coil inhomogeneity as follows:

$$\Delta Z_{ij} = \beta_0 + \beta_1(WG)_i + \beta_2(WG)_i^2 + \beta_3(\ln NT)_i + \beta_4(\ln AR)_i + C_i + e_{ij}$$

where i = 1-12 (coils) and j = 1-4 (repeat measurements). ΔZ_{ij} is the observed sensitivity (ohms) for the j-th repeat measurement on the i-th coil; $(WG)_i$, $(\ln NT)_i$, and $(\ln AR)_i$, are the construction factors associated with the i-th coil. C_i is a random shift on the i-th coil corresponding to the inhomogeneity among the coils. The factor e_{ij} is a random measurement error in the j-th reading on the i-th coil.

These initial results on quantifying construction parameter variations are quite promising. In the future this type of empirical modeling of construction effects will be extended to other parameters. This will allow users and manufacturers of eddy current probes to specify the kind of transducer most suited to the needs of any inspection situation.

One fact to keep in mind is that every data point represents the construction of a coil, making the acquisition of this information a slow and tedious process.

References:

1. T. E. Capobianco and F. R. Fickett, "A Proposed Military Standard for Commercial Eddy Current Probes Based on Performance Characterization," Proceedings of the 35th Defense Conference on Nondestructive Testing, pp. 134-141 (Ogden Air Logistics Center, Hill AFB, Utah, 1987).

2. T. E. Capobianco and D. Vecchia, "Coil Parameter Influence on Eddy Current Probe Sensitivity," Review of Progress in Quantitative Nondestructive Evaluation, vol. 7, in press.

Capacitive Array Sensor Applied to NDE

P. J. Shull
Fracture and Deformation Division
Institute for Materials Science and Engineering

J. C. Moulder
Center for Nondestructive Evaluation
Iowa State University

M. Gimple and B. A. Auld
Edward L. Ginzton Laboratory
Stanford University

The major objectives of this program are divided into two categories: (1) design and characterization of a capacitive array probe as an NDE sensor and (2) investigation of possible applications for the capacitive sensor. This type of probe, which was previously used as a robotic sensor at Stanford University [1], has the unique advantage over inductive sensors in its ability to interrogate dielectric surface and subsurface features, and conductor surface features.

The capacitive array sensor developed at NBS and Stanford University consisted of an array of basic capacitive elements. The basic element is essentially a parallel plate capacitor that has been unfolded such that the two electrodes lie in the same plane. A voltage is impressed between the two electrodes and the resultant current flow between these electrodes is measured. The work piece is placed in the lower half-space of the probe. For a dielectric, the current is enhanced, thus increasing the output signal. For a conductor, which is grounded, the current flow between the electrodes is reduced because of a parallel path provided by the grounded metal.

To reduce the effects of liftoff we operated the probe in a differential geometry. The probe was used in this mode to detect variations in the sample that are small in size compared with the probe's sensing area. Examples of such variations are edges or steps and flaws, such as voids or cracks.

Using printed circuit board technology a miniature differential capacitive probe was built with a sensing area of 0.75 mm x 2.5 mm and with each of the three electrodes 0.125 mm x 2.5 mm.

To fully understand the nuances of the probe response we designed a series of tests on well characterized flaws, both surface and subsurface. Series of notches were made on the surfaces of both a conductor and insulators with depths ranging from 0.025 mm to 0.5 mm and with varying widths. Figure 1 shows the effects of notch depth as the probe is scanned across the surface. Also determined from these specimens were the effects of notch width, liftoff, variation of dielectric constant, sample grounding, and probe shielding.

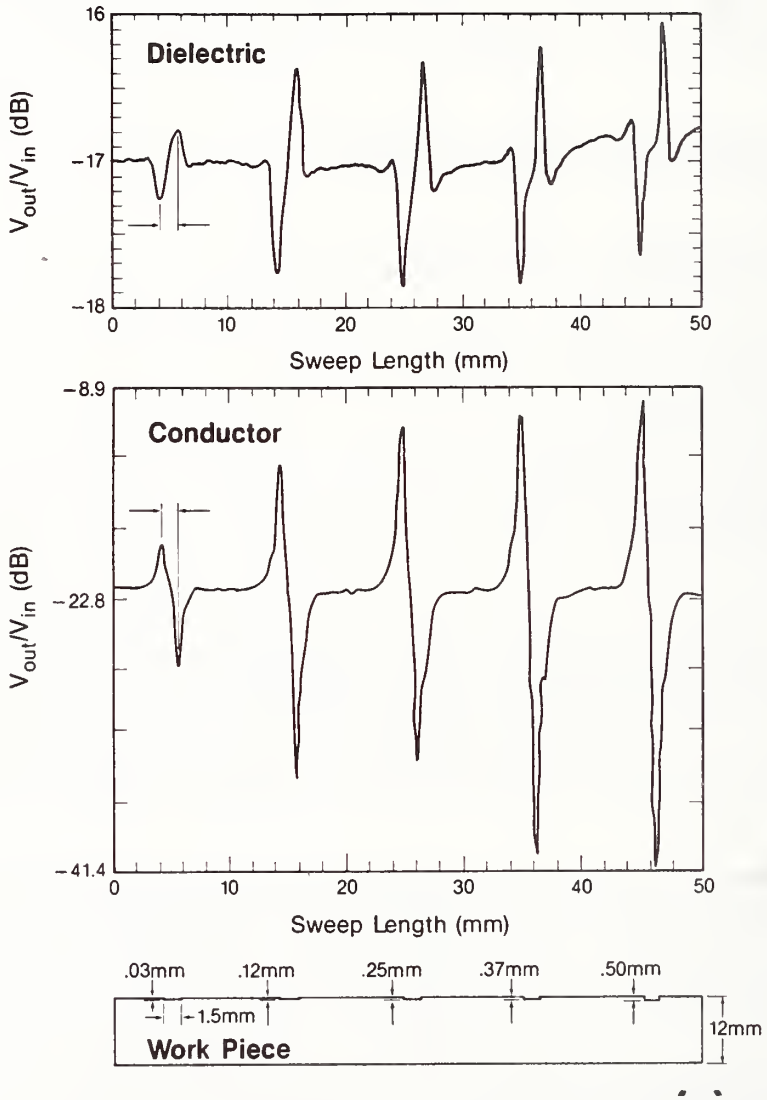


Figure 1. Capacitive array probe response to a series of surface notches in a dielectric and a conductor.

The probe response to subsurface flaws in dielectrics was characterized by a series of notches whose depth from the surface ranged from 0.3 mm to 4.0 mm, Figure 2. In addition to detecting the 4.0-mm deep flaw we discerned small machining defects on the edges of the 4.0-mm and 0.3-mm flaws.

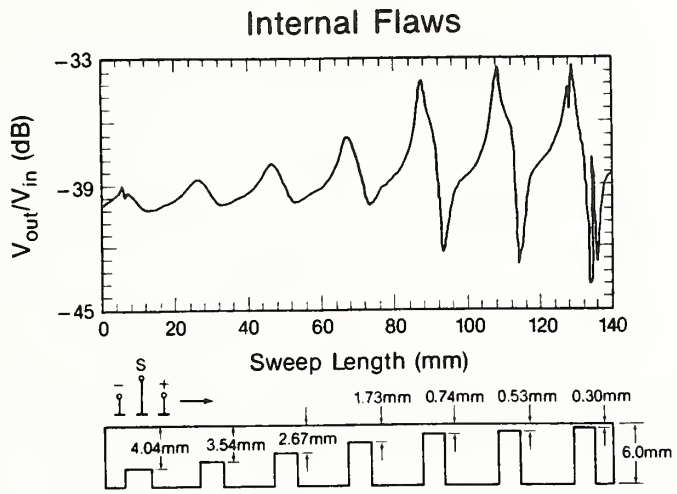


Figure 2. Capacitive array probe response to a series of subsurface notches in a dielectric.

Capacitive array modeling capability was developed by Paul Heyliger at NBS Boulder using finite element methods [2]. To date the results qualitatively agree with our experimental work at NBS and with Mark Gimple's work at Stanford using finite difference methods. This capability has been very useful to predict and interpret experimental results. An example for a notch in an insulator and a conductor is shown in Figures 3 and 4.

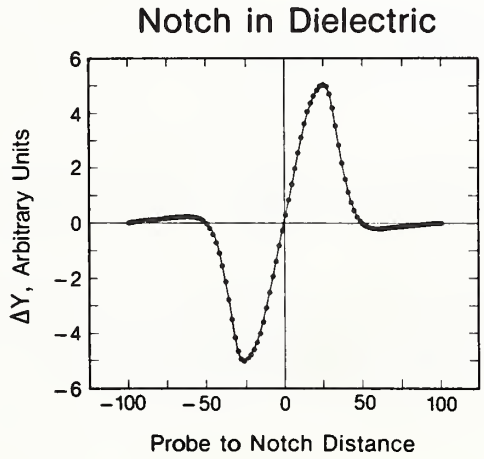


Figure 3. Calculated response to a notch in a dielectric.

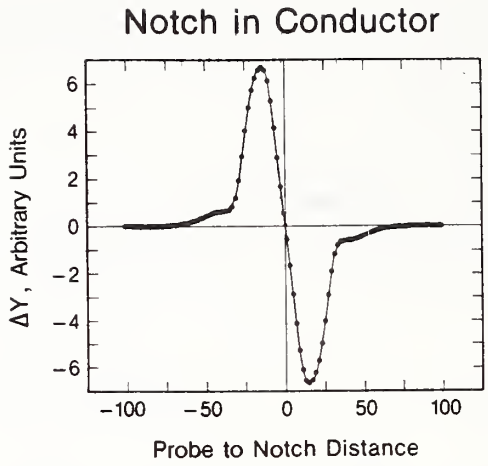


Figure 4. Calculated response to a notch in a conductor.

To determine the potential capabilities of the capacitive array sensor and future direction of this project, we performed preliminary studies of a variety of applications.

(1) Ceramics - We observed signals from a through crack (40 μm in width) in alumina, variations in the response from changes in thickness, and porosity of thermal barrier coatings. We also measured a crack in glass that was 4.0 μm wide.

(2) Dielectric Constant Monitoring - We observed two processes. The curing of 5-minute epoxy and the recrystallization of phenyl salicylate.

(3) Subsurface Flaws in Insulators - In plexiglass a flat disc-shaped flaw 5 mm in diameter, 0.1 mm in width and approximately 2 mm below the surface was interrogated.

In these applications, the flaws or processes were clearly observed with a signal to noise ratio of greater than 10.

References:

1. M. A. Gimple and B. A. Auld, "Capacitive Arrays for Robotic Sensing," Review of Progress in Quantitative NDE, Vol. 6 (Plenum, New York, 1987), p. 737.

2. P. Heyliger, J. C. Moulder, M. Gimple, B. A. Auld, and P. J. Shull, "Numerical Modeling of Capacitive Array Sensors Using the Finite Element Method," Review of Progress in Quantitive NDE, Vol. 7, in press.

Magnetic Methods and Standards for NDE

L. J. Swartzendruber
Metallurgy Division
Institute for Materials Science and Engineering

There is a considerable variety of magnetic methods available for use in nondestructive evaluation of both defects and material properties. Some of these, such as magnetic particle inspection, magnetic flux leakage testing, and magnetic permeability measurement, are widely used in industry; the first two for defect detection, and the latter for property determinations such as ferrite content in stainless steel welds. A number of methods have been used on a limited scale but still require development to make them more reliable and more widely applicable. These include Barkhausen noise, magnetoacoustic emission, magnetomechanical damping, and AC magnetometry. Finally, magnetic property measurements, including saturation magnetization, coercivity, and initial permeability, provide a wealth of information concerning material properties that has barely begun to be exploited.

Magnetic particle inspection is the most used of all nondestructive evaluation methods. It is probably also the most abused of all testing methods. Hence, this project has placed considerable effort on improvement of magnetic particle inspection procedures and standards. Extensive effort in the future on magnetic property measurement and its relationship to material properties is planned using new facilities being developed in the Magnetic Materials Group of the Metallurgy Division.

One of the primary standard artifacts used in magnetic particle inspection, both for testing of the magnetic particles used in the process and for testing the overall inspection process, has been the so-called Ketos ring. This is simply a circular ring made of a specific tool steel with specified dimensions and with twelve holes drilled at various depths. Based on measurements performed at NBS and elsewhere, it was recently realized that a wide variation in properties, and hence in test results, are exhibited by these rings. A new heat treatment has been selected for these rings which reduces their variability to an acceptable level. This new heat treatment is being incorporated into the ASTM magnetic particle recommended practice (ASTM E 709) through the efforts of ASTM Committee E7.03 and into Military Standard 1949 through our cooperative effort with the Army Materials Technology Laboratory.

Development of an improved heat treatment for the test ring can be considered only a temporary solution to providing an adequate test for proving magnetic particles and for testing the overall procedure used in the test of a particular part. To provide for a better standard we are currently coordinating an ASTM round-robin test on artificial flaw standards. These flaw standards consist of thin (25 and 50 μm) pieces of mild steel with chemically milled flaws. The flaw geometry is either in the shape of a circle or of a bar. The round robin is designed to relate the results observed on the flaw standards to results on the test ring and to results on actual parts using developed procedures. Based on the outcome of this round robin, and on further tests at NBS, the possibility of issuing such flaw standards as Standard Reference Materials will be considered. Insuring adequate part magnetization has been a difficult problem since the inception of magnetic particle testing. Our

results have shown that, in most cases, it is sufficient to measure the tangential component of the magnetic field at the part surface. Although this is an easily performed measurement in theory, it has proven difficult to put into practice because the requirements for the measurement instrument, for the placement of the measurement probe, and the exact values required for the magnetic field have been sources of confusion and controversy. The recent availability of suitable and inexpensive Hall-effect Teslameters, and the incorporation of better measurement instructions into the various standards, can be expected to greatly increase the use of tangential magnetic field measurements as a tool in magnetic particle testing. The combined use of such field measurement and suitable artificial flaw standards has the potential of greatly increasing the reliability of magnetic particle testing. This is of considerable industrial importance because economic forces can be expected to greatly increase the use of magnetic particle testing in the future.

During the year equipment was assembled to provide for automated measurement of magnetic leakage fields from flaws of importance for magnetic particle testing. Equipment has also been assembled to enable Barkhausen noise to be measured and analyzed under the control of a microcomputer. During the coming year this equipment will be incorporated into systems and software developed to control the experiments and analyze the results obtained.

Development of Leak Standards and Calibration Facilities

C. D. Ehrlich and C. R. Tilford
Temperature and Pressure Division
Center for Basic Standards

With the introduction last year of a Special Test Service for helium permeation leak artifacts in the range 10^{-8} to 10^{-11} mol/s (2×10^{-4} to 2×10^{-7} atm cc/s @ 0 °C), activities this year have focused on providing this service in an effective and timely manner. Thus far nine leak artifacts have been received into the Special Test Service, and at least seven will have been evaluated and returned to the customer by the end of the fiscal year. This includes two artifacts in use at Three Mile Island. The Primary Leak Standard has been improved with a new manifold system that increases the number of artifacts that can simultaneously be mounted for evaluation, without the necessity of opening the vacuum system, from two to six. This manifold, with dual pumping systems and a residual gas analyzer, provides the flexibility to measure flow rates from one artifact while performing additional experiments with another.

A more detailed investigation into the properties of the helium permeation artifact itself has yielded a much better understanding of the fundamental processes governing the flow rate, and of the proper ways in which to use these leaks. This has also resolved a discrepancy that exists in the recent literature concerning the functional dependence of flow rate on various parameters. Of primary issue is the time response of the leak rate from an artifact to thermal fluctuations. On the one hand, it is well-known that the leak rate from an artifact can take several days to stabilize when the leak reservoir is first filled with helium. On the other hand, leak rates from artifacts are known to stabilize much more quickly when subjected to discrete

changes in temperature, even though the helium reservoir pressure changes accordingly. Our experiments support part of the literature on the permeation of gases through vitreous glasses; that it is the helium concentration gradient, and not pressure gradient, that governs the flow rate.

The development and testing of a temperature-controlled leak artifact has shown considerable promise toward providing a stable artifact for both laboratory and field use. During observations made in the truck bay serving the two reactors at Three Mile Island, using uninsulated leak artifacts, it became clear that large thermal fluctuations in the truck bay could lead to serious misuse of the leak artifacts. Insulating the artifacts helps, but an artifact that is thermally regulated offers a greater degree of stability, and reduces the associated uncertainties. We have tested two different temperature controller designs, one commercial and the other developed at NBS. The NBS controller was found to give better regulation about a given temperature (10 mK vs. 200 mK); however, both were found to yield approximately the same shift in artifact temperature for a fixed change in the temperature of the environment (0.3 K for an environmental change of 37 K, or 0.8%). After final laboratory evaluation at NBS, an artifact of this design will be sent to TMI for actual field testing.

The calibration schedule for the new test service was badly delayed by instabilities in a number of leaks belonging to outside customers as well as NBS. After several months of testing, the problem was traced to an improperly installed valve on the leaks which allowed helium to leak out during heating and cooling cycling. Since this could cause large errors with time the manufacturer was advised of the problem. He has modified his assembly procedure and repaired the offending leaks.

Studies are being planned to evaluate the uncertainties associated with using the Leak Comparator, in conjunction with leak artifacts that have been measured on the Primary Leak Standard, to perform the entire calibration of customer leaks.

A Standard Leak Calibration Workshop, cosponsored by Sandia National Laboratories, was held at NBS on September 21-22, 1987, to provide a forum for sharing our findings with the user community and to learn about other new developments in this area. Besides talks directly related to leak calibration and design, the workshop featured speakers from the nuclear transportation, electronics, refrigeration, and leak detector manufacturing industries. Representatives from nuclear and military contracting organizations, as well as national standards organizations including the American Society for Testing and Materials, American Vacuum Society, American Society for Nondestructive Testing, American National Standards Institute and the Department of Defense, also presented talks outlining their organizations' activities.

Thermographic Techniques for Defect Characterization and Crack Growth Evaluation in Metals and Composites

Y. Huang[1], R. J. Fields, and G. E. Hicho
Fracture and Deformation Division
Institute for Materials Science and Engineering

[1]Guest Scientist, Institute of Metal Research, Academia Sinica, Shenyang, China

The objective of this project is to evaluate the sensitivity of thermographic techniques in the detection and characterization of flaws and flaw growth in materials of commercial importance. The local heat flow through a material body containing a flaw depends upon the characteristics of the flaw. In the presence of a temperature gradient (imposed by transient heating or cooling), a nonuniform surface temperature distribution will arise due to a subsurface flaw. Thermography provides a non-contacting means of imaging this distribution in real time. Its sensitivity to various defect parameters and its ability to detect small flaws were investigated. Furthermore, if a body containing cracks is load cycled, the crack tip regions will emit heat. The correlation between crack growth rate and thermal emission was also determined.

The effectiveness of thermography as a nondestructive evaluation technique was investigated for lamination defects in organic matrix composites and for fatigue crack growth in a superalloy. These studies indicate that thermography is extremely sensitive to lamination defects and may be optimized for on-line quality control monitoring of these types of defects in composite substructures. These studies also demonstrate the ability of thermography to detect very small fatigue crack growth rates and suggest the possible use of this technique in the field to nondestructively assess the growth rate of cracks in existing structures.

Detection of Lamination Defects:

The detection of lamination defects by thermography is based on measuring temperature differences in a composite subjected to a thermal gradient. The measured temperature distribution results from differences in thermal conduction around lamination defects. The variation of the heat flow through a sheet containing a subsurface defect depends on the location, orientation, geometry, and thermal properties of the defect.

Two types of composites were investigated: a model composite of stainless steel and polypropylene sheets separated by patches of teflon to simulate flaws, and commercial graphite/epoxy panels containing typical fabrication flaws.

To evaluate the sensitivity of the thermographic technique, a composite specimen was heated (or cooled) from behind while an infrared scanner, located in front, monitored the panel. Typical thermographic images of some flaws are shown in Figure 1. By varying the dimensions, locations, and other flaw parameters, the sensitivity of this technique was quantified. For example, the effect of defect depth below the surface on the temperature difference

between flawed and normal areas is shown in Figure 2. In this figure, the temperature difference is normalized by the average temperature of the flawed region. In Figure 3, the effect of defect thickness on the normalized temperature difference is shown.

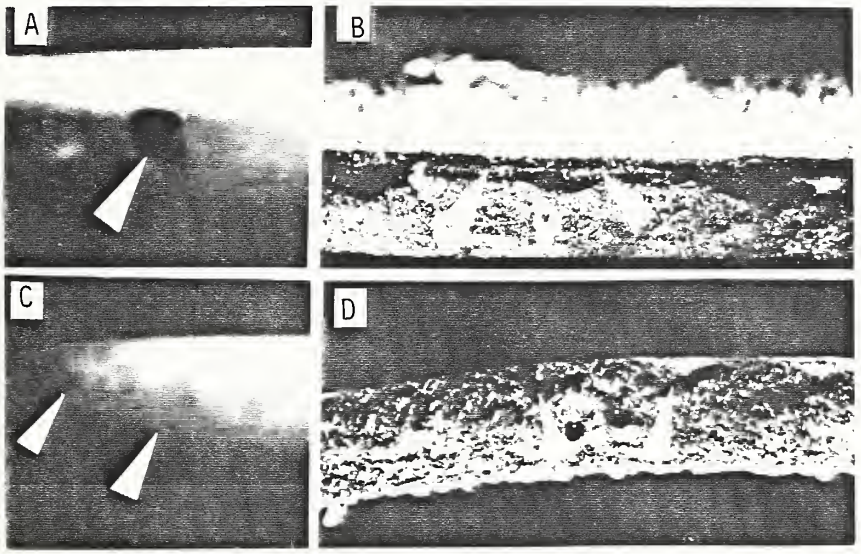


Figure 1. A and C - Thermograms of lamination defects in graphite/epoxy panels. B and D - Associated fractograms.

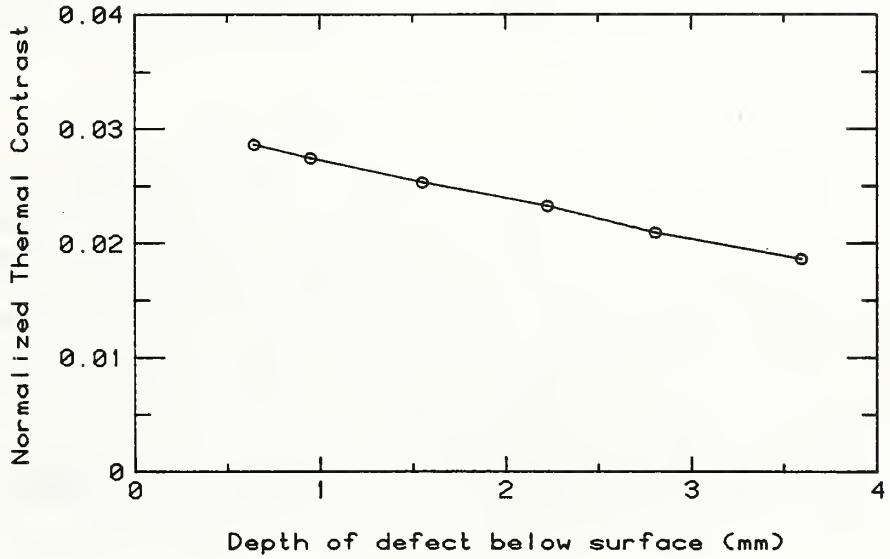


Figure 2. Effect of defect depth below surface on thermal contrast. The defect is 23 micrometers (0.001 in) thick.

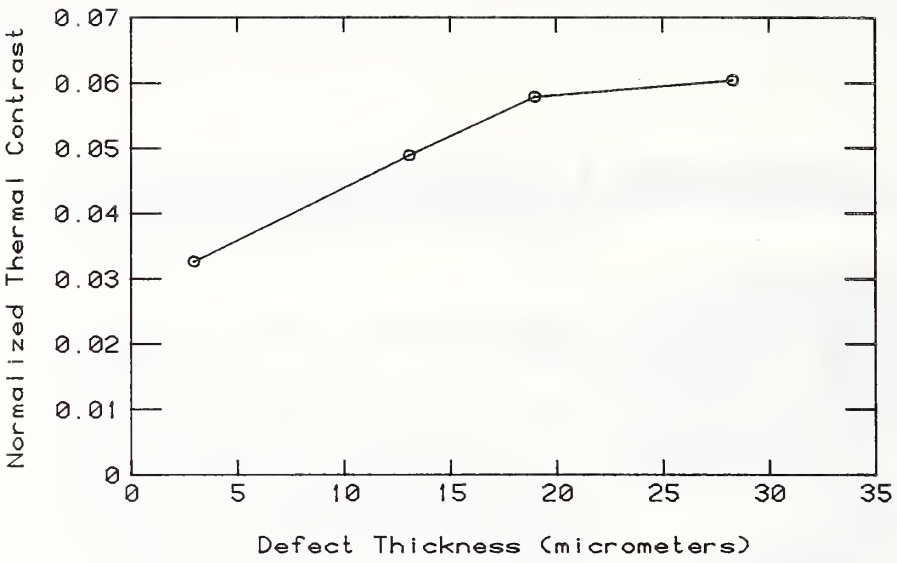


Figure 3. Effect of defect thickness on thermal contrast. The defect is located under a 1 mm sheet of polypropylene.

The results of this study indicate that thermography can detect flaws at the laminate structure interface. In general, it was found that the effect of a flaw on thermal response increases as the width and/or thickness of the flaw increases, and decreases as the depth of the flaw below the surface increases. The minimum area of a detected flaw in the graphite/epoxy panels was 0.97 mm^2 (0.0015 in^2) with the present experimental arrangements. Due to this high sensitivity and real-time capability, the technique appears to have good potential for on-line quality control of composite materials.

Measurement of Flaw Growth:

Growing fatigue cracks in a nickel-base superalloy were examined by thermography to determine the minimum detectable rate of growth and the correlation between thermal emission near the crack tip and crack growth rate. The experimental arrangements are shown in Figure 4. Noteworthy results are summarized in Figure 5. In this figure, the fatigue crack growth rate is plotted against the thermal emission, expressed as the total temperature rise ($\Sigma\Delta T$) summed over the plastic zone of the crack. A good correlation between thermal emission and crack growth may be noted. The resolution of the present thermographic set-up is about 10^{-5} mm/cycle, indicating a very high sensitivity to crack growth. Besides this sensitivity and good correlation, thermography offers noncontact sensing with real-time imaging. This suggests that thermography may be used in the field to nondestructively assess the growth rate of cracks in existing structures under real loading conditions.

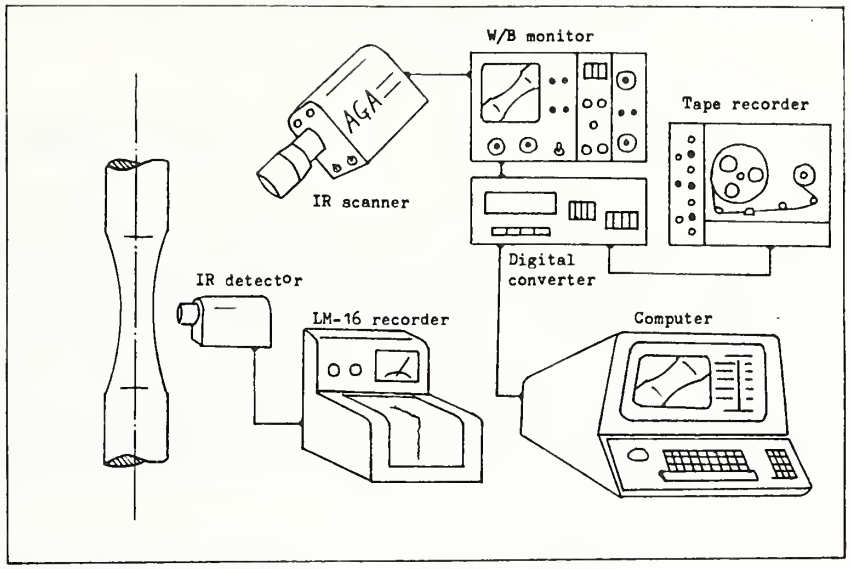


Figure 4. Infrared emission data acquisition system for fatigue testing.

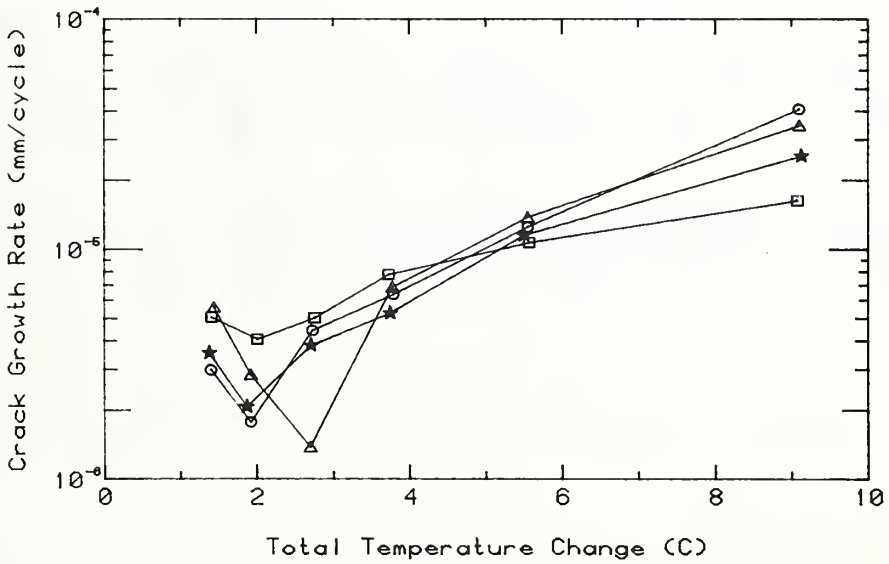


Figure 5. Total thermal emission from growing cracks for various integration times. Correlation becomes poor for growth rates much less than .01 μm/cycle.

Standard Test Methods for Characterizing Performance of Thermal Imaging Systems

J. Cohen
Radiometric Physics Division
Center for Radiation Research

Infrared thermography, the process whereby the normally invisible thermal radiation from an object is remotely detected and rendered into a visible display, is being applied extensively in industry for NDT purposes such as facility monitoring and detection of material defects, as well as for thermal design verification, in-process manufacturing control, and quality assurance. Thus, standard test methods for performance characterization of thermal imaging systems (TIS) are important, e.g., for instrument calibration and monitoring, for intercomparisons at the marketplace and in the laboratory, and for experimental design and feasibility studies.

The objective of this continuing project is to develop new (and improved) standard test methods for characterizing performance of TISs. The first such (ASTM) standard being developed addresses minimum resolvable temperature difference (MRTD), which relates to the system's capacity for distinguishing details in the imagery. The draft standard has been moving smoothly through the extensive ASTM regimen and early adoption as a standard is expected.

Work was initiated on the development of a new standard test method for a second performance measure, viz., minimum detectable temperature difference (MDTD), which is of particular importance to NDT of materials, as it relates to the TIS capacity to detect subsurface flaws such as voids and inclusions.

Two projects, both other-agency sponsored, have evolved from the present work. The objective of one (Army) is to provide consultation for an automated infrared inspection system for solder joint integrity in printed circuit boards; the objective of the other (Navy) is to assess testing methods for measuring TIS performance and to advise about ancillary instrumentation needs.

On invitation by the editor, a book chapter, "Fundamentals and Applications of Infrared Thermography for Nondestructive Testing," was written for the forthcoming volume of the International Advances in Nondestructive Testing (IANDT).

APPENDICES

A. NDE SEMINARS AT NBS

NDE Poster Session
January 5, 1987

Professor Subhendu K. Datta, University of Colorado
"Ultrasonic Characterization of Interfaces in Composites"
June 19, 1987

Dr. Alfred Van Clark, Fracture and Deformation Division, NBS
"Ultrasonic Monitoring of Texture and Formability"
June 29, 1987

Mr. Peter J. Shull, Fracture and Deformation Division, NBS
"Applications of Capacitative Array Sensors to Nondestructive Evaluation"
June 30, 1987

B. INVITED TALKS BY ONDE STAFF

"International Standards for Nondestructive Testing: What, Why, and How," L. Mordfin, Connecticut Valley Section, American Society for Nondestructive Testing, Hartford, Connecticut, November 11, 1986.

"Recent Advances at NBS in Standardizing NDT Measurements," L. Mordfin, Metropolitan New York-Northern New Jersey Section, American Society for Nondestructive Testing, Newark, New Jersey, November 12, 1986.

"International Standards for Nondestructive Testing," L. Mordfin, Hampton Roads Section, American Society for Nondestructive Testing, Hampton, Virginia, January 10, 1987.

"Intelligent Processing of Materials," H. T. Yolken, NBS Visiting Committee, Gaithersburg, Maryland, February 11, 1987.

"Intelligent Processing of Materials," H. T. Yolken, National Academy of Sciences Workshop on Needs and Opportunities in Materials Science and Engineering, Washington, D.C., March 7, 1987.

"The NBS-NDE Program," H. T. Yolken, NBS Workshop on Real-Time Radiology: Developing a Five-Year Plan for NBS Standardization Activities, Boulder, Colorado, April 2, 1987.

"National Bureau of Standards NDE Program," G. Birnbaum, 16th Symposium on Nondestructive Evaluation, San Antonio, Texas, April 22, 1987.

"Nondestructive Evaluation at the U.S. National Bureau of Standards," L. Mordfin, Korea Standards Research Institute, Taejon, Korea, May 6, 1987.

"Designing for Inspectability: Some General Principles," L. Mordfin, American Society of Mechanical Engineers, Fifth National Congress on Pressure Vessel and Piping Technology, San Diego, California, July 2, 1987.

"Recommendations for Future Research - Advanced Materials and Acoustic/Ultrasonics," H. T. Yolken, Workshop at Virginia Polytechnic Institute, Blacksburg, Virginia, July 14, 1987.

"Current and Future Activities in Materials Research and Nondestructive Evaluation at the National Bureau of Standards," H. T. Yolken, National Materials Advisory Board, National Academy of Sciences, Washington, D.C., September 17, 1987.

"Research on NDE at the National Bureau of Standards," H. T. Yolken, ISO/TC 164/SC4 on Fracture Mechanics, Gaithersburg, Maryland, September 21, 1987.

"Overview of the NBS Program in Nondestructive Evaluation," L. Mordfin, Standard Leak Calibration Workshop, Gaithersburg, Maryland, September 21, 1987.

G. PUBLICATIONS

Following is a partial listing of NBS reports and publications on NDE and related topics that have been issued since last year's Technical Activities report was prepared.

G. Alers and H. N. G. Wadley, "A Pulsed Laser/Electromagnetic Acoustic Transducer Approach to Ultrasonic Sensor Needs for Steel Processing," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 627-638 (Plenum, 1987).

W. E. Anderson, J. D. Ramboz, and A. R. Andrejka, "Final Report: Technical Contributions to the Development of Incipient Fault Detection/Location Instrumentation," NBSIR 86-3392 (April 1986).

Anon., "Nondestructive Evaluation," Cooperative Research Opportunities at NBS, NBS SP 723, pp. 44-45 (Dec. 1986).

Anon., "Process and Quality Control and Calibration Programs of the National Bureau of Standards; A Report by the National Bureau of Standards in Response to Public Law 99-574," NBSIR 87-3596 (1987).

M. Bello, "Researchers Study the Process to Improve Measurement Accuracy," NBS Research Reports, NBS SP 719, pp. 16-20 (July 1986).

G. Birnbaum, D. Nyyssonen, C. M. Vest, and T. V. Vorburger, "Optical Nondestructive Evaluation at the National Bureau of Standards," Holographic Nondestructive Testing: Status and Comparison with Conventional Methods, SPIE Vol. 604 (Intl. Soc. Optical Engrs., Bellingham, WA, 1986).

G. V. Blessing, "Review of IEEE 1985 Ultrasonics Symposium," Ultrasonics 24, No. 6, pp. 367-369 (Nov. 1986).

G. V. Blessing and D. R. Flynn, "Acoustic Evaluation of Thermal Insulation," U. S. Patent No. 4,672,851 (16 June 1987).

H. M. Bloom and N. R. Kuchar, editors, "A National Forum on The Future of Automated Materials Processing in U. S. Industry; The Role of Process Models, Artificial Intelligence and Computer Integration; Report of Workshop II," NBSIR 87-3544, 48 pp. (April 1987).

F. R. Breckenridge, T. M. Proctor, N. N. Hsu, and D. G. Eitzen, "Some Notions Concerning the Behavior of Transducers," Progress in Acoustic Emission III, pp. 675-684 (Japanese Society of NDI, 1986).

T. E. Capobianco, "Field Mapping and Performance Characterization of Commercial Eddy Current Probes," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 687-694 (Plenum, 1987).

T. E. Capobianco and Kun Yu, "Pickup Coil Spacing Effects on Eddy Current Reflection Probe Sensitivity," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 721-725 (Plenum, 1987).

N. J. Carino, M. Sansalone, and N. N. Hsu, "Flaw Detection in Concrete by Frequency Spectrum Analysis of Impact-Echo Waveforms," International Advances in Nondestructive Testing 12, W. McGonnagle, ed. (Gordon & Breach, New York, 1986).

Y. M. Chang and R. A. Grot, "Performance Measurements of Infrared Imaging Systems Used to Assess Thermal Anomalies," Thermal Imaging, SPIE Vol. 636 (Intl. Soc. Optical Engrs., Bellingham, WA, 1986).

A. V. Clark, Jr., A. Govada, R. B. Thompson, J. F. Smith, G. V. Blessing, P. P. Delsanto, and R. B. Mignogna, "The Use of Ultrasonics for Texture Monitoring in Aluminum Alloys," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1515-1524 (Plenum, 1987).

A. V. Clark, Jr., H. Fukuoka, D. V. Mitrakovic, and J. C. Moulder, "Ultrasonic Characterization of Residual Stress and Texture in Cast Steel Railroad Wheels," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1567-1575 (Plenum, 1987).

R. B. Clough, "The Energetics of Acoustic Emission Source Characterization," Materials Evaluation 45, No. 5, pp. 556-563 (May 1987).

R. B. Clough, F. S. Biancaniello, and H. N. G. Wadley, "Measurement of Fiber Fracture and Fiber-Matrix Interface Shear Strengths in Metal Matrix Composites," Proc. Nondestructive Testing and Evaluation of Advanced Materials and Composites Conference, pp. 91-102 (Nondestructive Testing Information and Analysis Center, San Antonio, TX, 1987).

R. B. Clough, H. N. G. Wadley, and F. S. Biancaniello, "Fracture Mechanisms in Single Crystal Composites," J. Acoustic Emission 5, pp. 569-570 (1986).

D. C. Creagh and J. H. Hubbell, "Problems Associated with the Measurement of X-ray Attenuation Coefficients. I. Silicon," Acta Cryst. A43, pp. 102-112 (1987).

S. K. Datta, R. E. Schramm, and Z. Abduljabbar, "Plate Modes Generated by EMATs for NDE of Planar Flaws," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 101-108 (Plenum, 1987).

S. K. Datta and H. M. Ledbetter, "Ultrasonic-Velocity Studies in Metal-Matrix Composites: Measurements and Modeling," Proc. Nondestructive Testing and Evaluation of Advanced Materials and Composites Conference, pp. 41-62 (Nondestructive Testing Information and Analysis Center, San Antonio, TX, 1987).

S. K. Datta, H. M. Ledbetter, Y. Shindo, and A. H. Shah, "Interface Effects on Attenuation and Phase Velocities in Metal-Matrix Composites," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1075-1084 (Plenum, 1987).

P. P. Delsanto, R. B. Mignogna, and A. V. Clark, Jr., "Ultrasonic Separation of Stress and Texture Effects in Polycrystalline Aggregates," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1533-1540 (Plenum, 1987).

E. Drescher-Krasicka, J. A. Simmons, and H. N. G. Wadley, "Guided Interface Waves," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1129-1136 (Plenum, 1987).

C. D. Ehrlich, "Present Status of the Leak Standards Program at the National Bureau of Standards," J. Vac. Sci. Technol. A5, p. 125 (Jan./Feb. 1987).

D. G. Eitzen and G. V. Blessing, "Ultrasonic Reference Blocks," Encyclopedia of Materials Science and Engineering, pp. 5181-5186 (Pergamon, 1986).

D. G. Eitzen, R. C. Placious, L. J. Swartzendruber, and L. Mordfin, "Nondestructive Evaluation of Monolithic Transportation Casks for Spent Nuclear Fuel," Report SAND86-7158, Sandia National Laboratories, Albuquerque, NM, 90 pp. (1987).

B. Fanconi, F. Wang, and R. Lowry, "Process Monitoring of Polymer Matrix Composites Using Fluorescent Probes," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1287-1295 (Plenum, 1987).

J. T. Fong, "Integration of Analysis and Databases for Engineering Decision Making," CIME - Computers in Mechanical Engineering 6, No. 2 (March 1986).

D. Gilsinn, T. Vorburger, L.-X. Cao, C. Giaque, F. Scire, and E.C. Teague, "Optical Roughness Measurements of Industrial Surfaces," Optical Techniques for Industrial Inspection, SPIE Vol. 665, pp. 8-16, (Intl. Soc. Optical Engrs., Bellingham, WA, 1986).

M. Greenspan, "Comments on 'Speed of Sound in Standard Air' J. Acoust. Soc. Am. 79 (1986)," J. Acoust. Soc. Am. 82, No. 1, pp. 370-372 (July 1987).

M. Greenspan, "The NBS Conical Transducer: Analysis," J. Acoust. Soc. Am. 81, No. 1, pp. 173-183 (Jan. 1987).

J. H. Hubbell, H. M. Gerstenberg, and E. B. Salomon, "Bibliography of Photon Total Cross Section (Attenuation Coefficient) Measurements 10 eV to 13.5 GeV," NBSIR 86-3461, 100 pp. (Oct. 1986).

J. H. Hubbell, "Radiation Gauging," Encyclopedia of Materials Science and Engineering, pp. 4040-4042 (Pergamon, 1986).

N. N. Hsu, "Plate Green's Function - A Computer Program and Its Application to Acoustic Emission," J. Acoustic Emission 5, No. 3, p. S28 (July-Sept. 1986).

D. A. Hutchins, K. Lundgren, R. P. Young, and N. N. Hsu, "Laser Simulation of Buried AE Sources," J. Acoustic Emission 5, No. 3, pp. S29-S33 (July-Sept. 1986).

L. J. Inglehart, J. Jaarinen, P. K. Kuo, and E. H. LeGal LaSalle, "Probing Through the Gas-Solid Interface with Thermal Waves: A Study of the Temperature Distribution in the Gas and in the Solid," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 263-270 (Plenum, 1987).

M. P. Jones and G. V. Blessing, "Ultrasonic Evaluation of Spray-Dried Ceramic Powders During Compaction," in Proc. of the Second International Symposium on the Nondestructive Characterization of Materials, Montreal, Canada, (July 21-23, 1986).

A. H. Kahn and H. N. G. Wadley, "Application of Eddy Current Methods to the In-Process Measurement of Temperature Distributions," Proceedings of Aluminum Association Workshop on Sensors, May 13-14, 1986, Atlanta, GA, pp. 129-151 (Aluminum Association, Washington, D.C.).

R. D. Kriz and H. M. Ledbetter, "Elastic Representation Surfaces of Unidirectional Graphite-Magnesium Composites," Proc. Nondestructive Testing and Evaluation of Advanced Materials and Composites Conference, pp. 63-76 (Nondestructive Testing Information and Analysis Center, San Antonio, TX 1987).

P. J. Lamperti, T. P. Loftus, and R. Loevinger, "Calibration of X- and Gamma-Ray Measuring Instruments at NBS," NBS Internal Report (April 1987).

R. D. Larrabee and M. I. Bell, editors, "Nondestructive Evaluation Activities in the Semiconductor Materials and Processes Division, NBSIR 86-3495, 32 pp. (Dec. 1986).

B. R. Lawn and C. J. Fairbanks, "Toughness and Flaw-Responses in Nontransforming Ceramics: Implications for NDE," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1023-1032 (Plenum, 1987).

J. R. Lawson, W. D. Walton, and D. D. Evans, "Measurement of Drop Size and Velocity in Sprinkler Sprays," NBSIR 87-3583 (1987).

K. E. McCulloh, C. R. Tilford, C. D. Ehrlich, and F. G. Long, "Low-Range Flowmeters for Use with Vacuum and Leak Standards," J. Vac. Sci. Technol. A 5, p. 376 (May/June 1987).

L. Mordfin, editor, "NDE Publications: 1984," NBSIR 87-3552, 31 pp. (April 1987).

L. Mordfin, "Advances Toward the Standardization of Residual Stress Measurements," Advances in Surface Treatments, Vol. 4, Residual Stresses, A. Niku-Lari, ed., pp. 341-345 (Pergamon, 1987).

J. C. Moulder, P. J. Shull, and T. E. Capobianco, "Uniform Field Eddy Current Probe: Experiments and Inversion for Realistic Flaws," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 601-610 (Plenum, 1987).

Tinh Nguyen and A. Rosencwaig, "Thermal-Wave Microscopy," Chemtech 16, pp. 634-640 (American Chemical Society, 1986).

Tinh Nguyen, E. Byrd, and A. Tsao, "Characterization of Epoxy Coatings on Cold-Rolled Steel by Reflection/Absorption Fourier Transform Infrared Spectroscopy, I. Effects of Film Thickness and Angle of Incidence," J. Appl. Polym. Sci. 32, p. 6339 (1986).

Tinh Nguyen and E. Byrd, "Application of Reflection/Absorption Fourier Transform Infrared Spectroscopy for Studies of Degradation of Coatings on Steel," J. Coatings Tech. 59, p. 748 (1987).

Tinh Nguyen, D. Bentz, and E. Byrd, "Quantitative Studies of Coatings on Steel by Reflection/Absorption Fourier Transform Infrared Spectroscopy," Proc. Am. Chem. Soc. Annual Meeting, Sept. 1986, pp. 665-669 (1986).

Tinh Nguyen and E. Byrd, "Reflection/Absorption Fourier Transform Infrared Spectroscopy of the Degradation of Protective Coatings on Mild Steel," Advances in Organic Coatings Science and Technology 9, A.V. Patsis, ed., pp. 178-190 (1987).

D. Nyyssonen, "Linewidth Calibration for Bright-Chromium Photomasks," NBSIR 86-3357 (May 1986).

S. P. Pessiki and N. J. Carino, "Measurement of the Setting Time and Strength of Concrete by the Impact-Echo Method," NBSIR 87-3575, 121 pp. (July 1987).

M. T. Postek, "Nondestructive Submicron Dimensional Metrology Using the Scanning Electron Microscope," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1327-1338 (Plenum, 1987).

C. Presser, A. K. Gupta, R. J. Santoro, and H.G. Semerjian, "Laser Diagnostics for Characterization of Fuel Sprays," Proc. 5th Int. Congress on Appl. of Lasers and Electro-Optics (ICALEO 1986) Vol. 58, pp. 160-167, Arlington, VA (1986).

C. Presser, A. K. Gupta, H. G. Semerjian, and R. J. Santoro, "Droplet/Air Interaction in a Swirl-Stabilized Spray Flame," Proc. 2nd ASME/JSME Therm. Eng. Joint Conf., Vol. 1, pp. 73-83, ASME, New York, NY (1987).

T. M. Proctor, Jr., "More Recent Improvements on the NBS Conical Transducer," J. Acoustic Emission 5, No. 4, pp. 134-142 (Oct.-Dec. 1986).

W. Sachse and N. N. Hsu, "Ultrasonic Transducers," Encyclopedia of Materials Science and Engineering (Pergamon, 1986).

E. B. Salomon and J. H. Hubbell, "Critical Analysis of Soft X-ray Cross Section Data," Nuclear Instruments and Methods in Physics Research A255, pp. 38-42 (1987).

M. Sansalone, N. J. Carino, and N. N. Hsu, "A Finite Element Study of Transient Wave Propagation in Plates," J. Res. NBS 92, No. 4, pp. 267-278 (July-Aug. 1987).

M. Sansalone, N. J. Carino, and N. N. Hsu, "A Finite Element Study of the Interaction of Transient Stress Waves with Planar Flaws," J. Res. NBS 92, No. 4, pp. 279-290 (July-Aug. 1987).

M. Sansalone, N. J. Carino, and N. N. Hsu, "Finite Element Studies of Transient Wave Propagation," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 125-133 (Plenum, 1987).

M. Sansalone, N. J. Carino, and N. N. Hsu, "A Finite Element Study of the Stress and Displacement Fields Produced by Point Impact," J. Acoustic Emission 5, No. 3, pp. S24-S27 (July-Sept. 1986).

R. E. Schramm and T. A. Siewert, "Sizing Canted Flaws in Weldments Using Low-Frequency EMATs," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1731-1736 (Plenum, 1987).

T. A. Siewert and R. C. Placious, "Summary of Workshop on Real-Time Radiology: Establishing a 5-Year Plan for NBS Standardization Activities, April 2 and 3, 1987," 13 pp. (Office of Nondestructive Evaluation, 1987).

T. A. Siewert and R. E. Schramm, "Compressive Stress Effects on the Ultrasonic Detection of Cracks in Welds," Review of Progress in Quantitative Nondestructive Evaluation 6B, pp. 1593-1600 (Plenum, 1987).

F. J. Shorten, editor, "NBS Reactor: Summary of Activities July 1985 through June 1986," NBS TN 1231, 164 pp. (1987).

P. J. Shull, T. E. Capobianco, and J. C. Moulder, "Design and Characterization of Uniform Field Eddy Current Probes," Review of Progress in Quantitative Nondestructive Evaluation 6A, pp. 695-703 (Plenum, 1987).

T. V. Vorburger, D. E. Gilsinn, E. C. Teague, C. H. W. Giaque, F. E. Scire, and L. X. Cao, "The Wind Tunnel Model Surface Gauge for Measuring Roughness," NBSIR 87-3586 (1987).

H. N. G. Wadley, "Sensors for Intelligent Processing of Materials," J. Metals 38, No. 10, pp. 49-53 (Oct. 1986).

H. N. G. Wadley, S. J. Norton, F. Mauer, and B. Droney, "Ultrasonic Measurement of Internal Temperature Distribution," Phil. Trans. R. Soc. Lond. A 320, pp. 341-361 (1986).

H. T. Yolken, editor, "Nondestructive Evaluation, Technical Activities 1986," NBSIR 86-3434, 77 pp. (Jan. 1987).

H. T. Yolken and L. Mordfin, "Automated Processing of Advanced Materials," Standardization News 14, No. 9, pp. 32-35 (ASTM, Oct. 1986).

M. Young, "Scratch-and-Dig Standard Revisited," Applied Optics 25, No. 12, pp. 1922-1928 (June 15, 1986).

D. AWARDS AND APPOINTMENTS

IR-100 Awards

Two of six coveted IR-100 awards won by NBS this year were for NDE-type sensors for monitoring materials processing. One, developed by Drs. Melvin Linzer and Haydn Wadley of the Metallurgy Division, in a collaborative research effort with the American Iron and Steel Institute, Argonne National Laboratory and Magnaflux Corporation, has resulted in the development of the first commercial sensor to detect and measure internal porosity and flaws in hot steel during processing. The sensor is an ultrasonic transducer device that utilizes a zirconia buffer rod to insulate the transducer from the hot surface, and liquid glass provides the acoustic coupling between the buffer rod and the steel.

The second award-winning sensor, also ultrasonic, monitors the quality of ceramic powders during compaction, prior to sintering. This development is the result of a collaboration between Martin Jones of the Ceramics Division (now with Alcoa Research Labs) and Dr. Gerald Blessing of NEL's Automated Production Technology Division. The velocity and the attenuation of ultrasonic signals passing through the compact, while it is in the mold, can be related to critical processing parameters such as density, porosity, moisture and composition, providing a basis for automated control of the process.

These awards bring to six the number of IR-100 awards received by NBS for Nondestructive Evaluation Program activities. The four previous awards were for the development of new standards and techniques for nondestructive evaluation.

Allen V. Astin Measurement Science Award

The Astin Award recognizes outstanding achievement in the advancement of measurement science or in the delivery of measurement services. The FY 1987 recipient of this major NBS award was Norman B. Belecki of the Electricity Division in the Center for Basic Standards. Mr. Belecki was recognized for his many outstanding contributions to electrical metrology during his distinguished career at NBS, including the initiation of a research program to develop conductivity standards for eddy current nondestructive evaluation instrumentation, which led to several Standard Reference Materials.

Department of Commerce Bronze Medal

The Bronze Medal Award is the highest honorary recognition available for Bureau presentation. Recipients during FY 1987 included Dr. David S. Lashmore of the Metallurgy Division. Dr. Lashmore was cited for his outstanding contributions to the development of electrodeposited, composition-modulated alloys, which consist of alternating layers of different metals or alloys. One of the earliest implementations of this development was Standard Reference Material 1850, a test block for checking the sensitivity and performance of liquid dye penetrants.

Eugene Casson Crittenden Award

The Crittenden Award recognizes superior achievement by employees who perform supporting services which impact technical programs beyond their own. One of this year's awardees was David R. Kelley of the Metallurgy Division. Mr. Kelley was honored for the outstanding and unique electroplating services he provides to the NBS staff. One of those services is the careful preparation and certification of Standard Reference Material 1850 (see above).

NBS Safety Award

The NBS Safety Award for Superior Accomplishment recognizes unusually significant contributions to the NBS Occupational Safety and Health program activities. The award is presented to individuals and to organizational units. The Office of Nondestructive Evaluation was honored with an organizational award in recognition of a 7-year unblemished safety record.

Dr. Gerald V. Blessing, Automated Production Technology Division, Center for Manufacturing Engineering, was appointed Publications Chairman for the 1987 International Ultrasonics Symposium of the Institute of Electrical and Electronic Engineers.

Dr. Charles D. Ehrlich, Temperature and Pressure Division, Center for Basic Standards, was appointed Co-Chairman of the Calibrated Leak Standards Subcommittee of the American Vacuum Society, and Vice Chairman of ASTM's Subcommittee E07.08 on Leak Testing Methods.

NBS-114A (REV. 2-8C)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NBSIR 87 3611	2. Performing Organ. Report No.	3. Publication Date October 1987

4. TITLE AND SUBTITLE

Nondestructive Evaluation, Technical Activities, 1987

5. AUTHOR(S)

H. Thomas Yolken, editor

6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions)

NATIONAL BUREAU OF STANDARDS
U.S. DEPARTMENT OF COMMERCE
GAITHERSBURG, MD 20899

7. Contract/Grant No.

8. Type of Report & Period Covered

Annual, FY 1987

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

SAME

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

A review of the Nondestructive Evaluation Program at NBS, for fiscal year 1987, is presented in this annual report.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

acoustic emission; composite materials; eddy currents; materials processing; metal forming; nondestructive evaluation; powders, ceramic and metal; radiography; sensors; standards; thermal testing; ultrasonics.

13. AVAILABILITY

☒ Unlimited

☐ For Official Distribution. Do Not Release to NTIS

☐ Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

☒ Order From National Technical Information Service (NTIS), Springfield, VA. 22161

14. NO. OF PRINTED PAGES

83

15. Price

$13.95

USCOMM-DC 6043-P8