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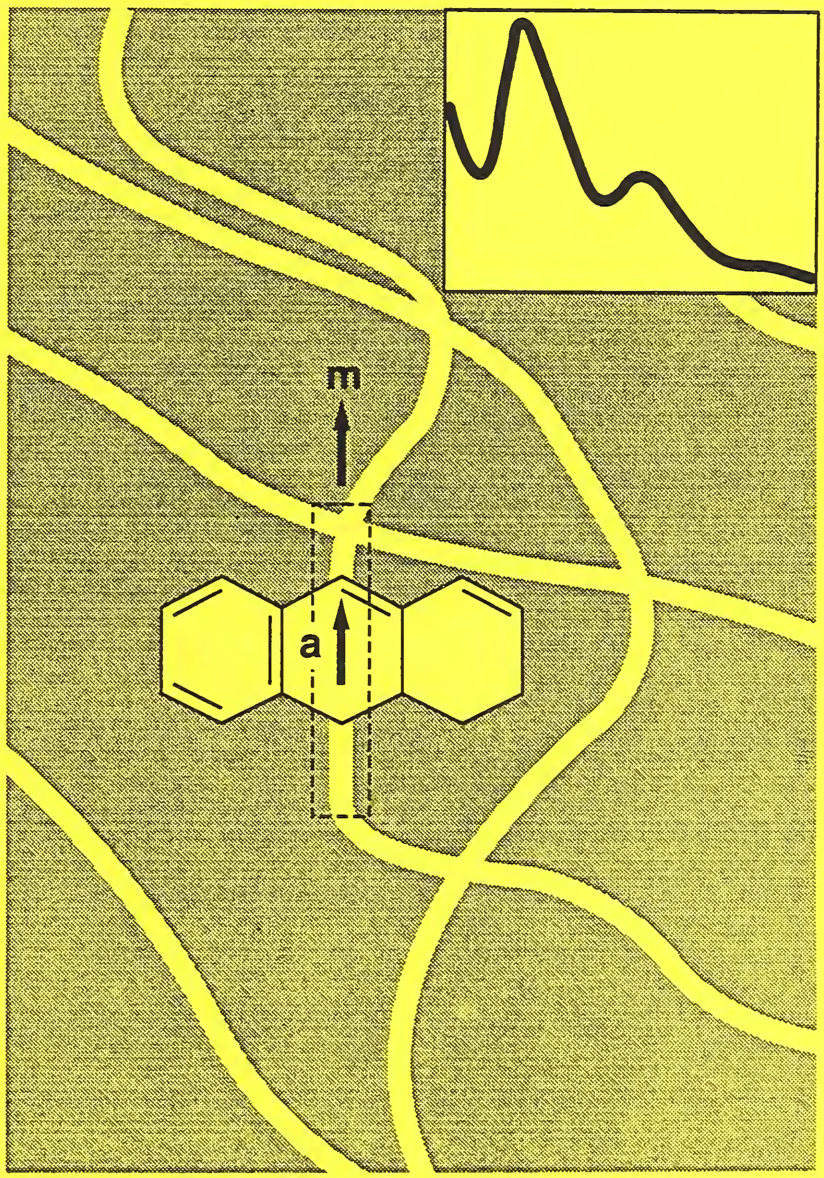
# INTELLIGENT PROCESSING OF MATERIALS




NAS-NRC
Assessment Panel
November 14-15, 1991

NISTIR 4693
U.S. Department of Commerce
National Institute of Standards
and Technology

Technical Activities
1991

A naphthalene flourescent tag is shown inserted in the center of a polymer chain. The tagged chain is added to a polymer melt at a concentration as low as $10^{-5}$ mole per cent. The vectors shown on the naphthalene show the polarization of the emitted flourescent light (The insert is the spectrum of the naphthalene tag.) The polarization of the flourescence can be used to monitor the orientation of a polymer melt on processing. See the article by A. J. Bur and F. W. Wang for details.

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# INTELLIGENT PROCESSING OF MATERIALS

H.T. Yolken, Chief
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U.S. DEPARTMENT OF COMMERCE, Robert A. Mosbacher, Secretary
National Institute of Standards and Technology, John W. Lyons, Director

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# INTRODUCTION

In fiscal year 1991, the Office of Nondestructive Evaluation (NDE) completed its redirection into the broader area of intelligent processing of materials. The name change to the Office of Intelligent Processing of Materials (IPM) was associated with an expanded mission and additional funding to carry it out. These changes were based largely on the formal strategic plan for this Office which projected the research thrust into the 1990's. The research focus was changed to incorporate process modeling, development of NDE sensors for on-line process control, and in some cases, integration of these elements with an expert control system to demonstrate key aspects of intelligent materials processing.

In past years the research program and this annual report were focused on various aspects of NDE research. The report for this year incorporates research from the new areas of intelligent processing of materials and process modeling, and continues research in NDE measurement methods and standards. This approach provides enhanced opportunity to link research in this program with other NIST efforts in materials science and engineering, and provides greater opportunity for interactions with industry.

In FY 1991, researchers supported by the Office of Intelligent Processing of Materials made a number of significant scientific and technical advances. Although this report contains more detailed descriptions, I would like to highlight a few of the more significant advances.

There have been two noteworthy accomplishments in the efforts concerned with research on intelligent processing of steel. The first addresses the formability of sheet metal, which in large part depends on grain texture and orientation. Texture, a preferred crysallographic orientation of grains, is a significant variable resulting from processing and important in the manufacture of such diverse items as automobile panels and appliances. In order to improve process control, Dr. R. Van Clark and colleagues in the Materials Reliability Division have been working for several years to develop a non-contacting ultrasonic measurement system to determine grain texture. This year's research, in collaboration with Ford Motor Company, demonstrated that the technique is applicable to rapidly moving sheet steel.

The second accomplishment in the efforts focused on intelligent processing of steel involves a new project to develop a magnetic NDE sensor for measuring the mechanical properties of steel sheet during rolling. Research this past year by Dr. L. Swartzendruber and Mr. G. Hicho of the Metallurgy Division demonstrated initial feasibility of utilizing magnetic Barkhausen noise measurements to determine mechanical properties of sheet steel. Current efforts are aimed at evaluating mechanical properties for a wider variety of steels, and for the case where the sample is moving. Both of these accomplishments move NDE measurement well into the process stream and focus on mechanical properties rather than defects.

Another advance was the demonstration of the feasibility of applying eddy current sensor techniques to on-line monitoring of the pyrolysis of carbon/carbon composites at high temperatures. This research was the result of a cooperative effort between General Dynamics Corporation and Dr. A. Kahn and coworkers in the Metallurgy Division. Use of an on-line eddy current sensor is intended to correlate conductivity changes with various stages of the pyrolysis used to process the composites.

Two years ago, this Office established a NIST-wide project to develop intelligent processing technology for electronic solder connections. Supported by the U.S. Army Harry Diamond Laboratories, this project is part of a larger Department of Defense (DoD) program to control the cost of electronic components through advances in manufacturing. Researchers Dr. R. Dewitt from the Metallurgy Division and Dr. D. Reed from the Materials Reliability Division made significant progress this year in evaluating and modeling the mechanical properties of solder joints. In addition, Dr. Roger Clough in the Metallurgy Division developed metal-matrix composite solders. These composites which employ particulates of Cu-Sn intermetallic compounds in a matrix of lead-tin solder showed marked improvement in mechanical properties over conventional lead-tin solders.

Several new projects were started this year. In FY 1990, a three year industry-NIST effort on intelligent processing of rapidly solidified metal powder was successfully completed. At the request of the industrial partners, planning was completed for a second research program, in this case focused on the demonstration of all aspects of intelligent processing as indicated by on-line control of powder particle size for Inconel 625, a high nickel austenic alloy. Industrial and other agency sponsors for this program, initiated in March 1991, include Pratt and Whitney, General Electric, Crucible Materials, Martin Marietta Energy Systems (Oak Ridge National Laboratory), and the U.S. Department of Energy.

Another new project started in FY 1991 is aimed at applying an electroacoustic technique to measure the dispersion of ceramic particles in dense slurries. This research is potentially important to the ceramics processing industry. New research programs were also included in a project on modeling of droplet detachment in welding (in cooperation with Professor Julian Szekely of the Massachusetts Institute of Technology), and the previously mentioned Barkhausen noise NDE technique for determining the mechanical properties of steel.

In closing, I would like to repeat what I said last year. The prospects look bright for the new program on intelligent processing of materials. This Office is positioning itself to have a major impact on American industry through its research programs, active communication with a broad range of materials processing specialists, and a growing collaboration with industry to transfer elements of intelligent processing to the American factory. Technology transfer, as measured by the number of cooperative research agreements with industry, will continue to gain importance in research planning and execution. We look forward with great enthusiasm to working with the American Iron and Steel Institute and their member companies, producers of advanced metal alloys, the polymers industry, the ceramics industry, and other users and producers of advanced materials.

H. Thomas Yolken, Chief
Office of Intelligent Processing of
Materials

For more information on projects in this report, kindly contact the individual investigators, or contact the Office of Intelligent Processing of Materials, Materials Building, Room B342, National Institute of Standards and Technology, Gaithersburg, Maryland 20899 (301-975-5727).

# TECHNICAL ACTIVITIES

## A. INTELLIGENT PROCESSING OF MATERIALS RESEARCH

This grouping of programs comprises the main focus for integration of intelligent processing technology and collaboration with U.S. Industry. The three major topics include processing of metal powders through gas atomization, advanced steel making, and electronic solder processing.

- A research consortium comprising General Electric Aircraft Engines, United Technology Pratt & Whitney, Crucible Materials Corporation, Martin Marietta Energy Systems, and the Office of Industrial Processes of the U.S. Department of Energy was formed with NIST to demonstrate on-line production control of particle size for Inconel 625 in the Supersonic Inert Gas Metal Atomizer

- Research was initiated in March 1991, and dealt primarily with development of improved analyses and models to describe the breakup of the liquid metal in the atomization die, improvement of the accuracy and sampling rate of the Fraunhofer diffraction system of on-line powder particle size sensing, and modification of the atomizer to increase the melt capacity and improve atomizing chamber pressure control.

- Three previously funded programs and one new project were grouped to address elements of intelligent processing in advanced steel making. An expansion of the project to measure texture in sheet steel during processing using electromagnetic acoustic transducers resulted in substantial progress in improving the accuracy of such measurements, while extending measurement capability to include the case of moving sheet. An effort to generate correlations includes basic metallurgical data and ultimately validate and improve microstructural evolution models for hot rolled steel product was initiated. In this case, the research was centered in the NIST thermomechanical processing simulator, and initially addressed austenite grain evolution models, as well as continuous cooling transformation information for Nb-treated microalloyed steels. The research in magnetic methods was redirected to use Barkhausen noise analysis of sheet steels to quantify key physical and mechanical properties with an intention to move such measurements into actual processing. The investigation of methods of ultrasonic sensing of liquid/solid interfaces in metals was expanded to incorporate laser generation and interferometric detection following establishment of feasibility.

- Research in intelligent processing of solder assemblies continued to be focused on the physical and mechanical metallurgy of intermetallic compounds and sensor development for on-line inspection of fabricated components. This research was directed toward methods for producing composite solders employing particulates of Cu-Sn intermetallic compounds, electrochemical deposition of intermetallic compounds, modeling of the interfacial mechanics, development of X-Ray laminographic standards, evaluation of acoustic microscopy for interface characterization, and validation of real-time analysis methodologies for assessment of the reliability of surface mount technology solder joints. This research is a continuation of the NIST-wide project being supported by the U.S. Army Harry Diamond Laboratories.

**Intelligent Processing of Rapidly Solidified Metal Powders by Inert Gas Atomization**

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In August 1990, following completion of a three year NIST/US industry consortium effort on intelligent processing of metal powder, planning for a second phase program was initiated. On March 1, 1991, the Phase 2 program was initiated following two meetings with potential industrial sponsors. Its objectives are to demonstrate on-line production control of particle size for Inconel 625 in the NIST supersonic inert gas metal atomizer (SiGMA) and to disseminate intelligent processing methodologies to consortium members to enable factory-floor utilization of NIST research and development results.

The main elements of the approach of this program are as follows:

(1) develop and demonstrate real-time, ruggedized particle size sensing using Fraunhofer diffraction;

(2) design, build and evaluate fluid flow rate sensors for the liquid system which, in conjunction with the existing gas flow rate sensor, will enable on-line computation of gas-metal flow rate ratio;

(3) develop needed material properties of liquid Inconel 625 as a function of temperature;

(4) develop computational fluid dynamics (CFD) and experimental correlations which describe the dynamic behavior of Inconel 625 atomization in the NIST atomizer;

(5) design and develop appropriate hardware to control process parameters;

(6) devise and implement intelligent control strategies to achieve desired particle size distributions;

(7) operate SiGMA under on-line particle size feedback control and quantify success in achieving desired particle size distribution.

This program was initiated following establishment of cooperative research and development agreements, direct contracts, and work requests with General Electric Aircraft Engines, Crucible Materials Corporation, United Technologies Corporation, Martin Marietta Energy Systems, Oak Ridge National Laboratory for the United States Department of Energy, and the Department of Energy Industrial Energy Conservation Program. The research reported here summarizes the results of the first six months of this new three year effort.

(1) Process and Model -- Efforts continue at developing the necessary understanding of droplet formation during the gas atomization process. New high speed movies using a copper vapor (C-V) laser to illuminate the atomization plume are providing better temporal resolution of the gas-liquid interaction. Previously, the plume's radiant incandescence required exposure time to be $\geq 10\ \mu s$; however, the 12 watts of laser power combined with the 15 ns pulse duration of the C-V laser will significantly improve the image quality.

Gas only flow studies have continued using Schlieren photography and computational fluid dynamics (CFD) to study compressible gas jets. Euler (inviscid) and Navier-Stokes (viscid) CFD computer codes have progressed to include a compiled code that will run on 32 bit personal computers (PC). In this new version of the atomizer design analysis system, gas only supersonic flow of atomization assemblies can be computed and visualized by coupling the numerical results to a CAD program. This code was delivered to the consortium members for evaluation by their design engineers. The next generation of this software will incorporate an adaptive gridding technique to improve solution speed and accuracy.

(2) Sensors -- The optical access for particle size sensing has been dramatically improved since the completion of the first phase activities. New powder-gas homogenization and window cleaning hardware installed in the exit region of the atomizer have eliminated the particle size sensor beam steering and obscuration. Figure 1 shows data from a recent Inconel 625 run incorporating these new modifications. The gradual drop in diode 0 intensity (the central diode used for alignment and obscuration measurements) and relatively steady output from diode 1 (the innermost scattering detector) are indicative of a homogenized powder mixture that is well suited for particle size measurement.

A new high speed PC with a 33 MHz i486 processor and i860 RISC co-processor was purchased for development of next generation particle sensing software. A new approach for the real-time estimation of the particle size distribution has been investigated which makes use of non-linear optimization searching techniques. This new algorithm has demonstrated significant improvement in accuracy over the previous algorithm; however, the computational requirements also have increased. The algorithm has been implemented on the i486 processor. Work has begun to transfer the new algorithm particle sizing software to the i860 RISC processor.

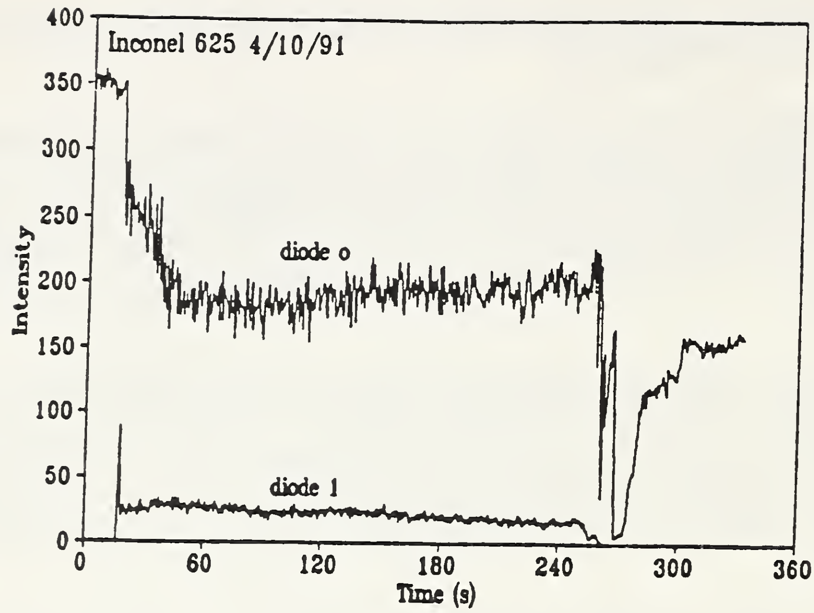


Fig. 1 Laser diffraction data showing outputs from the central diode (Diode 0) and the innermost scattering detector (Diode 1)

Calibration of the particle size sensor will require well characterized size standards. The laser diffraction technique currently in use can be evaluated with stationary spots of known size distributions. These size calibration reticles are currently being studied using microscopy coupled with image analysis software. This work will result in a new size standard reticles with spot sizes ranging from 3 to 100 $\mu$m in various number distributions.

New liquid metal flow rate sensor capability is currently under development to provide data for control of the gas/metal mass flow rate. Two techniques have been identified: Gravimetric measurement using strain sensors and electromagnetic field measurement using eddy current sensors. The strain sensor technique is presently being studied with encouraging results.

(3) Intelligent Control System (ICS) -- A new version of the Expert Control System Shell (ECSS) has been implemented which incorporates more efficient memory management, more powerful knowledge-base construction capabilities, more efficient knowledge-base inferencing mechanisms, additional actuator and display capabilities, and more user friendly data management and play back utilities. Efforts continue in the modelling of the dynamics of various atomizer sub-systems (dome pressure regulator, backfill regulator, etc.) and in the development of control strategies based on the derived models. Work has been performed to develop the communication device driver for the new particle sizing program mentioned above.

(4) Supersonic Inert Gas Metal Atomizer Modifications -- Efforts have been directed to completion of several atomization runs of Inconel 625 and determination of the best crucible and liquid delivery tube materials for this alloy. Several ceramic materials have been used including BN, ZrO, $Al_2O_3$, BN-$Al_2O_3$, BN-SiC, and BN-ZrO. Other hardware additions and/or modifications include: i) a new containment system that enables up to 22.7 kg steel measure to be melted, ii) incorporation of a fiber optic system in near-field viewports modified to enhance high-speed movies, and iii) new pressure regulator and exhaust system components to improve atomizing chamber pressure control.

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**Progress Towards Development of an Intelligent Processing System for On-line Control of Steel Sheet Formability**

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Texture, or preferred orientation, has been shown to be a dominant factor in the formability of high quality sheet metal. This material is important in many applications, especially production of automobile bodies. The American Iron and Steel Institute (AISI) recently endorsed the concept of intelligent processing of sheet metal formability [1]. As currently envisioned, the system will be placed in the continuous annealing line where the recrystallization textures are controlled to obtain the desired formability. AISI set goals of resolving the formability measure, $\overline{r}$, to an accuracy of $\pm 0.05$, capability of performing measurements on rapidly moving sheet (speeds of 150 m/min), and rugged, reliable, cost effective performance. NIST has tailored its ongoing formability research in FY 1991 to meet these goals.

Proof of concept measurements [2] determined that a correlation existed between $\overline{r}$ and $\overline{V}$, the average in-plane velocity of guided waves propagating at 3 different angles in the sheet. These measurements also showed a spread of $\pm 0.2$ in the data. Sources of scatter in the data were identified as metallurgical variability and artifacts in measurements of velocity. Metallurgical variability could arise because texture is not the only effect, influencing and variations in thermomechanical processing influence formability. This led to a search for correlations based on manufacturer.

Error analysis shows that velocities need to be resolved to $\pm$ 0.2% to obtain $\overline{r}$ to $\pm 0.05$. The major uncertainty in the velocity determination is diffraction which causes the acoustic field to spread out and causes wavefront curvature. The relation between $\overline{r}$ and $\overline{V}$ is based on "plane wave" theory where the beam is collimated with straight-crested wave fronts. Thus, there is a need to correct the measured arrival times to the arrival times for plane waves. A model has been developed to do this [3]. The wavefield generated by the transmitting transducer was measured. This amplitude distribution was put into a diffraction integral and evaluated to determine phase (and/or arrival time) of the wave. One component is the "plane wave" phase; there are additional terms due to diffraction. By subtracting the latter from the measured arrival times, we correct to the "plane wave" arrival times. These results were found to be in excellent agreement with theory. Fig. 1 shows the difference between measured arrival times and "plane wave" arrival time, d/V. Here d is the acoustic path length and V is the phase velocity. The theory was used on data obtained on sheets from two manufacturers. When corrected for diffraction, the results shown in Figure 2 are obtained. This shows little scatter in correlation curves, for the "S" and for the "K" sheets.

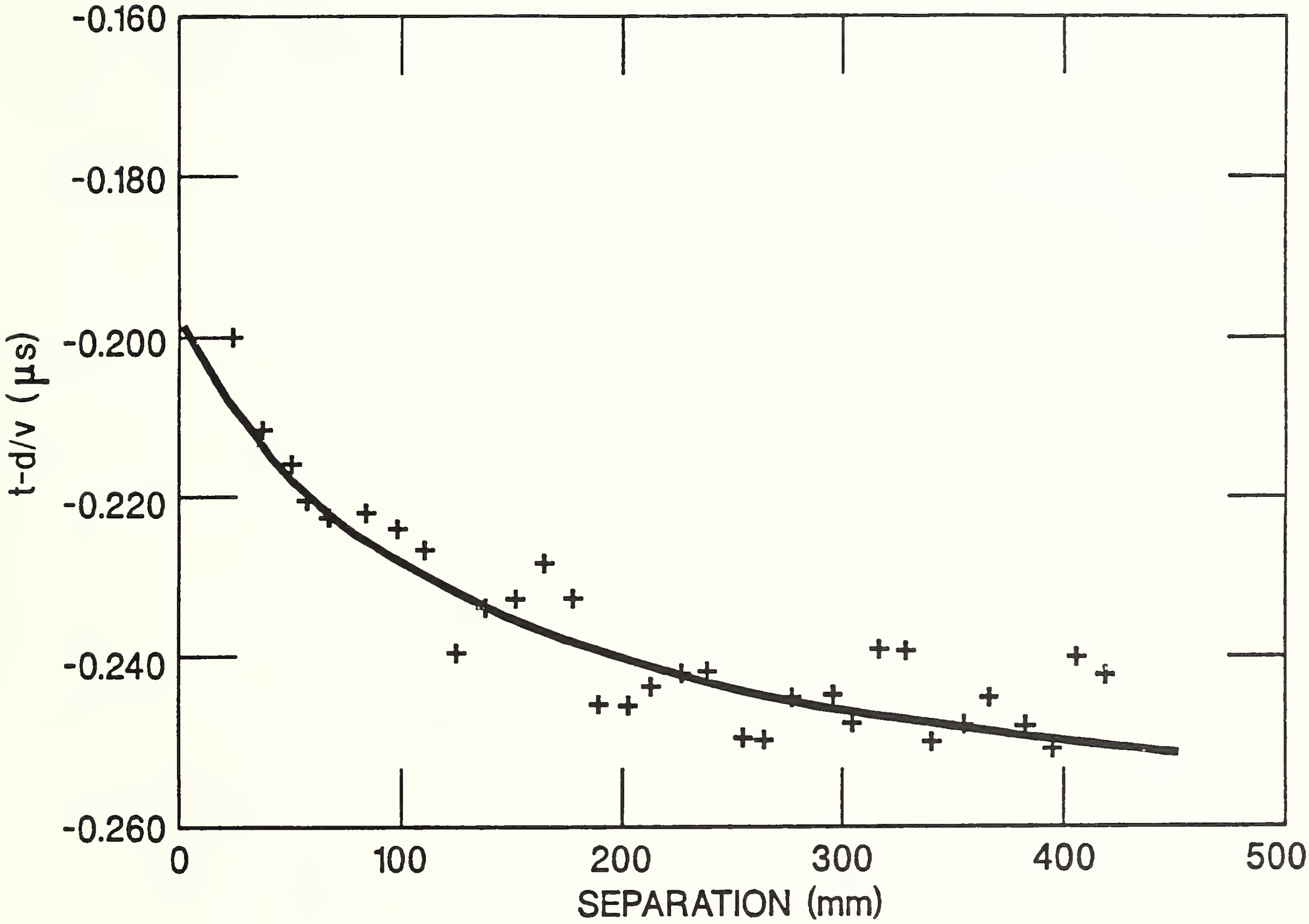


Fig. 1 Difference between measured arrival time, t, and diffraction and time, d/v, for a plane wave (solid line is theory)

A facility was developed for making measurements on rapidly moving sheet [4] as part of a collaboration between NIST and the Ford Motor Company. The principle of operation is as follows. Compressed air enters a cylinder about seven meters in length. This forces a piston down the cylinder. A cable attached to the piston passes over a pulley arrangement and propels a "sled" down a track. A sheet specimen is mounted on the sled and carried under a "bridge" which spans over the track. The bridge houses an array of transducers.

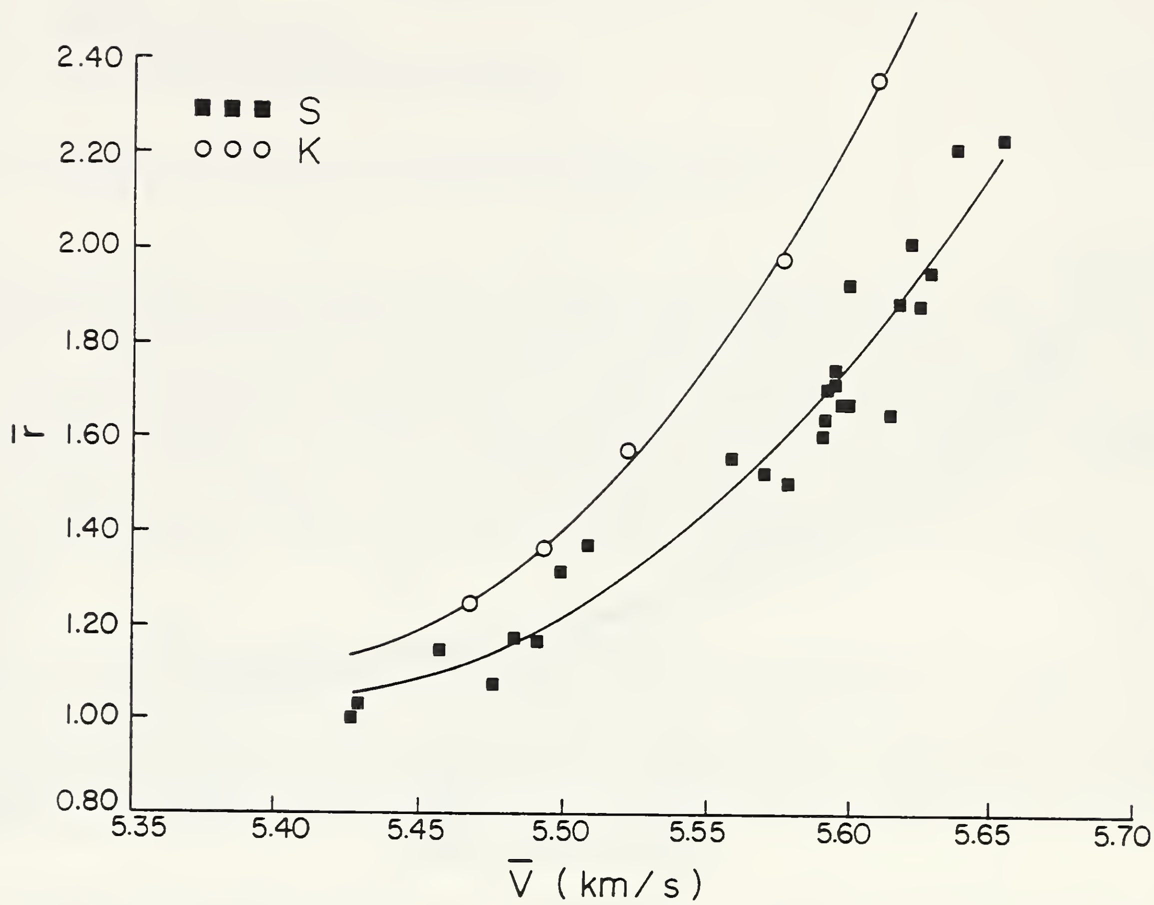


Fig. 2 Result of applying diffraction theory to data taken on sheets supplied by "K" and "S" manufacturers.

Previous measurements (such as those shown in Figure 2) were all made in the static mode. There are several reasons why there could be differences between the static and dynamic mode. For example, when sound is propagated in direction of sheet motion, the wave arrives sooner and takes longer when the sound wave is propagated in the opposite direction. We refer to this as the "relative velocity shift" effect. Also, the receiving transducer has an equivalent circuit which consists of an open circuit voltage source in series with an inductance and resistance. These depend upon clearance (liftoff) between the transducer and the sheet. The effect of changing the circuit reactance is to cause a phase shift. This will depend upon liftoff. A change in phase causes a change in the arrival time. A model was developed to predict the phase as a function of liftoff and frequency [5], with excellent agreement with experiment as shown in Figure 3. This model was used to desensitize the system to liftoff.

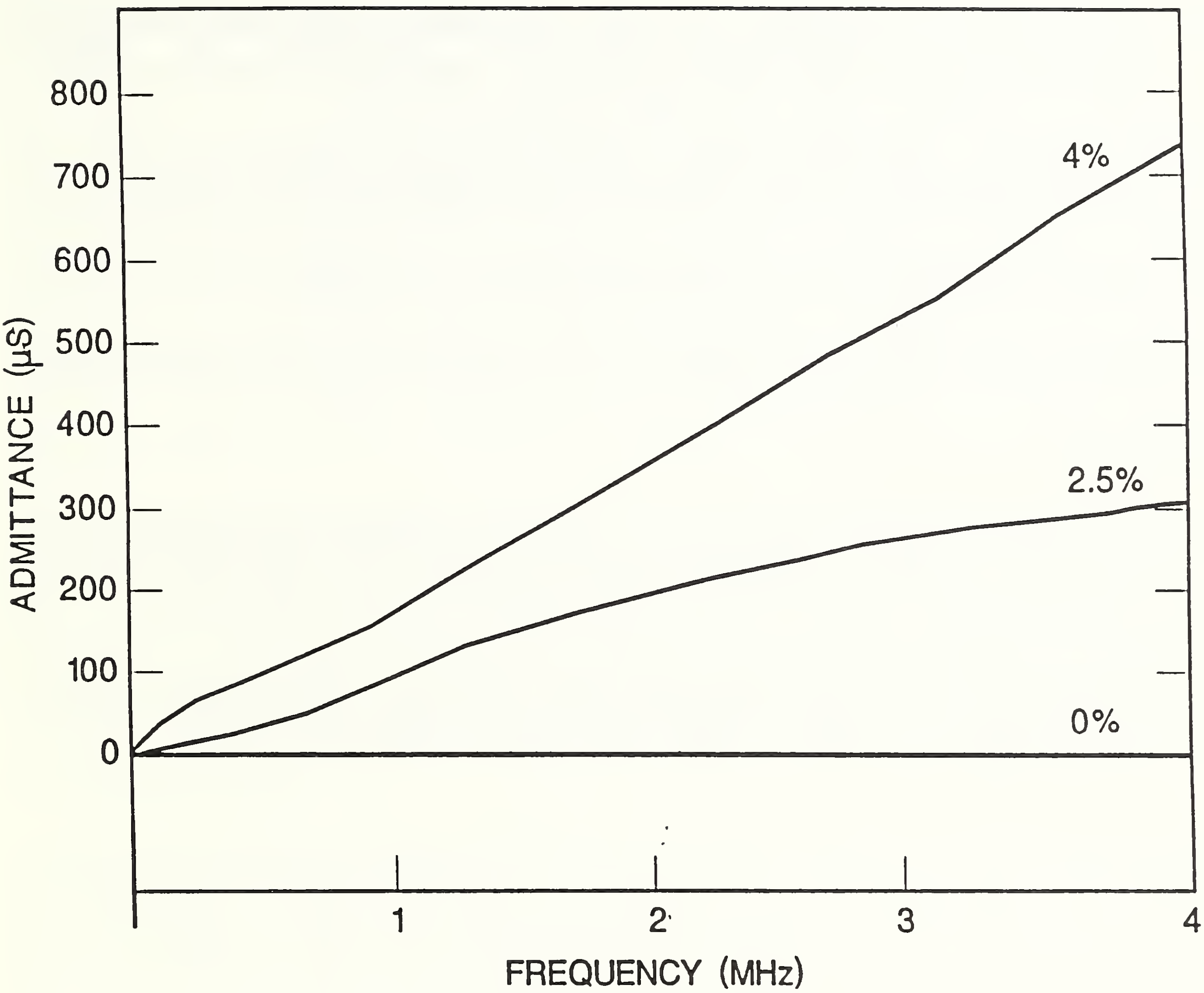


Fig. 3 Comparison of calculated and measured phase for receiving transducer.

Tests were performed on several steel sheets. The major effect was the "relative velocity" effect. When this was accounted for, the difference between the static and dynamic measurements was about 0.02% (10% of the error budget). The NIST Robotics Systems Division and Materials Reliability Division are now collaborating on development of a control system for intelligent processing of sheet in a continuous annealing line. The processing parameters which must be controlled are being identified. Models of the annealing process will be developed, and plans call for selection and implementation of a control strategy using the Real Time Control System developed at NIST.

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**Thermomechanical Processing of Steels**

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The objectives of this research program are to generate basic metallurgical data and to develop, improve, or validate microstructural evolution models that are needed in the intelligent processing of steels. Thermomechanical processing (TMP) is a continuous process for producing steel products by controlling the time-temperature-deformation schedule during hot working such that the product properties in the as-fabricated condition equal or exceed those of heat-treated grades. The key to improving TMP is intelligent control of the time-temperature-deformation schedule used in hot working. Basic metallurgical data and microstructural evolution models are two of the ingredients that are required in implementation of intelligent processing. Currently, with a few exceptions, models used in industry are qualitative and empirical. Research is needed to experimentally verify and improve these models and to develop new ones based on physical principles. Verification, improvement, and development of models all need basic metallurgical data.

The nonequilibrium metallurgical events that occur during TMP, and the high-temperature flow behavior of steel are measured in the solid state. The data obtained are presented in the form of high-temperature, high-strain rate, stress-strain curves and continuous-cooling-transformation (CCT) diagrams. Experiments are performed to verify the microstructural evolution models that appear in the literature. The experiments are being conducted with a TMP simulator which was designed and built at NIST/Boulder. The results obtained to date are summarized and discussed in the following three areas: (1) experimental verification of austenite grain evolution models during TMP; (2) determination of continuous-cooling-transformation (CCT) diagrams following deformation; and (3) generation of high-temperature, high-strain rate stress-strain curves.

In steels, a fine austenite grain size is a prerequisite for obtaining fine ferrite grains that simultaneously increase strength and improve toughness. At high temperatures, austenite grains recrystallize after deformation (static recrystallization). The recrystallized grain size, $d_{rex}$ is a function of the grain size before deformation, $d_o$, and the amount of applied strain, $\epsilon$. Sellars [1] proposed the equation, $d_{rex} = C\ d_o^m\ \epsilon^n$, to describe the relationship for Nb-treated microalloyed steels, where C, m and n are constants. The equation can be used for optimizing the deformation schedule to achieve the finest austenite grain sizes possible during TMP.

Experimental results obtained to date in this investigation indicate that Sellars' model works very well in cases where $d_o$ is relatively large (57 and 137 $\mu$m). When $d_o$ is in the order of 20 $\mu$m, the model underestimated the $d_{rex}$. The comparison of experimental and calculated recrystallized grain sizes are presented in Figure 1 where the x-axis is the amount of strain applied to a specimen in one deformation, and the y-axis is the recrystallized grain diameter after one deformation. The results show that grain refinement is most effective when $d_o$ is relatively large. However, no or little grain refinement can be achieved when $d_o$ is in the order of 20 $\mu$m. Figure 1 also shows that deformation temperature, which was not taken into consideration in Sellars' model, does not affect the results significantly. However, for Ti-V-treated microalloyed steels, the grain size predictions by the model proposed by Roberts et al.

[2] did not agree well with limited experimental results obtained to date. Additional experiments will be performed to check the models.

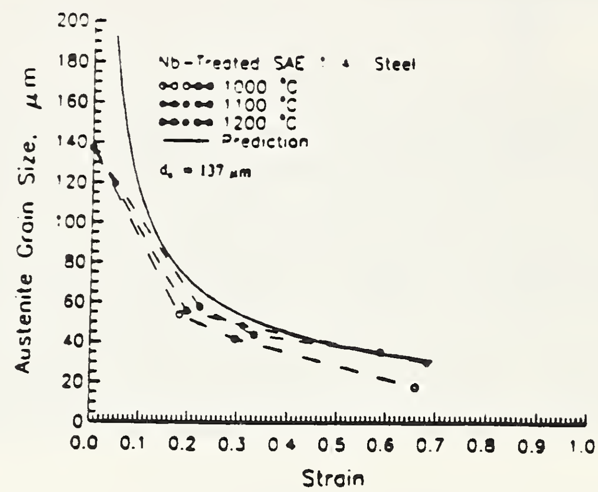


Fig. 1 Comparison of experimental and calculated recrystallized grain sizes as a function strain for a Nb-treated microalloyed steel.

In the study of CCT characteristics, specimens of a Nb-treated microalloyed steel were reheated to 1218 °C and compressed 50% at 900, 1000, and 1100°C. Following compression, austenite-to-ferrite (or other transformation products) transformation temperatures were determined from dilatation-vs.-temperature curves and from cooling rate-vs.-temperature curves. The results show that CCT diagrams determined following compression shifted the ferrite-plus-pearlite (F+P) nose to a shorter time and produced a much finer F+P microstructure. Figure 2 present two CCT diagrams: one determined without deformation (Figure 2) and the other with a compression of 50% thickness reduction. In Figure 3, CCT diagram determined without deformation, the tip of the (F+P) region is at about 20 s. With a deformation of 50% thickness reduction at 900°C, the tip shifts to about 8 s. The shift of (F+P) region to a shorter time is due to the influence of effective austenite grain size before transformation on hardenability. The effective austenite grain size is much smaller after deformation (Figure 1), especially in the case of deforming at 900°C, at which the austenite grains did not recrystallize. The CCT diagrams can be used to estimate the hardenability of the steel, to estimate the microstructure after processing, and to estimate the mechanical properties of the steel.

For a Nb-treated microalloyed steel, stress-strain curves have been determined under different temperatures and different strain rates. Cylindrical specimens were compressed at temperatures of 900, 1000, 1100, and 1200°C and at constant strain rates of 0.1, 1, and 10 $s^{-1}$. The results show that the flow stress decreases with increasing test temperature. The flow stress also decreases with decreasing strain rate, except in the case when precipitation occurred at 900°C with the strain rate of 0.1 $s^{-1}$. In the latter case, the flow stress is higher than that of testing at the strain rate of 1 $s^{-1}$.

In the course of conducting TMP research on steel, extensive interactions with researchers in the steel industry have been established and maintained. Chrysler Motors Corporation, National Steel Corporation, Inland Steel Corporation, and the American Iron and Steel Institute were included in these industrial contacts during FY 1991.

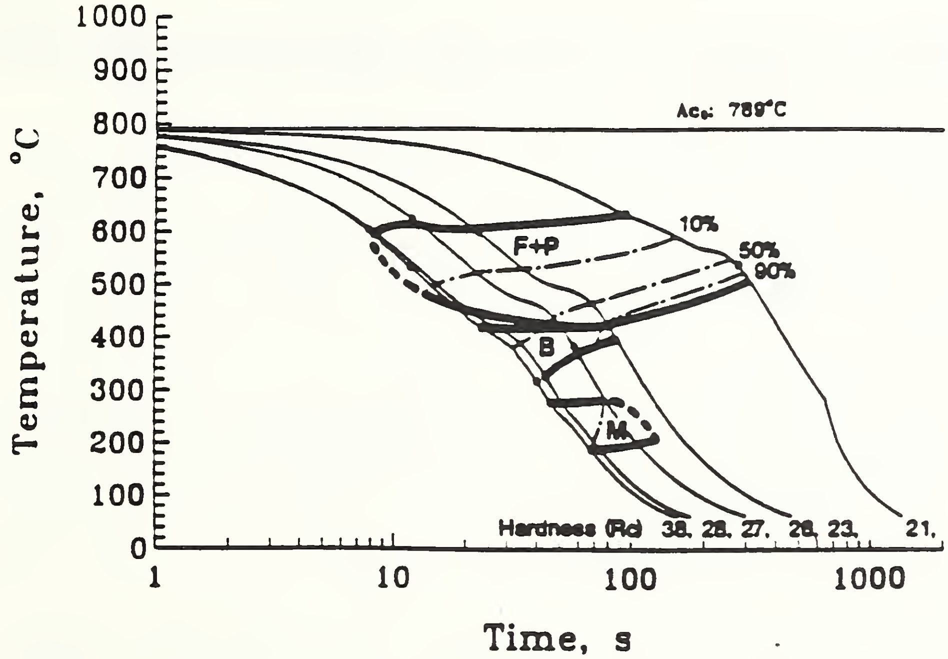


Fig. 2 The CCT diagram of a Nb-treated microalloyed 1141 steel after reheating to 1218 °C and compressed 50% at 900°C. F: ferrite; P: pearlite; B: bainite; M: martensite.

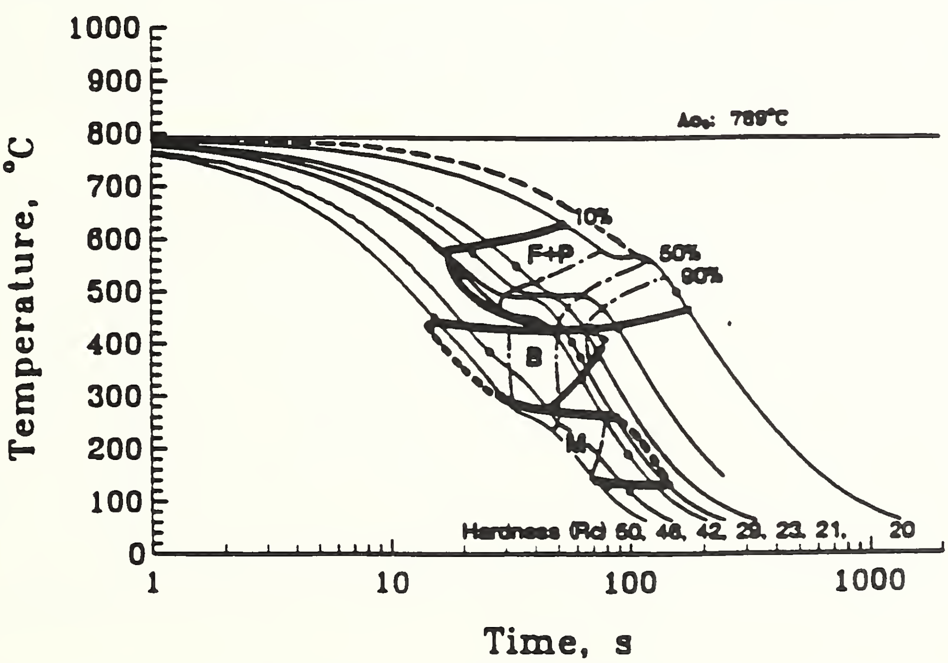


Fig. 3 The CCT diagram of a Nb-treated microalloyed 1141 steel after reheating to 1218 °C. F: ferrite; P: pearlite; B: bainite; M: martensite.

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## Magnetic Methods for Evaluation of Mechanical Properties of Steels

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The objective of this investigation is to improve and extend the applicability of magnetic methods for gaging the mechanical properties of steel. Current emphasis is on the use of magnetic Barkhausen noise on steel sheet.

The mechanical properties of steel depend on microstructural parameters such as grain size, chemical composition, precipitate concentration and morphology, and defects such as interstitial or substitutional impurities and vacancies. Although a large number of laboratory techniques are available to determine microstructure and thus infer mechanical properties, few of these are applicable in an intelligent processing environment where results must be obtained quickly and continuously. Because magnetic properties can be measured rapidly and are sensitive to the same microstructural parameters that affect mechanical properties, they have potential for processing control of ferritic steels.

Magnetic properties of interest include magnetic Barkhausen noise, magnetoacoustic emission, residual magnetization, hysteresis, permeability, coercive force, and flux leakage. All these properties are sensitive, to a greater or lesser degree, to the microstructure, applied stress, residual stress, plastic deformation, grain size, and hardness. The challenge in using magnetic measurements is to select those measurements, or combination of measurements, which can be determined rapidly in a production environment and which will be effective in determining changes in those properties of most interest. For the case of rolled sheet steel, two critical properties are of interest. These are the ferrite grain size, which controls the yield strength, and the volume fraction and morphology of the pearlite. Because magnetic Barkhausen noise is known to be sensitive to these properties, current emphasis has been on this property.

In order to assess possible relationships between mechanical properties and magnetic properties in sheet steel, experiments were conducted on four thicknesses of carbon sheet steel supplied in both the cold rolled (CR) and hot rolled (HR) conditions. Microstructural examinations, hardness, mechanical property tests, and Barkhausen measurements were conducted on these specimens. The sheet steels used in this study were AISI 1015/1020 with specified compositions of 0.15/0.20 weight percent carbon, 0.30/0.90 weight percent manganese, 0.040 weight percent phosphorus maximum, 0.050 weight percent sulfur maximum, and the remainder iron. Thicknesses ranging from 0.58 mm to 3.17 mm were selected. These thicknesses represented different percentages of cold reduction and therefore were likely to have different percentages of pearlite in the final microstructure. For comparison purposes, a hot rolled steel was also selected. This steel was used to ascertain the effect of cold reduction on the Barkhausen measurement. Hot rolled steel is reported to have a more equiaxed grain structure, and if the cooling rate was slow enough, more pearlite in the final microstructure. Texture effects, produced by heavy rolling in one direction and accentuated in cold rolled steel, should be minimal in this steel.

Prior to preparing the tensile test specimens, sections were removed from each thickness, metallographic specimens prepared, and the as-polished and etched microstructures determined. The microstructure of the as-received steels were examined in three orthogonal directions. Ferrite grain size and hardness were measured. Mechanical properties were determined using standard flat tensile specimens prepared and tested according to ASTM Method A 370. The ultimate tensile strength and 0.2% offset yield strength, reduction in area, and elongation in 50.8 mm were determined. In addition the work hardening exponent was determined from the representative stress-strain curves.

Magnetic Barkhausen noise spectra were obtained on each sample using a ferrite core surface pickup transducer. The field and the pickup coil axis were parallel to the rolling direction. Although some of the samples displayed considerable anisotropy in their noise spectra, this aspect will not be discussed here. An applied magnetic field with a maximum value of 2.5 mT was used. From the data obtained from each sample, the power density spectrum, the jumpsum vs. applied field, the jumpsum rate, and the noise emission rate vs. field, could be determined. The jumpsum, which represents a sum of the amplitude of individual Barkhausen jumps observed as the field is increased, is a particularly useful method for analyzing Barkhausen data. The jumpsum rate is the derivative of the jumpsum curve after suitable smoothing. Figure 1 shows the jumpsum rate vs. the applied field for the five samples. In addition the jumpsum rate from SRM 493 is shown. SRM 493 is a sample of spheroidized cementite in ferrite which gives a reproducible magnetic noise signal and is included for reference. Considerable structure is evident in the jumpsum rate.

The relationship of the structure seen in the jumpsum rate curve of Fig. 1 to the microstructure of the material poses an interesting challenge. There are two aspects of the noise signal which are obvious choices for parameters to relate to mechanical properties. One, the total jumpsum, $J_t$ is the total area under the jumpsum rate curve. The other is the magnetic field value, $H_{jc}$, at which the area under the rate curve reaches half its total value. The latter is preferred because it is less sensitive to details such as probe liftoff, amplifier drift, etc. In general, $J_t$ and $H_{jc}$ are closely related.

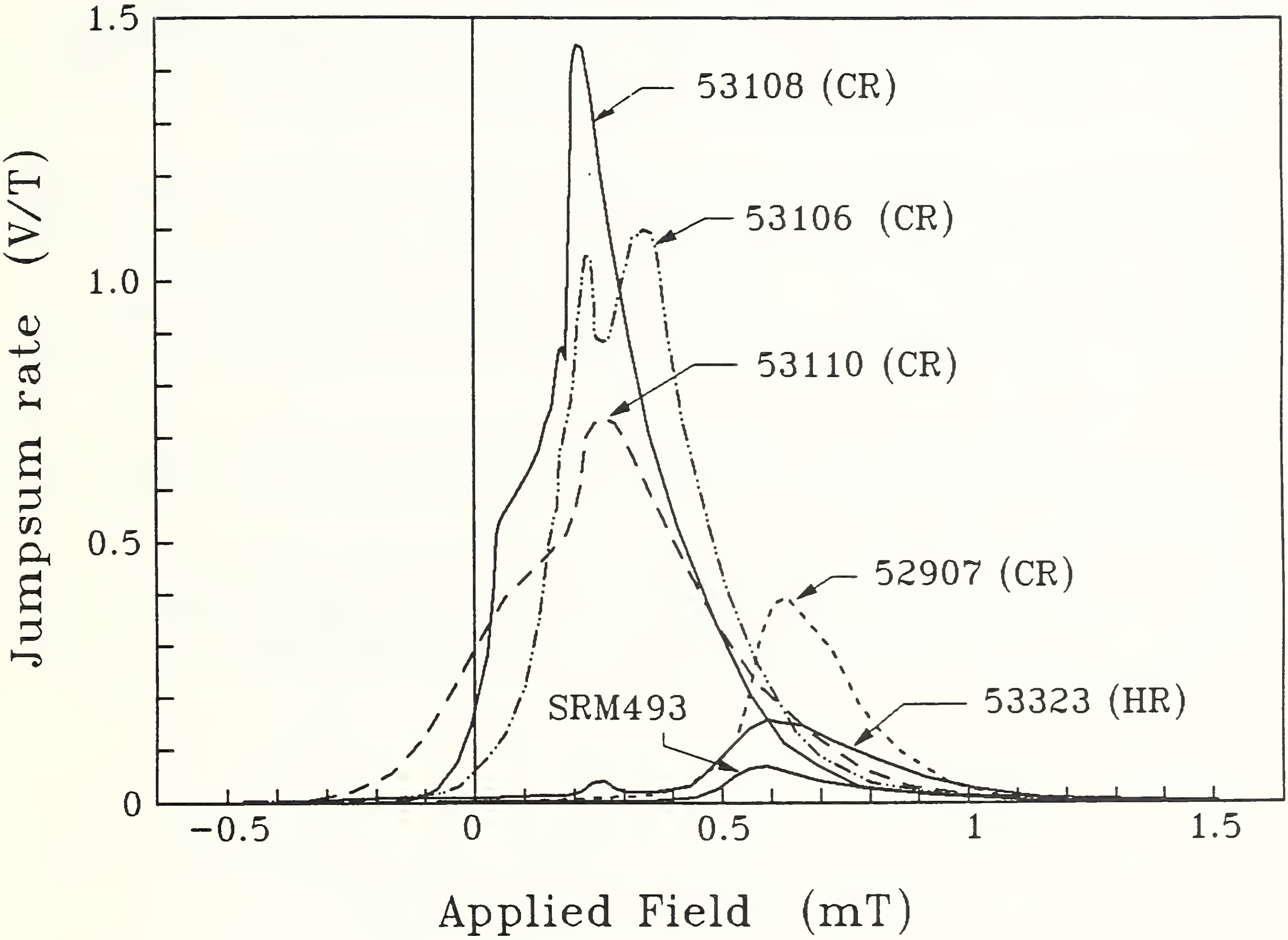


Fig. 1 Barkhausen noise data obtained during analysis of cold rolled (CR) and hot rolled (HR) mild steel sheet and standard reference material SRM 493

It was found that both $J_t$ and $H_{jc}$ were are good predictors of hardness. The yield strength (YS) and ultimate tensile strengths (UTS) correlate well with the measured hardness. The hardness was found to correlate well with YS and UTS for the cold rolled materials, but the hot rolled material did not lie on the curves. Similarly, both YS and UTS correlate well with $J_t$ and $H_{jc}$ for the cold rolled material, but the hot rolled material lies off the curves.

The theoretical relationship between grain size and YS was not observed. This is probably due to the large variance in grain size within each sample. Microstructural analyses revealed that the HR specimen had fine, equiaxed ferrite grains and the CR specimens had large colonies of pearlite. These both result in higher hardness for the HR material.

Measurements were made of the derivative of the stress-strain curve, $d\sigma/d\epsilon$, from which the work hardening coefficient, n, was determined. Little correlation between n and the other mechanical properties was obtained. This may be due to the fact the incremental differences used in measuring $\sigma$ and $\epsilon$ were too small, resulting in large errors in measurement of n.

Although good correlation between YS and $H_{jc}$ was found, it was found that the correlation was better if another characteristic of the jumpsum rate curves shown in Figure 1 was used. This parameter is called the normalized second moment. This value is obtained by calculating the second moment of the jumpsum rate curve and dividing this value by the total area of the curve and then divided by the same quotient found for SRM 493. A plot of the measured YS versus the normalized second moment is shown in Figure 2. As for all other plots attempted, the HR sample does not fall on the correlation curve for the cold rolled samples.

Concomitant with these studies, methods and transducers for obtaining noise data from moving sheet are being explored. The best methods to use will depend on which magnetic characteristics correlate best with desired mechanical properties.

Plans call for the exploitation of the above results to observe the changes in magnetic properties of an AISI 1015 sheet steel during mechanical reduction. The effect of processing variables, including austenitizing temperature and percentage cold reduction, on the mechanical and magnetic properties will be investigated.

## Reference


1. L. J. Swartzendruber, L. H. Bennett, H. Ettedgui, and I. Aviram, "Barkhausen Jump Correlations in Thin Foils of Fe and Ni," J. Appl. Phys. 67, (1990), 5469.

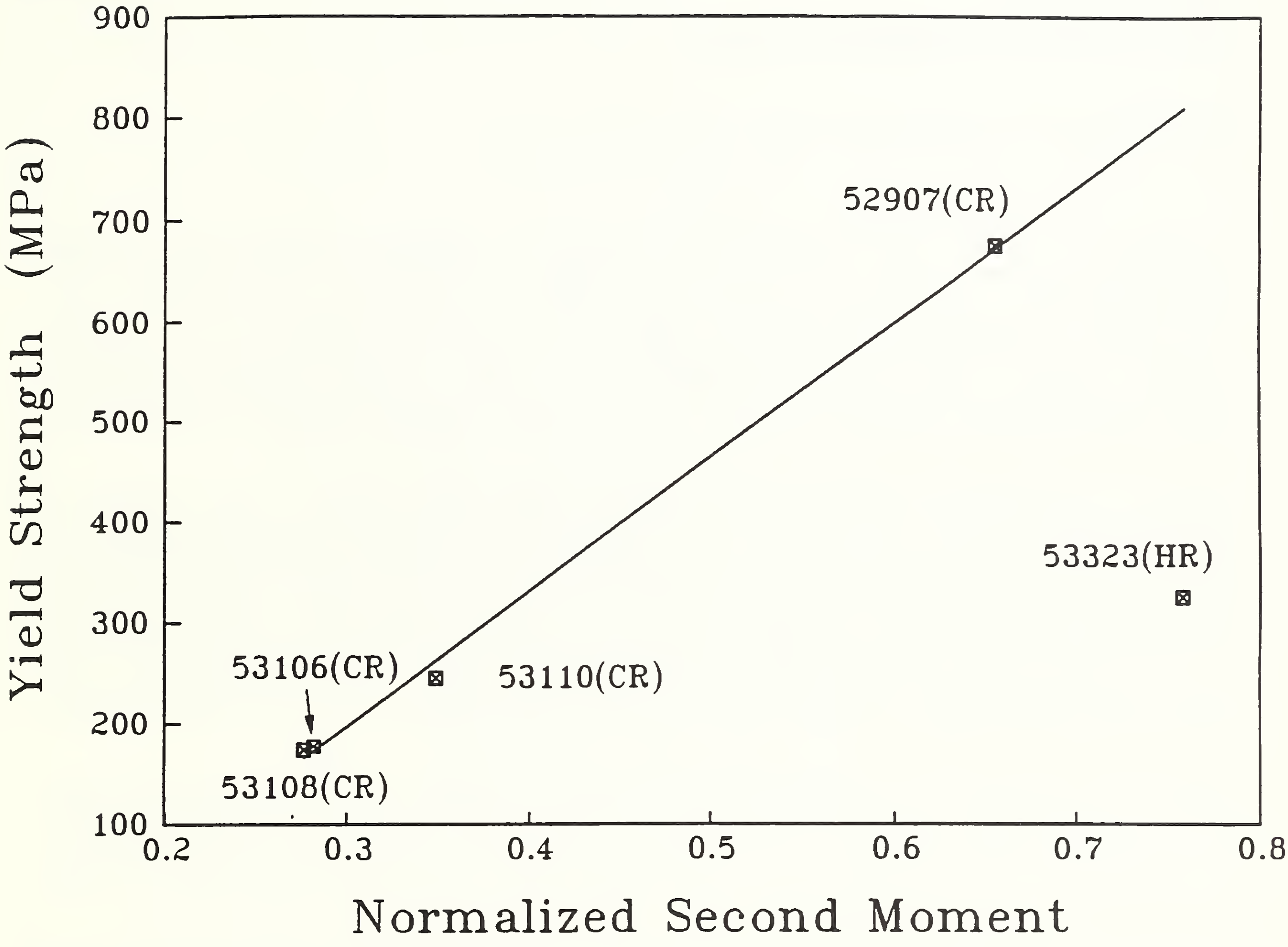


Fig. 2. Correlation between yield strength and a characteristic of the curves in Fig. 1.

## Ultrasonic Sensing of Liquid/Solid Interfaces in Metals

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In metallic alloys produced by directional solidification, the mechanical properties of the finished material are greatly influenced by the details of the solidification process. Characterization of the liquid/solid interface is, therefore, of primary importance for both process modeling and process control. Unfortunately, it is not currently possible to monitor in real-time the microstructure of a metallic liquid/solid interface in bulk material, and monitoring of the position of interfaces has been accomplished only with simple geometries in the laboratory. Experimental verification of solidification models for microstructural evolution must rely on time consuming analysis of quenched samples.

This continuing research project has been motivated by the realization that ultrasonic waves constitute one of the few forms of probing energy which can provide real-time information on liquid/solid interfaces in metals. Unlike most past research in this area, a major goal of this project has been to obtain information on not just the interface position but also microstructural features of the interface. The very significant scattering of ultrasonic waves from grain boundaries in the solid makes it impossible to obtain information on the microstructure of the interface by means of ultrasonic waves transmitted through the solid. Therefore, the approach has been to use waves generated and received in the liquid to study ultrasonic scattering from the interface.

Past work on this project explored the feasibility of a number of sensor configurations and performed model scattering studies on simple periodic structures. Also a unique ultrasonic resonance technique [1] was developed for measuring attenuation in metallic droplets undergoing solidification in order to provide reference data which is considered necessary for the interpretation of scattered waveforms.

During FY 1991, the focus has been on sensor design and basic hardware construction. From among a variety of considered transduction schemes, two configurations have been selected for further research. One of these employs a wire waveguide encased in a tube and in contact with liquid metal only at one end. Both piezoelectric and magnetostrictive techniques have been employed for generating and detecting ultrasonic waves in these waveguides. A second sensor configuration employs laser ultrasonics. A transparent rod is immersed in the liquid metal and the wave is generated and received at the surface of the liquid which is in contact with the end of the rod Figure 1. A Nd-YAG laser is used to excite the ultrasonic wave in the liquid at the end of the rod and an interferometer is used to detect the scattered wave after it is reflected off the liquid/solid interface.

Tests have been performed with a hybrid configuration employing laser generation at the end of a silica rod and magnetostrictive detection through a 2-mm-diameter nickel wire, Figure 2. The magnetostrictive transducer was configured to detect primarily at 0.5 MHz so that the wavelength in the nickel was much greater than the wire radius and the received pulses were relatively non-dispersive. The silica rod and encased nickel wire were immersed in liquid tin and ultrasonic reflections off an interface with solid stainless steel were observed. The received signals showed a pulse corresponding to the direct transit between the transmitter and receiver followed by a pulse corresponding to reflection off the stainless/tin interface. The arrival time of the second of these pulses changed in a predictable manner as the interface was moved.

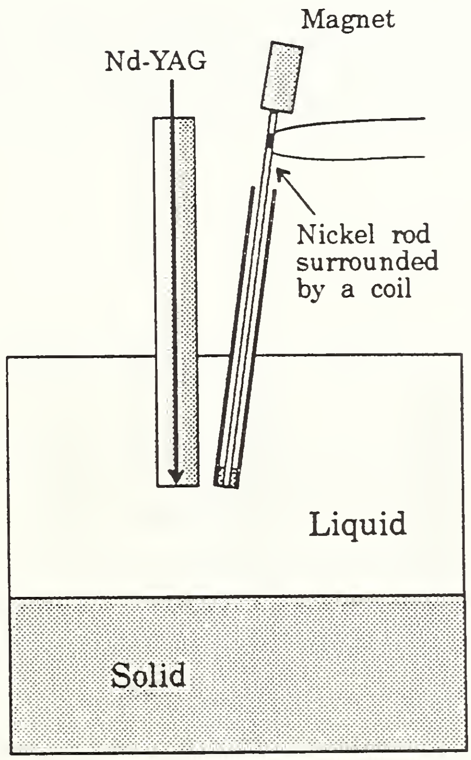


Fig. 1 Ultrasonic configuration consisting of an immersed magnetostrictive waveguide for ultrasonic reception and an immersed transparent rod through which pulsed Nd-YAG laser light is passed to generate ultrasonic pulses in the liquid metal.

The configuration employing laser generation and interferometric detection, Figure 1, was initially tested at The Johns Hopkins University in collaboration with Drs. James Wagner and James Spicer. A small, pulsed Nd-YAG laser (~15 mJ) was used for the ultrasonic wave generation and a two-beam interferometer was used for detection. The light was passed through a silica rod which was immersed in liquid tin interfaced to solid brass. A small signal was observed corresponding to reflection off the interface, but detection was complicated by saturation of the interferometer from the generating laser pulse and spurious ultrasonic signals arising from reflections inside the silica rod.

During this fiscal year, it has been a major objective to implement laser ultrasonic detection at NIST. A state-of-the-art laser ultrasonics facility is regarded as important for a wide variety of sensing applications in addition to liquid/solid interface characterization. A two-beam interferometer has been constructed and a Fabry-Perot interferometer is partially constructed. The second of these is most useful in the typical materials processing situation where the sample surface is not optically flat.
The initial test at has been followed by tests at NIST employing this interferometer and a higher power 150 mJ Nd-YAG generating laser. The tests were performed with liquid tin interfaced to solid stainless steel. Separate immersed silica rods were used for generation and detection in order to reduce the spurious signals encountered at Johns Hopkins. Clear signals corresponding to the interface reflection were observed.

The continuing effort to obtain reference data on attenuation in solidifying alloys has been focused during FY 1991 on hardware construction. A custom-designed gated amplifier has been constructed for generating high-power ultrasonic pulses up to 100 ms duration. A new vacuum furnace insert is also being constructed which will facilitate ultrasonic measurements through the melting transition of steel.

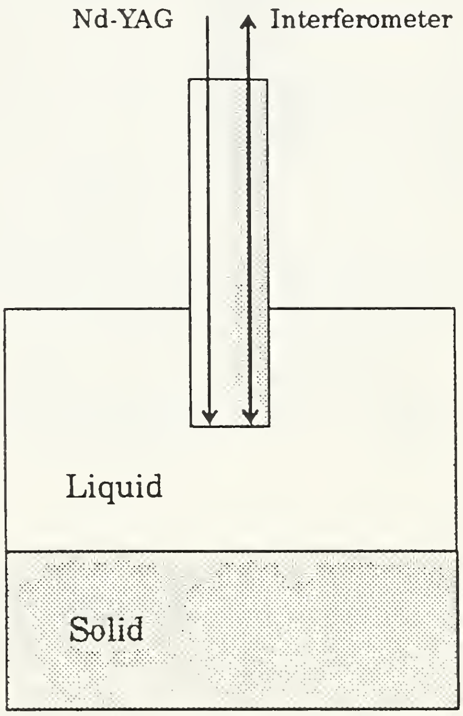


Fig. 2 Laser-ultrasonic configuration consisting of a Nd-YAG pulsed laser for ultrasonic generation and an interferometer for detection where laser light is passed through a transparent rod to the liquid metal.

## Reference

1. W. L. Johnson, S. J. Norton, F. Bendec, and R. Pless, "Ultrasonic Spectroscopy of Metallic Spheres Using Electromagnetic-Acoustic Transduction", submitted to the Journal of the Acoustical Society of America.

## Physical and Mechanical Metallurgy and Sensor Technology Development for Intelligent Processing of Soldered Assemblies


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During fiscal year 1991, research on several topics related to processing and inspection of soldered joints for electronic components was continued under sponsorship of the U.S. Army Harry Diamond Laboratory. This manufacturing technology support is ultimately directed to the reduction of cost in manufacturing reliable electronic components, and in this fiscal year was entirely focused on developing a scientific base in the areas of solder intermetallic compound metallurgy and inspection technology.

The intermetallic compounds of interest in soldered joints form by reaction of copper (or a copper-rich compound) with liquid tin, and thus usually exist in the form of thin layers or fine needles at the interface between the copper and the solder. To make materials composed almost entirely of the desired compounds, it is necessary to rapidly solidify a melt having the composition of the desired compound. The rapid solidification prevents the constituents of the melt from segregating into copper-rich and tin-rich regions. During FY 1991, Biancaniello and Schaefer produced a melt of 60 wt.% Sn, 40 wt.% Cu ($\eta$ phase composition) by inert gas atomization using the NIST Supersonic Inert Gas Metal Atomization System. Several kilograms of powder had the particle size distribution shown in Figure 1.

Even when rapidly solidified, the powder microstructure contains three phases ($\epsilon$, $\eta$, and Sn). The fraction of phases present in the as-atomized powders, as a function of particle size, was investigated by x-ray diffraction. The smallest, and thus most rapidly solidified, powders show the highest fraction of the $\eta$ phase, while the largest particles show a large fraction of the $\epsilon$ phase. The volume fraction of the Sn phase appears to be almost independent of particle size. During a heat treatment such as that experienced in hot isostatic pressing at 300°C, these powders transform to essentially single-phase $\eta$. From a portion of the powder produced in this run, the 25 to 45$\mu$m size fraction was separated by sieving. This size fraction showed considerably better flow properties than did the general cut of the powder, and it was supplied to researchers at the U.S. Army Harry Diamond Laboratory for studies of composite solder.

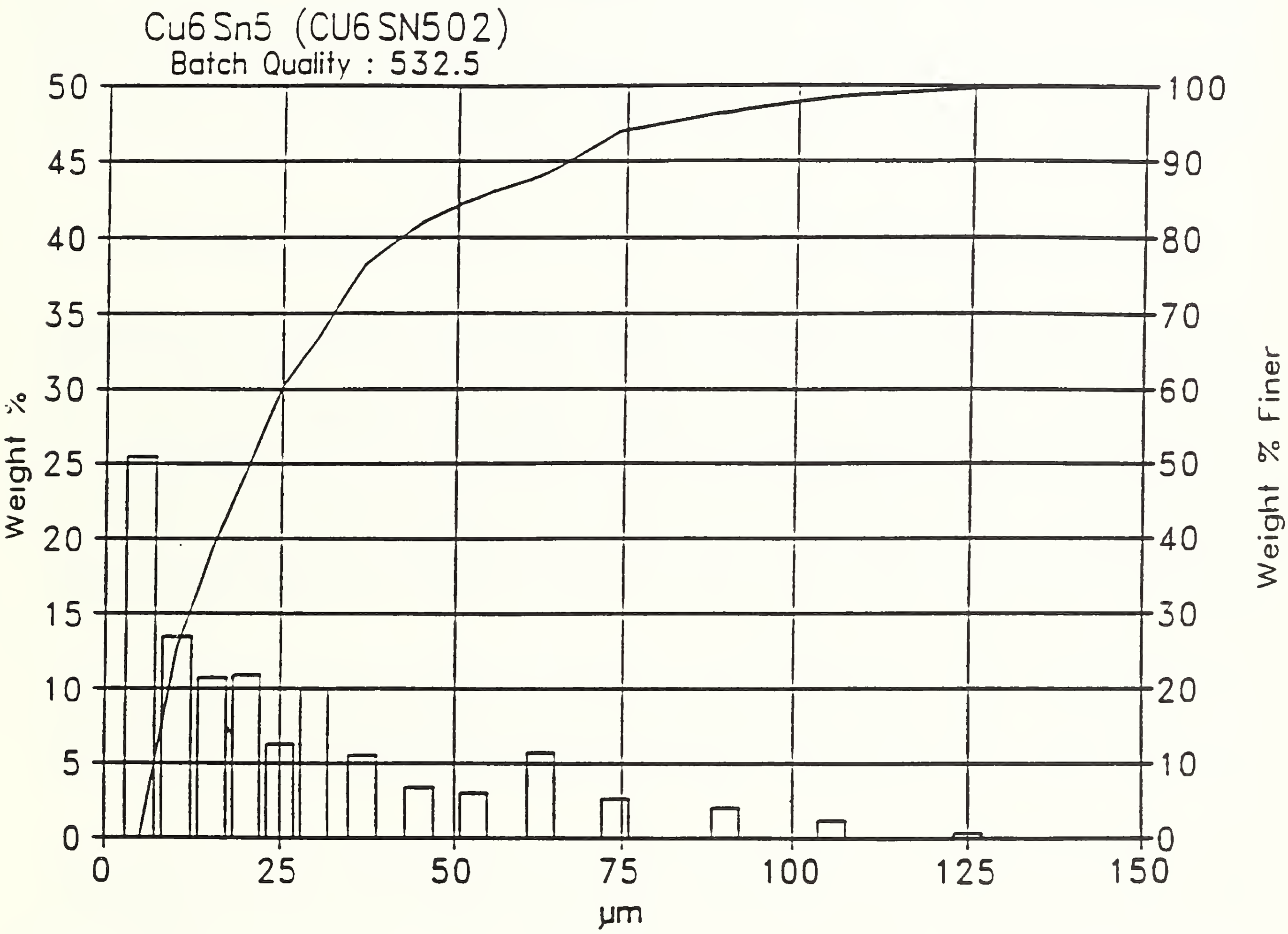


Fig. 1 Particle size distribution for atomization of $Cu_6Sn_5$

In 1990, Clough and Patel reported the successful preparation of composite solder utilizing a fine dispersion of $Cu_6Sn_5$ powder in a melt of eutectic Pb-Sn solder. Initial evaluation of mechanical properties of lap joints prepared with this solder were encouraging, and this investigation was continued in 1991. Composite solder can be expected to improve the mechanical performance of soldered joints by increasing the flow stress. The addition of intermetallic Cu-Sn particulates can also, in principle, inhibit growth of the intermetallic at the copper substrate interface through an equal reduction in the amount of free tin available in the solder matrix. Both of these effects can inhibit the formation and growth of thermal cycle-induced creep fatigue cracks.

Figure 2 shows the flow stress as a function of reinforcement level for a variety of processing methods. The processes that produced lower porosities yielded stronger composites. Results in this phase of research showed that property improvements were not realized if appreciable porosity (10% or more) was present. The porosity in powder solders is produced by residual flux, which coats each particle and is inhibited from escaping due to Stokes Law for particulate (or bubble) motion in viscous fluids. The hardening mechanism for particulates of this size (2 $\mu$m or greater) is a combination of initial thermal stresses from cooling and Eshelby-type (rule of mixtures) hardening on straining. The low volume fraction porous materials obey Eshelby-type hardening predictions, but there is no theory available to predict the lack of hardening in the porous composites, since at higher volume fractions the stress fields begin to overlap.

Thermal cycling tests which include monitoring with acoustic emission to detect the onset and growth of creep fatigue cracks are being conducted.

Wheeler, Sarafidis, and Lashmore developed a non-cyanide Cu-Sn electrolyte from which deposits containing approximately 35 to 45 atomic-% tin have been electrodeposited. These compositions were deposited onto a copper-coated mild steel panel using a Hull-cell, an asymmetric electrochemical cell in which the anode and the sample panel are not parallel but positioned at an angle to one another. This arrangement sets up a current density gradient across the panel. Thus, if the tin composition varies with current density, it will also vary with position on the panel. The Cu-Sn deposition was followed by deposition of tin and, finally, annealing at 200°C for 24 hrs. in a vacuum. Preliminary metallographic examination showed that one portion of the panel was much less friable during mechanical polishing, suggesting that this material might be more ductile. Further research is in progress to determine the factors that preserved the structural integrity.

DeWit developed an approximate calculation of the stresses and strains at a solder/intermetallic interface due to the growth of intermetallic particles of $Cu_6Sn_5$ from a copper substrate into tin. Initially, this problem was modeled as the growth of a spherical inclusion in a large body of tin. Two extreme assumptions were made for the growth.

> (1) The inclusion grows by simply adding copper to it and forming an additional $Cu_6Sn_5$ layer from the reaction with the surrounding tin. In this case the inclusion has a uniform dilatation of $e_0 = 0.432$.
>
> (2) The inclusion grows by replacing every five tin atoms around the inclusion with six copper atoms. In this case the uniform dilatation is $e_0 = -0.0597$.

In both cases, the calculated strains were large enough to produce plastic flow around the inclusion. For a perfectly plastic material, the calculated plastic zone around the inclusion for case (1) is about eight times the inclusion radius and for case (2) about four times. In the plastic zone, the difference between the circumferential and radial stress equals the yield stress. Outside the plastic zone the stresses drop off with an inverse cube dependence.

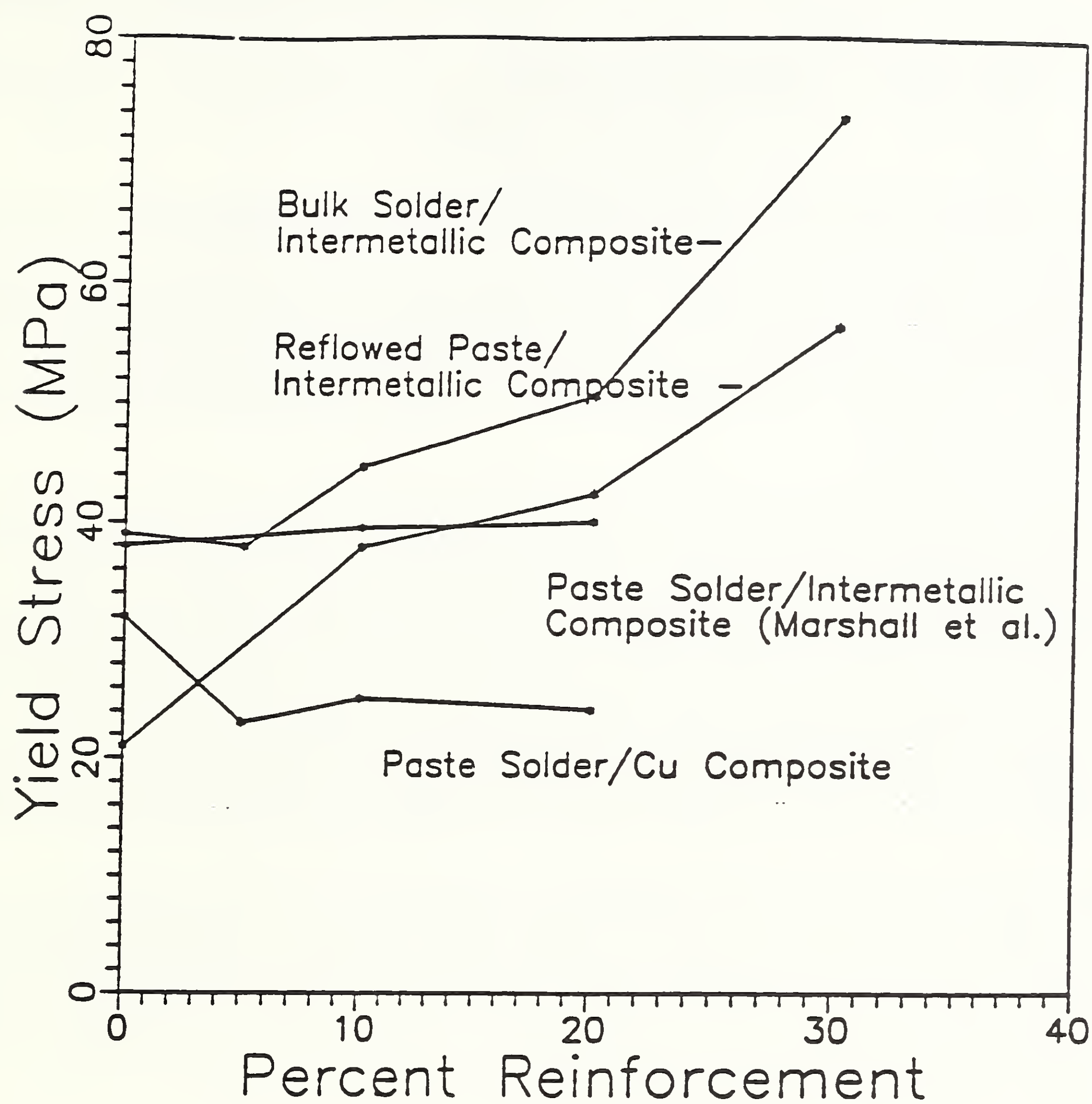


Fig. 2 Compressive yield strength of particulate composite solders as a function of reinforcement level

Dewit and Boettinger also addressed the moving boundary value problem of the diffusional growth of the copper-tin intermetallics at a flat copper-tin interface. When the copper and tin are joined together at a flat interface, the intermetallics $Cu_3Sn$ and $Cu_6Sn_5$ begin to form and grow. The alloy concentration in each of the four phases is then determined by the diffusion equation. The solution is a linear expression in the error function, which depends on the position, the square root of the interdiffusion coefficient, and the square root of the time. Two constants of integration are determined by the concentrations at the phase boundaries found from the copper-tin phase diagram. The motion of the three phase boundaries was calculated by the solution of three nonlinear boundary value equations for an assumed temperature of 200°C. The phase boundary positions in meters as functions of time, t, in seconds are calculated as follows:

| | |
|---|---|
| $x = -6.2 \times 10^{-9}\sqrt{t}$ | for the $Cu/Cu_3Sn$ boundary |
| $x = -3.2 \times 10^{-9}\sqrt{t}$ | for the $Cu_3Sn/Cu_6Sn_5$ boundary |
| $x = 3.9 \times 10^{-9}\sqrt{t}$ | for the $Cu_6Sn_5/Sn$ boundary |

The $Cu_6Sn_5$ is predicted to grow about twice as thick as the $Cu_3Sn$ at this temperature.

Siewert and Austin continued their evaluation of the technical merits of X-Ray laminographic inspection of circuit boards, focusing on development of measurement standards based on fundamental units. They procured a commercial laminography system to measure its suitability for Department of Defense circuit board inspection. This system was also sent to other government contractors for evaluation in circuit board inspection applications.

To develop measures of the system performance, a series of 300-mm-square boards (similar in size to the circuit boards normally inspected with the system) populated with devices was constructed. Some boards included a series of holes that were used to measure the X and Y dimension resolution of the system. Independent optical measurement of the hole positions formed the reference for the measurement. This prototype X and Y dimension calibration board is being evaluated in a round robin, which includes a variety of laminography systems. These users include electronic board producers (with both government and industrial contracts), as well as the manufacturer of the laminography system. Upon successful completion of the round robin test, plans call for a series of these boards to be constructed, calibrated, and distributed through the Office of Standard Reference Materials to users of laminography systems (or other automated x-ray systems) as a standard device for system calibration.

Siewert and Austin also developed a new type of image quality indicator for measuring the Z dimension resolution. A different board contained an x-ray line pair gage. Images of this gage were sent to an image processor for production of a plot of image contrast versus line spacing. This contrast transfer function provided a more precise characterization of the system performance than a single value of spatial resolution (at an arbitrary contrast value). Finally, development of a solder thickness gage for calibration of the gray-scale image of an x-ray system was initiated.

A series of thermal and electrical stress tests of the commercial laminography system was performed while monitoring the system performance by the X and Y dimension resolution and the contrast transfer function. Within the 19.7° and 29.2° C (67.5 and 84.5° F) operating range specified by MIL-STD-1695, the system functioned as specified. In voltage stress testing, the system continued to operate over the 208 V $\pm 10$ % range specified by the manufacturer. At both the high and low voltage extremes, the system failed gracefully, losing detector rotation at high voltage and stage motion at low voltage. The system recovered from both failure modes easily and without damage.

During FY 1991, Read continued development of analysis methodologies for real-time assessment of the reliability of surface mount technology (SMT) solder joints based on fitness-for-purpose assessment. The key inputs to the complete quality assessment are the actual solder joint geometry, derived from recently developed x-ray and optical inspection technologies; the printed wiring board geometry, available from specifications; the material properties, and the service conditions. These input data are combined to characterize the quality of individual solder joints through a fitness-for-purpose assessment strategy.

The first computer-generated finite element meshes, based on sets of perimeter points supplied to us by the vendors of the inspection systems, were produced late in FY 1990. Two trial data sets were meshed, one from an x-ray laminography system and one from a machine vision system. The first result for FY 1991 was that the finite element analysis (FEA) successfully calculated stresses from the computer generated meshes. Several analyses on the original mesh and variations were performed. The resulting stress values were well-behaved and plausible.

When this work was presented to industry representatives early in FY 1991, they pointed out that the analysis of x-ray laminography images to produce perimeter points was a key part of the procedure, and that this research did not at that time address this operation. Read then developed software to locate perimeter points of objects imaged by laminography. Perimeters were defined by a selected x-ray intensity value or by the maximum intensity gradient. Results from tests on artifacts supplied by Harry Diamond Laboratory show that the gradient criteria for perimeter location is better than the threshold criteria for relatively sharp images.

The most difficult aspect of automated mesh generation was the treatment of intermediate features, such as pores and cracks, on the solder joint sides, especially making them connect vertically to one another properly. The original program handled the trial solder joint, but could not handle joints with large section changes, such as those for leadless chip carriers. To remedy this problem, the program was revised to incorporate more detailed information on the overall structure. Read also developed an alternate method of interpreting FEA results that can compensate for the absence of human engineering judgement in the refinement of the finite element mesh at high-stress points. By an application of the J-integral originally suggested by J. R. Rice, the stress-strain concentration at a notch root can be accurately estimated if the radius of curvature is known. The stresses and strains at the notch root are insensitive to the fineness of the mesh. These results also provide justification for using convenient choices of material properties to lighten the computational burden.

The scanning acoustic microscope was utilized by Drescher-Krascika for detection of the intermetallic layers in electronic solder joints samples of intermetallic compounds at the interface between copper or nickel and tin or lead/tin alloys. Previous studies utilizing metallography and scanning electron microscopy (SEM) have shown that $Cu_3Sn$ forms next to the copper, and $Cu_6Sn_5$ forms between $Cu_3Sn$ and the lead/tin alloy. Feasibility tests were performed at two different frequencies: 1 GHz, in cooperation with Professor Bernhard Tittmann from Pennsylvania State University on an "Olympus" Acoustic Microscope, and at 30 MHz on "Sonex" Acoustic Microscope in our Acoustic Microscope Laboratory at NIST. This study utilized bulk intermetallic samples for ultrasonic measurements to determine the elastic properties of the single phases of the intermetallics (absolute velocity measurements). Independent density measurements allowed evaluation of the acoustic impedances of these materials.

One result obtained at 1 GHz frequency is shown in Figure 3. The acoustic image was taken at 6.5 $\mu$m below the surface of the solder joint sample. Figure 1 shows the grain structure of intermetallic $Cu_6Sn_5$. The B-scan technique (x-z) measures the thickness of the intermetallic layer at the interface between copper and tin, and the thickness measured at high frequencies by the acoustic microscope technique agrees well with the destructive metallographic results. The feasibility test at much lower frequencies revealed the intermetallic layer using the echo technique.

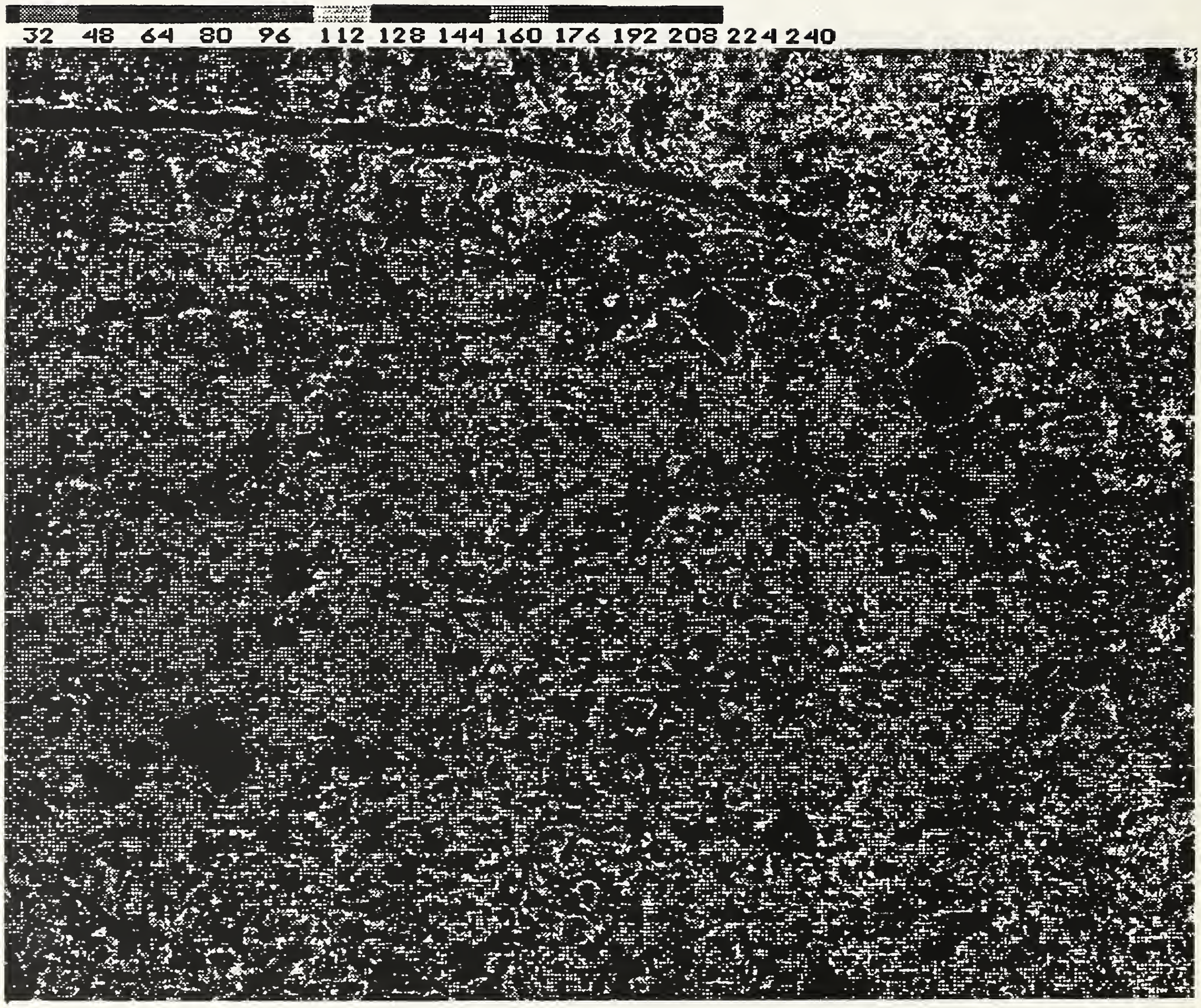


Fig. 3 Scanning Acoustic Micrograph of Solder Interface 6.5 $\mu$m under the emitance of tin, 100 X 75.

Recognizing that no calibration standard is available for the scanning acoustic microscope, old, current, and new procedures and techniques for calibrating individual acoustic devices were evaluated. Evaluations with ball and point targets to characterize beam diameter and depth of field, flat bottom holes to calibrate penetration depth and resolution, surface roughness gages to determine resolution and depth of field, stage micrometers and precision drilled holes for x-y system calibration, and line pair gages along with the modulation transfer function to determine x-y system resolution were accomplshed. This preliminary work is intended to lead to the design and manufacture of a calibration standard to be used to evaluate the system performance and repeatability.

## Publications

R. J. Schaefer, F. S. Biancaniello, and R. D. Jiggetts, "Preparation of Bulk Samples of Copper-Tin Intermetallic Compounds", submitted for publication in symposium on "The Metal Science of Joining" at TMS 1991 Fall Meeting, Cincinnati, Ohio: Oct. 20-24, 1991.

J. S. Hwang, G. Lucey, R. B. Clough and J. Marshall, "Futuristic Solders - Utopia or Ultimate", Surface Mount Technology, to be published, 1991.

T. A. Siewert, M. W. Austin, G. K. Lucey, and M. J. Plott, "System Evaluation and Qualification Standards for an X-ray Laminography System", submitted to Circuits Assembly, 1991.

D. T. Read and G. K. Lucey, "Automated Finite Element Mesh Generation for SMT Solder Joints", accepted for publication in the Journal of Electronic Packaging.

## B. NONDESTRUCTIVE EVALUATION PROGRAM

The goal of this activity is to develop generic approaches, sensors, and procedures for quantitative NDE of (1) metals, polymers, and ceramics; (2) composites/interfaces; and (3) measurement standards and methods. The emphasis in (1) and (2) is on measurements that can be made during critical stages of the material/product formation and to provide the data to control the process to optimize quality and productivity. Since the knowledge base on composites/interfaces (e.g., thin films) is far from complete, we expect that a portion of this activity will be concerned with relating important composite/interface characteristics with performance and then developing monitoring methods. The activity devoted to measurement standards and methods (3) has been concerned primarily with the requirements of post-manufacturing and in-service inspection, although it is expected that the need for testing the performance of sensors used in process monitoring will become important. This included research on using eddy currents to measure the internal temperature of aluminum during processing and to monitor the pyrolysis of carbon-carbon composite sheets during high temperature processing; developing capacitive and microwave probes for characterizing dielectric materials; and developing in-line measurement technology based on fluorescent probes to monitor important polymer processing parameters. In addition, a variety of other techniques were explored: light scattering techniques for inspecting coated metal interfaces for defects; measurement of dispersion of ceramic powders in dense slurries using electroacoustics; application of NMR imaging as a diagnostic technique for ceramic materials; and the use of transient elastic waves for the evaluation of composite materials where interfacial conditions are important. Also, included is the following work on measurement and standards methods: x-ray radioscopy, thermography, eddy current and ultrasonic standards.

- The method employed to detect important polymer processing parameters involves the detection of fluorescence spectra from fluorescent dyes which have been doped in very small concentrations into the processed polymeric material. Such measurements were used to determine fluorescence anisotropy on applied shear stress and in the study of fluorescence behavior to detect phase transitions and temperature in polymers during processing. A successful NIST/industry workshop was organized from which the nucleus of a NIST/industry consortium has evolved for the purpose of developing in-line measurement technology based on optical and fluorescent methods.

- The feasibility of eddy current sensing of carbon-carbon composites during processing was investigated. It was found that sensing the resistivity of carbon-carbon sheets by through-transmission eddy current methods at temperatures up to 850°C is feasible and practical. Furthermore, a simple model for describing the temperature dependence of the electrical conductivity of carbon-carbon was proposed.

- Work proceeded to model the behavior of a non-contact capacitive (electrostatic) probe and a microwave probe (open-circuit coaxial), and to perform laboratory experiments to demonstrate their practical utility. A model was developed to determine the effect of inhomogeneities in dielectric properties on the performance of the capacitative probe. Analysis of the coaxial probe was initiated to allow separating out the effect of lift-off from the measurement of complex dielectric constant. Samples of green sand obtained from a General Motors (GM) Foundry were prepared with known moisture content. Measurements with the capacitative and coaxial probes demonstrated a strong correlation of moisture content. However, it is necessary to have an appropriate dielectric mixing rule which relates to the ratio of water to sand. The resulting value of moisture content would then be an input to an existing system which controls the amount of water added to the sand.

- A long term successful program on eddy current standards and methods involving investigators at NIST and Stanford University is being terminated this year. Efforts this year focused on producing an empirical model for predicting probe sensitivity by winding coils with very specific construction parameters and using a statistical analysis to determine the functional dependence of probe sensitivity on these parameters. In another direction, a technique using a differential eddy current sensor was developed to monitor the formation and growth of fatigue cracks.

## Temperature Measurement in Aluminum Processing

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The objective of this project is the development of a non-contact method of measuring the internal temperature of aluminum during processing operations such as rolling and extrusion. Eddy current impedance measurements can determine the electrical resistivity of the material under test by non-contact electromagnetic induction of current in the material. The resistivity, in turn, depends upon the temperature of the product. If the temperature dependence of the resistivity of the alloy of interest is known, then eddy current methods can be used to provide measurement of temperature during the processing [1,2].

A limitation of this approach arises from variations in the composition of the alloys being processed. Corresponding to the allowed range of concentrations of elements within an industry specification for an alloy is an attendant resistivity variation. In turn, this variation will produce a corresponding shift in the temperature reading if no correction is made for the composition variation. The principal problems now being addressed are the determination of the basic accuracy of the eddy current system and establishment of limits on the apparent temperature shift associated with variations in composition and degree of solid solution of alloying components in commercially specified grades of aluminum.

The eddy current method employed in this period used through-transmission coil geometry in the examination of rolled sheet material. One coil above the sample acted as a driver, inducing currents in the test sample, and a second coil below received a signal influenced by the test material. Measurements of the ratio of the magnitude and phase difference of the receiver voltage relative to the driving current provided the information for calculating the impedance. The measurements were made at 200 Hz, which was approximately equal to the frequency of maximum dissipation of the impressed excitation, the vicinity of greatest sensitivity. It was found empirically that a parameter related to the complex impedance of the sample-coil system, varied linearly with temperature over a range of 200°C. This linearity provided a calibration of the eddy current system.

A series of tests was performed on samples of aluminum alloy designation 3004 with eight concentration variations of the elements Mn, Mg, and Cu. The samples were heated in a furnace to 500°C and then transferred to the sensor, which was at room temperature. During the subsequent cooling, readings were recorded by the eddy current sensor and by a thermocouple embedded in each sample. A statistical analysis of the experimental results was conducted by Dr. Eric Lagergren of the Statistical Engineering Division of NIST. For these research samples it was found that the standard deviation of eddy current temperature measurements on similar composition specimens was 7.7°C; the corresponding standard deviation for repeatability on the same specimen was 2.9°C. Sensitivities to the individual elemental variations were also determined.

The target 1-sigma variation aimed for in this project was 2°C. Rather than attempting to do a more controlled laboratory experiment to achieve the desired improvement, NIST members and the Task Group of the Aluminum Association decided that the best procedure from this point would be the performance of a hot test on long coils of sheet aluminum passed through an annealing furnace in which the temperature of the product would be steady. Also, the effects of velocity of the product could be studied [3]. The test will be performed by the invitation of Alcan, Ltd. at its annealing facility at Kingston, Ontario. Various member companies of the Aluminum Association will supply coils of specified grades and composition ranges for a statistical test of temperature reading variation. Speed and temperature will be varied and thickness will be monitored. Assistance with engineering installation and data collection will be provided by the Data Measurement Corporation (DMC) on contract with the Aluminum Association.

The Aluminum Association represents many U.S. and Canadian companies. The Association contributed financial support in the form of a Cooperative Research Associate and through its contract with DMC. Member companies supplied materials and have invited NIST researchers to their operating facilities for testing. Efforts to commercialize the sensor and broaden applicability are continuing.

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## Fluorescence and Optical Monitoring of Polymer Processing

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The objective of this program is to develop in-line measurement technology based on optical measurement methods to monitor important polymer processing parameters. The major optical method employed involves the detection of fluorescence spectra from fluorescent dyes which have been doped into the processed polymer material. The character of the fluorescence, i.e., its intensity, polarization, and wavelength distribution, yields information about the state of the polymer matrix. Efforts have concentrated on developing concepts and methods to measure molecular orientation, shear and extension stress, shear and extension strain rate, non-Newtonian viscosity, velocity, flow instabilities, quality-of-mix of ingredients, residence time distribution, the onset of the glass transition, and the liquid to crystal transition. Work on quality-of-mix, residence time distribution and flow instabilities, carried out in collaboration with the Naval Surface Warfare Center (NSWC), has been completed [1,2]. In collaboration with Drexel University, fluorescent probes are now being used to monitor temperature and phase transitions. Significant progress at NIST has been made in developing the relationship between fluorescence anisotropy and shear stress, non-Newtonian viscosity, and molecular orientation.[3]

Over the past year the specific objectives of this program have been to develop a model of the relationship between fluorescence anisotropy and molecular orientation factors, to examine the temperature dependence of fluorescence from molecular rotor dyes and excimer producing dyes, to conduct a workshop on new measurement technology for polymer processing involving the participation of industry scientists, and to establish a NIST/industry consortium to develop in-line measurement technology for polymer processing. Three significant achievements are summarized.

- The measurement of the fluorescence anisotropy of a tagged polymeric probe molecule doped into a polymer matrix and its dependence on applied shear stress. A model to describe this observation was developed.

- The investigation of fluorescence behavior as a function of temperature for molecular rotors and for excimer producing dyes and the use of these fluorescent dyes to detect phase transitions and temperature in polymers during processing. This work is being carried out in collaboration with Drexel University.

- The administration of a successful NIST/industry workshop from which the nucleus of a NIST and industry consortium has evolved for the purpose of developing in-line measurement technology based on optical and fluorescence methods.

### Fluorescence Anisotropy Measurements

This measurement involves the use of polarized light to determine the anisotropy of a fluorescent dye molecule which has been doped into a polymer matrix at low concentrations. The objective is to determine shear stress, molecular orientation, and non-Newtonian viscosity. Laboratory confirmation of this measurement concept and an extensive experimental study of anisotropy using a polymeric fluorescent dye have been carried out [3]. The polymeric dye (or molecular probe) is polybutadiene tagged with anthracene which was synthesized at NIST. The dye was doped into a polybutadiene matrix and anisotropy was measured as a function of the applied shear stress using an optically instrumented rheometer. It was found that anisotropy decreased with increasing shear stress, an observation opposite to that which occurs when the polymer was stressed in extension. The difference between the shear and extensional effects was examined by developing a model for the relationship between anisotropy, molecular orientation factors and shear stress. It was found that, if a reasonable orientation distribution function $f(\theta,\phi)$, $\theta$ and $\phi$ being polar coordinates, is assumed, then the model predicts that anisotropy decreases with applied shear stress.

## Fluorescence Dependence on Temperature

Two types of fluorescent dyes have been used in this study, an excimer producing dye and a molecular rotor. The excimer producing dye, bis-(pyrene propane) (BPP), consists of two fluorescently active pyrene rings separated by a flexible trimethylene link. When one pyrene molecule absorbs excitation energy at 340 nm, two paths of fluorescence decay are possible, one, by monomer decay at 380 and 400 nm, i.e., the pyrene molecule displays its characteristic fluorescence without interaction with the other pyrene, and two, by excimer decay in the range 450 to 550 nm which occurs when the excited pyrene forms an excimer with its unexcited pyrene neighbor by rotating to a position of close molecular contact. The probability that the excited pyrene can rotate into the proper position to form an excimer before its own decay occurs is dependent on the $\tau_r$, the intramolecular rotational relaxation time of the dye, which is proportional to the ratio $\eta/T$, where $\eta$ is a microscopic or molecular viscosity and T is temperature. Fluorescence decay from molecular rotor dyes is also influenced by its intramolecular rotational relaxation. Upon absorbing excitation energy, a molecular rotor can decay to its ground state via fluorescence radiation or by radiationless decay, i.e., energy transferred to molecular vibrations or rotations. The amount of radiation less decay is regulated by intramolecular bond rotations. Intramolecular rotational motion creates potential radiationless decay paths if $\tau_r$ is shorter than, or approximately equal to, the fluorescence decay time, $\tau_f$, of the dye, usually tens of nanoseconds. For $\tau_r >> \tau_f$, maximum fluorescence radiation and minimum radiationless decay is observed. Since $\tau_r \propto \eta/T$, fluorescence intensity will be a function of temperature. Thus, for excimer producing dyes as well as for molecular rotors, rotational relaxation time plays a prominent role in the production of fluorescence radiation.

The temperature dependence of the ratio of excimer to monomer fluorescence intensity for BPP doped into polystyrene is shown in Figure 1. The onset of the glass transition, $T_g$, is clearly indicated by the knee in the curve at 105°C. Below $T_g$, the lack of rotational mobility inhibits the formation of excimers, but as the temperature increases above $T_g$, the excimer fluorescence intensity increases. These data indicate how BPP can be used as a phase transition marker and a temperature-sensing probe.

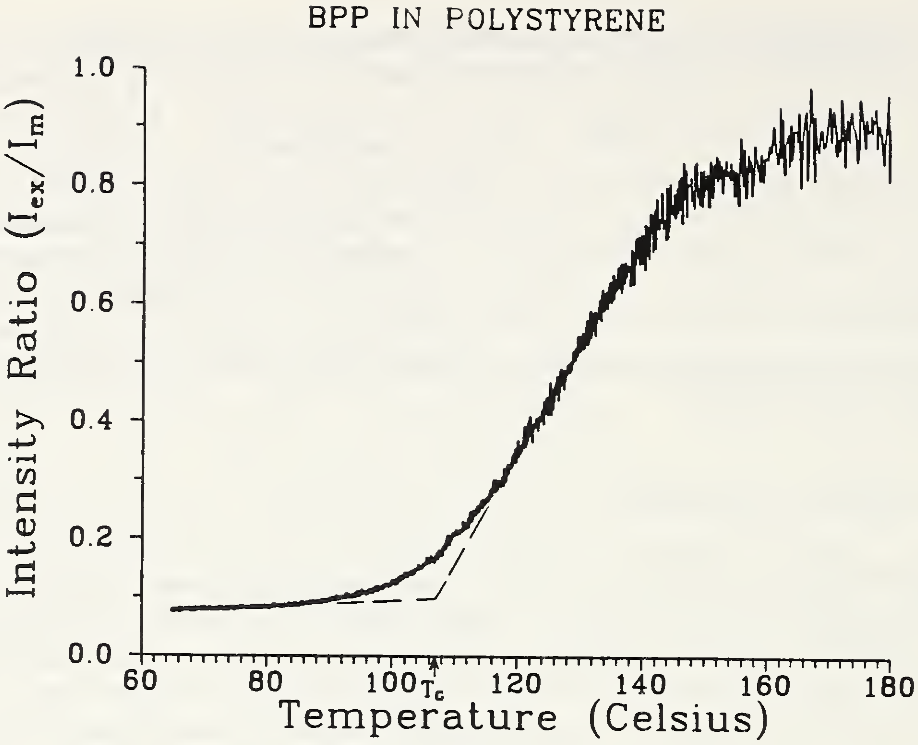


Fig. 1 The ratio of excimer to monomer fluorescence plotted versus temperature for polystyrene doped with BPP and exited with an excitation wavelength of 340 nm.

Workshop on New Measurement Technology for Polymer Processing

This workshop was held at NIST on December 3-4, 1990 [4]. In attendance were nineteen industry scientists and engineers from polymer processing and instrumentation manufacturing companies. The objectives of the workshop were to identify what on-line, real-time measurements during polymer processing are required but are not possible because the measurement technology does not exist, and to plan a collaborative NIST/industry research program for the development of new measurement technology. Processors identified their measurement problems by describing various processing operations. Tire and rubber manufacturing, thermoplastic compounding, injection molding, film processing, and reactive processing were described with respect to related measurement problems. Workshop discussions, yielded a consensus of the following measurement needs: (a) in-line rheological measurements, (b) improved in-line temperature measurements, and (c) in-line and on-line measurements of polymer morphology. The direct interaction with industry at this workshop has provided the framework for establishing a NIST/industry consortium dedicated to the development of new in-line measurement technology for polymer processing.

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## Coatings Technology Consortium

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The objective of this program is to establish a consortium to develop automated electro-optical methods for inspecting coated metal surfaces for defects. These include orange peel, craters, bumps, dewetting, haze, sagging, seeds, and solvent popping.

The presence of defects on painted metal surfaces is an important industrial problem, particularly in industries like the automotive and appliance industries, where the appearance of a product is a primary component of consumer acceptance. Defects in the painted surface can ruin a car's appearance and, in some cases, can lead to corrosion of the metal below. Defect detection is a time consuming task that in general is performed by human inspectors.

The development of automated on-line inspection techniques would be desirable from several points of view.

- The automated system could work in more hostile environments than human inspectors and closer to the sources of problems.
- The measurements and standards could be better quantified.
- Automated inspection would enable the development of detection and feedback loops for on-line coating application process control.
- As a result of the improved process control, the materials and time wasted on reworking processes could be reduced.

Recent advances have taken place in analysis of the images of defects at NIST. For example, a method was developed to quantify the severity of orange peel and crater depths with a vision system and to correlate the results with topographic parameters measured by using stylus instruments. The excellent correlation of these measurements points the way to two types of advances: (1) the development of image analysis tools for other types of defects as well, such as haze, sagging and seeds; and (2) the automated detection and quantification of these defects in terms of real physical parameters, such as surface waviness or crater depth, rather than human subjective evaluation by comparison with visual standards.

The consortium will involve research on techniques for automatically detecting and quantifying defects in coatings on metal. The planned tasks include the following.

(1) Development of machine vision hardware and software for detecting and quantifying a wide range of coating defects.

(2) Correlation of vision sensor results with objective measures obtained with surface profiling instruments.

(3) Development of high speed vision processing to enable the inspection of large components in real time.

(4) Installation of the defect sensors on robot motion systems to enable the automated detection of defects on curved surfaces such as automobile panels.

(5) Development of feedback loops for on-line process control of the coating operation.

The overall goal is the application of the results to inspection of painted surfaces on shop floors.

A workshop was held on May 9 and 10, 1991, at NIST, to establish a consortium on "On-line Detection and Control of Coating Defects". Thirty-seven participants, representing approximately twenty companies, attended the workshop. During the two day workshop, NIST researchers made presentations demonstrating the feasibility of detecting coating defects in a production line. From discussions following the presentations, however, it was clear that the objectives of the proposed consortium were too narrow. It was recommended, therefore, that a small committee, comprised of a cross-section of the industries represented at the workshop, be convened to discuss alternative consortium objectives and the output from this small committee could then be used as the basis for proposing a new consortium. This small committee met on August 7, 1991 and, from it, a new consortium, entitled the "Coating Technology Consortium" with much broader scope and objectives, was proposed. The objectives of the proposed consortium will be to develop technologies, methodologies, and procedures for:

(1) incoming material characterization, measurement, modeling, and control;

(2) painting process characterization, measurement, modeling, and control; and

(3) defining, measuring, and standardizing appearance and other quality attributes of the finished product.

The committee recommended that another consortium organizational meeting be held at NIST in December 1991. Extensive interactions were made with industry prior to, during, and after the consortium workshop. The industries with which NIST interacted can be divided into three groups including (1) coatings companies; (2) automotive, appliance and other end-users of coatings; and (3) equipment and computer companies. The coating companies included PPG, Glidden ICI, DuPont, AKZO, BASF, and Sherwin-Williams. The automotive, appliance, and other end-users of coatings included General Motors (GM), Ford, Caterpillar, and A.O. Smith. The equipment and computer manufacturers included Hughes, Nordson, Diffracto, Data Measurement Corporation, Aspect, and DataCube Corporation.

**Measurement of Dispersion of Ceramic Powders in Dense Slurries Using Electroacoustics**

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The primary objective of this research program initiated in FY 1991 is to develop an electrokinetic sonic amplitude (ESA) technique for on-line measurement of powder dispersion in aqueous suspensions.

The ESA of ceramic powders on a laboratory scale is measured by placing the probe in a suspension. This measurement is found to be directly related to electrophoretic mobility, an important surface chemical property of the particles in liquid environments. In the past few years, the ESA system has been extensively studied for the measurement of the isoelectric point ($pH_{iep}$, a pH at which the particles carry a net zero charge) of ceramic powders in aqueous suspensions, containing up to 10% by volume of solids [1]. These studies have shown excellent correlations between the $pH_{iep}$ measurements obtained from electrophoretic techniques and those from the ESA technique [2]. However, very little theoretical or experimental understanding of the interrelationships between the ESA and electrokinetic (especially zeta potential) measurements has been developed. This information is crucial in extending the ESA measurements for the determination of dispersion of powders in liquids.

The ESA measurement is related to the high frequency mobility, $\mu_d(\omega)$, of the particles as follows (3):

$$ESA(\omega) = c \, \Delta\rho \, \phi \, G_f \, \mu_d(\omega),$$

where c is the sound velocity in liquid, $\Delta\rho$ is the density difference between particles and liquid, $\phi$ is the volume fraction of particles, $G_f$ is the geometric factor for electrode geometry, and $\omega$ is the angular frequency. An improved understanding of the parameters of this equation, especially the high frequency mobility, is a key to the application of the ESA technique for dispersion measurements. Understanding and control of dispersion of submicrometer ceramic powders is critical in that it affects properties of green and densified ceramics. In practical suspensions, the powder dispersion is achieved by enhancing interparticle repulsion between the particles by the application of specific interactions, such as electrostatic, steric or electrosteric. Optimization of the influence of these interactions may reduce the tendency to form agglomerates.

In dense slurries, the particles tend to form agglomerates as the concentration of solids is increased. At close distances, the van der Waals attractive forces are dominant. Therefore, the interplay between these attractive and repulsion forces (electrosteric and electrostatic) is a key factor affecting the interaction of electrical double layer charges around the particles. These interactions determine the stability of particles in a suspension. The ESA is one of the few techniques that is known to provide an understanding of the complex interactions at the surface of particles in aqueous suspensions. Significant progress has been made at NIST in the ESA measurement of dilute slurries on a laboratory scale. The goal of this project is to extend this measurement to dense suspensions and, further, to the on-line monitoring of dispersion for intelligent processing of powders.

This project constitutes the following activities.

- Experimental investigation of interrelationships between the ESA and electrophoretic measurements and parameters affecting the ESA measurements.

- Collaborative effort between Matec Applied Sciences and NIST in the development of an on-line measurement system.

- Extending theoretical understanding of acoustophoresis measurements.

Experimental investigation of the ESA measurement was initiated with a model silicon nitride powder (SNE-3) from Ube Industries. Initial effort was directed towards complete characterization of the powder, dispersant and binder in an aqueous environment. The ESA measurements of polymethacrylic acid (PMAA) as a dispersant, and polyvinyl alcohol (PVA), and polyethylene glycol (PEG) as binders were carried out. Figure 1, which presents results of ESA measurements as a function of suspension pH, demonstrates the capability of the ESA system for measuring the response of small ionic species, and distinguishes ionic species (PMAA) from molecular species (PEG or PVA). These measurements were carried out at 3.2 weight % of SNE-3, 5 weight % of PMAA and 10 weight % of PVA or PEG in water. The variation in % weight was required for the development of sufficiently large ESA signal. These data show that PMAA exhibits a strong negative charge over the entire pH range investigated whereas SNE-3 has a definite $pH_{iep}$ at pH = 4.5. The PVA exhibited a mildly negative charge up to pH = 3.2. However, further decrease in the pH did not convert the surface charge. Additional experiments are in progress to understand the phenomena controlling the ESA variation at this pH range. The behavior of PEG is different from that of PVA. Throughout the pH range of interest, the PVA exhibits a slight negative charge. Currently, experiments are in progress to determine the effects of molecular weight and concentration of surfactants, and the concentration of powder on the ESA in aqueous environments at various pH. In addition, the study of interactions between water-powder-PMAA-PVA or PEG is also in progress using the ESA measurement.

The primary activities in FY 1992 will be directed towards establishing interrelationships between the ESA measurements of silicon nitride powders and their surface chemical properties in aqueous environments. In addition, the existing ESA system will be upgraded to enable the measurement of ESA at a number of frequencies and at high solids concentrations.

Collaboration with Matec Applied Sciences (MAS) has been initiated. In the near future, a formal Cooperative Research and Development Agreement (CRDA) will be established. Under this agreement, MAS is expected to provide hardware and software assistance in the development of an on-line measurement system. Theoretical understanding of the acoustophoretic measurements in dense suspensions will be developed in collaboration with Dr. Ray Mountain, NIST.

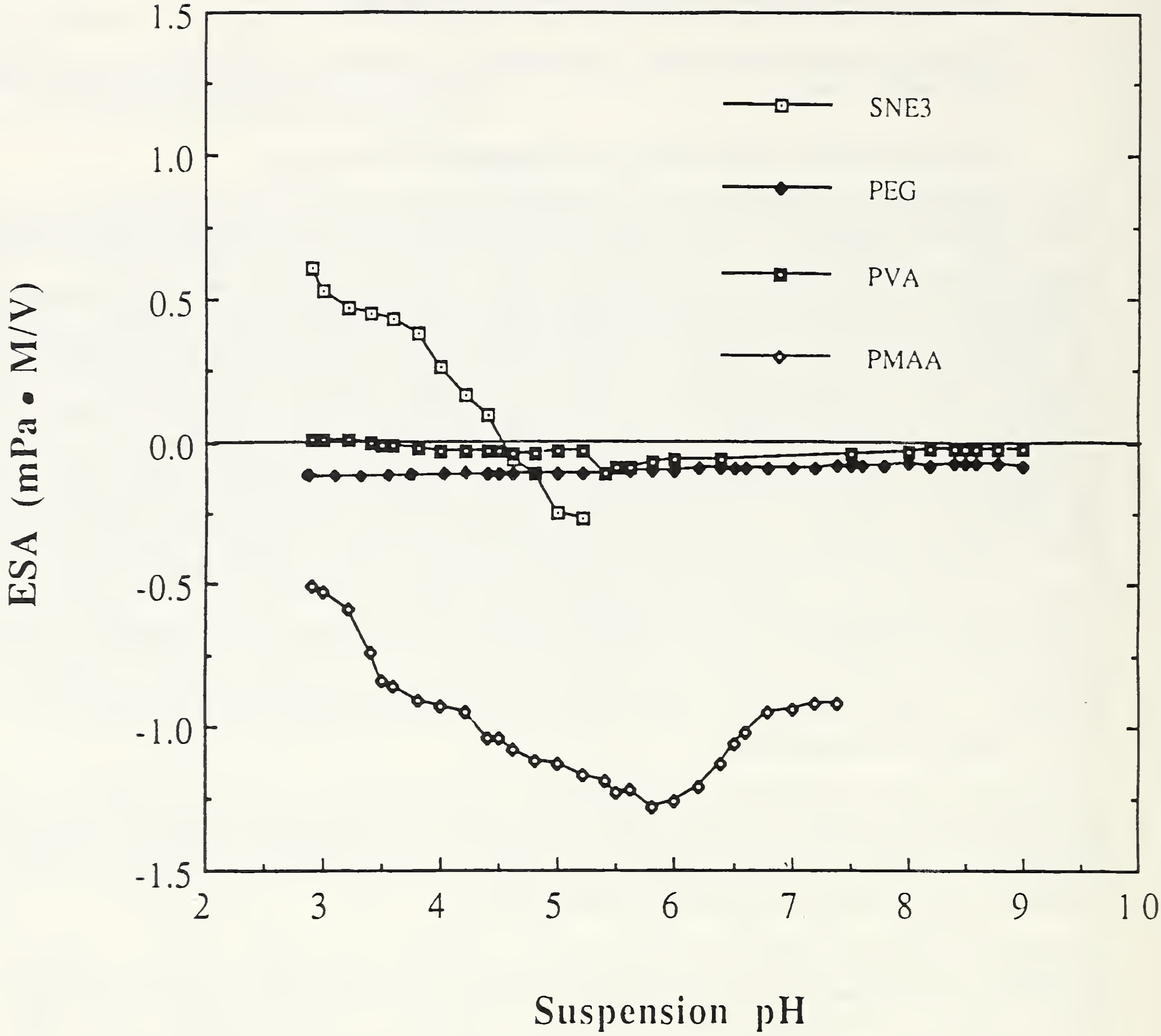


Fig. 1 Variation of ESA as a function of suspension pH for SNE-3 silicon nitride powder, polyethylene glycol (PEG, molecular wt. 3,400), polyvinyl alcohol (PVA, molecular wt. 50,000), and polymethacrylic acid (PMAA, molecular wt. 10,000).

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### Application of NMR Imaging Technique for Intelligent Processing of Materials

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In advanced materials, material failures often occur due to defects undetectable by the conventional methods. Nuclear magnetic resonance (NMR) imaging is an emerging technology which provides a unique material diagnostic technique by in-situ, internal mapping. It can provide information not only on the material distribution (nuclear spin density), but also on the chemical and physical characteristics of these materials (nuclear spin relaxation times). The binder distribution, for example, is a critical parameter in a ceramic green body that predicts its mechanical properties, thermal conductivity, and material compatibility. Understanding of the interfacial reaction between the binder and the ceramic powders under various processing parameters is essential to the intelligent processing of ceramic powders. The broader objective of this program is to establish a high resolution solid NMR imaging facility at NIST for use in developing models for the intelligent processing of materials.

The NMR imaging method uses a magnetic field gradient generated by a set of gradient coils to encode the positions of the nuclear spins with a spatially varying Larmour frequency. Once the variations in resonant frequency have been decoded, an NMR image can be constructed [1]. However, due to nuclear spin dipole-dipole interaction in the solid state, the NMR spectroscopic signal will be very broad. To perform NMR imaging based on these broad lines is extremely difficult and a huge field gradient is required.

In the past year, a unique NMR imaging facility for the study of solids utilizing the huge gradient in the stray field was assembled. A planar surface under the edge of the superconducting coil in the 9.394 Tesla magnet was utilized for this purpose. At this surface, a static field gradient strength of approximately 5000 gauss per centimeter was measured. Since the field is static, the sample has to be moved during irradiation to get the back projections for all three dimensions. A maximum of 512 points will be irradiated by a "solid echo" multipulse train [2]. This linear movement can be repeated as many times (number of scans) as desired and echo signals can be accumulated to achieve a good signal-to-noise ratio. All these movements are controlled automatically by an ASPECT 3000 computer. Figure 1(a) shows the schematic of sample motion and the principal functions. Figure 1(b) shows a solid echo pulse sequence. The achievable resolution is 40 $\mu$m.

The binder distribution in the ceramic green body of silicon nitride/polyethylene glycol (binder)/ polyacrylic acid (dispersant)/water system is currently being studied. Sixty four pictures were taken along the X, Y, and Z directions for a cylindrical sample of 12 mm diameter and 10 mm thick. $^1$H echo signals at 162.7 MHz were backprojected to construct these 192 pictures. Figure 2 shows two examples from these pictures. This sample contains 10.4 wt. % binder. Figure 2(a) is a cross section internal slice which shows the binder is fairly well distributed. Figure 2(b) is a vertical slice; a low binder region was detected. The white spot on each of the two pictures is due to the epoxy, a high proton-containing polymer, used to glue the sample on the sample holder.

Since proton magnetic resonance outside of the magnet was observed, the resonance was measured at 162.7 MHz instead of 400.13 MHZ. A theoretical calculation showed that this resonance coil can also be used for $^{19}F$ resonance. Since $^{19}F$ resonance occurs at 4% lower in frequency than $^{1}H$, moving the probe 4mm closer to the center of the magnet is able to compensate the difference in frequency. This is important because $^{19}F$ is also an essential part of material.

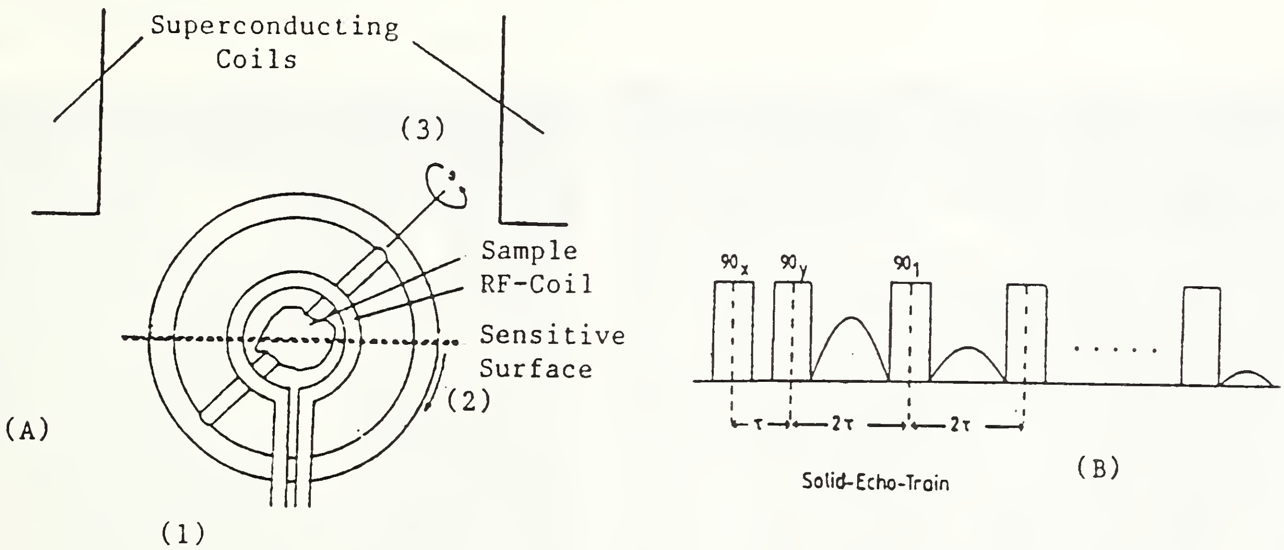


Fig. 1 (A) Schematics of probe movement: (1)linear motion along Z-axis, (2) sample rotation in YZ-plane,(3) second rotation of the sample to get a three- dimensional imaging; (B) multipulse train to detect solid echoes.

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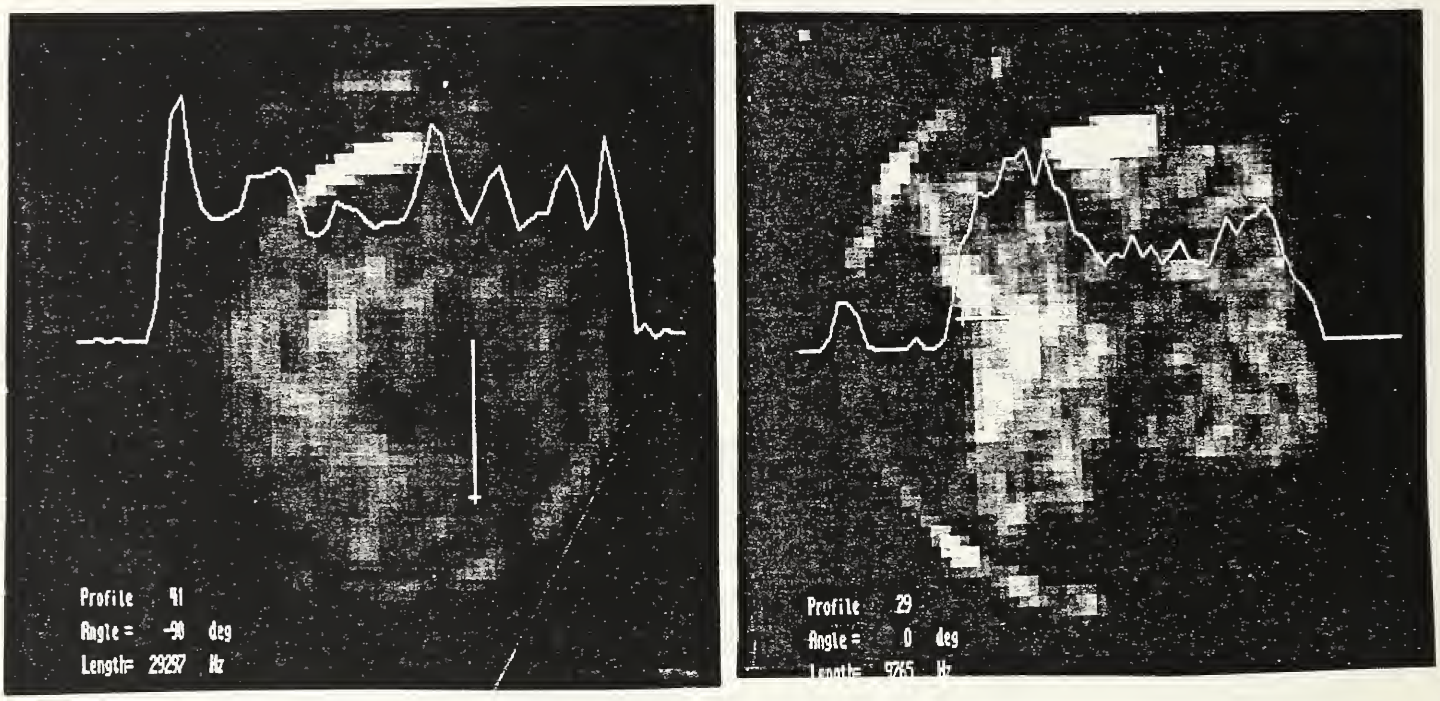


(A) (B)

Fig. 2 (A) Cross section and (B) vertical slice of a silicon nitride ceramic green body with 10.4 wt. % polyethylene glycol to show the binder distribution.

**Eddy Current Sensing of Carbon-Carbon Composites During Processing**

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This one-year project was undertaken to evaluate the feasibility of using an eddy current system for monitoring the pyrolysis of carbon-carbon composite sheets during high temperature processing. Carbon-carbon composite is prepared by heating a graphite-fiber, resin-impregnated structure until the resin matrix completely carbonizes. Electrical conductivity increases of the order of 100% are observed during this heating [1]. Thus conductivity measurements, particularly by the non-contact eddy current method, should prove useful for monitoring the processing. This project was cost-shared with the General Dynamics Corporation, Fort Worth, Texas, and DARPA.

Using a through-transmission configuration of coils similar to that used for aluminum sheet [2], but adapted for the higher frequencies needed for the more resistive carbon-carbon, a high temperature test system was designed and assembled. This system operates at fixed frequencies, offering, it is believed, greater precision and more direct interpretation than by the pulse method previously used [1]. Flat spiral coils of platinum wire were placed on opposite sides of the test materials. Through-transmission measurements were performed at a series of frequencies ranging from 5 kHz to 2 MHz. The resistivity was found by comparison of the frequency of peak power absorption with the theoretical prediction of Dodd and Deeds [3] appropriately modified for this geometry. Measurements at room temperature were performed on a SI sample of carbon-carbon of thickness 5 mm. To check the accuracy at room temperature, a four-probe van der Pauw DC measurement [4] was performed which obtained agreement within 1.4%. Because unprotected carbon-carbon oxidizes at elevated temperatures, this check could be performed only at room temperatures.

Stainless steel sheets offered the possibility of making concurrent high temperature eddy current and four-probe DC measurements on a substitute material with the same conductance (conductivity x thickness) as typical carbon-carbon samples. Agreement was excellent up to 500°C, at which point the stainless steel reacted with air, producing a magnetic oxide which corrupted the AC magnetic eddy current measurement.

Finally, a 15 $\times$ 15 $\times$ .25 cm sheet of fully pyrolyzed carbon-carbon, coated with silicon carbide to prevent oxidation, was put through four heating cycles to 350°, 575°, 850°, and, lastly, 800°C, with eddy current measurements carried out during heating and cooling. The results indicate that the precision of the conductivity measurements was approximately 2.5% over the entire temperature range. In this temperature range the conductivity increased approximately 100%. A conductivity model based on contributions of two mechanisms was proposed. On the assumption of temperature-independent conduction by the fibers oriented along the direction of the current and thermally activated conduction by the semiconducting matrix (as by activated carrier concentration and/or activated mobility), an excellent fit to the data was achieved. The matrix conductivity activation energy was found to be 0.13 eV. The fit is shown in Figure 1.

The conclusion reached is that sensing of the resistivity of carbon-carbon sheets by through-transmission eddy current methods at temperatures up to 850° C is feasible and practical. In addition, a simple mechanism for describing the temperature dependence of the electrical conductivity of carbon-carbon was proposed.

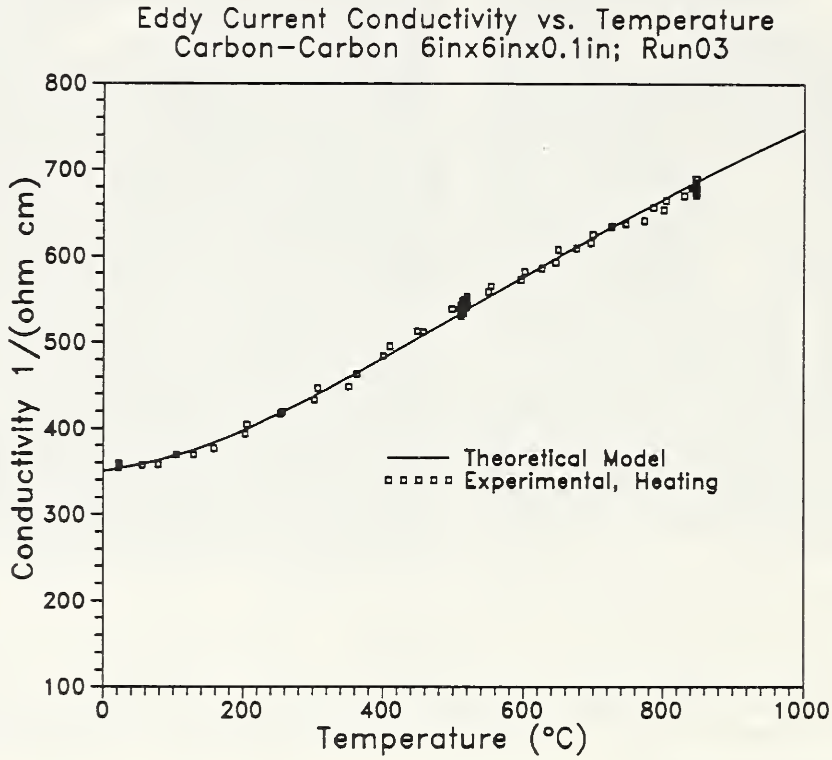


Fig. 1 Comparison between fitted model and eddy current conductivity of coated carbon-carbon plate.

## Publication

M. Gvishi, A. H. Kahn, and M. L. Mester, "Eddy Current Testing of Carbon-Carbon Composites," presented at the Review of Progress in Quantitative Nondestructive Evaluation, Brunswick, ME, July 28 - Aug. 2, 1991, to be published.

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## Development of Electromagnetic Probes for Intelligent Processing of Dielectric Materials

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Electromagnetic probes are useful for characterizing the dielectric properties of materials and can be used in the intelligent processing of materials. For example, to make a homogeneous ceramic specimen with the right dielectric properties, it is necessary to have properly compacted material in the green state. Improperly compacted material can lead to the wrong complex permittivity and also to problems such as residual stress and cracking in the sintered products. It is desirable to detect improperly processed material before the "value-added" step of sintering. Another example from the electronics packaging industry is the production of printed circuit boards. Here, it is desired to have homogeneous properties; for example, no delaminations in multi-layered printed circuit boards and, also, a consistent dielectric constant. A less obvious example is foundry operations. Here, "greensand" (a mixture of sand, clay, coal and salts) is wetted with water to produce molds for casting. Improper moisture content leads to poor quality molds and rejectable parts.

Two divisions within NIST, the Electromagnetic Fields Division and the Materials Reliability Division, are collaborating to explore the potential of electromagnetic probes for these and other practical examples. Work is proceeding to model the behavior of the probe and to perform laboratory experiments to characterize the probes and to demonstrate their practical utility.

The probes used in this research can operate with a clearance between probe and specimen (lift-off) to inspect material in a process control mode. Since these probes are sensitive to lift-off, the separation of the effects of lift-off and complex permittivity is currently being investigated theoretically.

### Theory of Capacitive Probe

Research was focused on capacitive (electrostatic) probes and microwave (open-circuit coaxial) probes. The former generates primarily an electric field, but also a very small magnetic field and, hence, it cannot work with highly conductive specimens. The coaxial probe can have an appreciable magnetic field. The capacitive probe is easier to analyze and the "shape" of the electric field is more readily controlled.

The capacitive probe consists of an array of electrodes with varying potentials impressed upon them. Changing the potential pattern changes the configuration of the electric field. Varying the potential allows, for example, the concentration of the electric field to search for inhomogeneities near the surface of ceramic specimens. The electric field can be calculated by first solving Laplace's equation for the potential in source-free regions, with the appropriate boundary conditions. The electric field is then calculated from the gradient of the potential. Calculations were performed for the case of an infinite plane over a dielectric half-space. A sinusoidal potential was impressed on the (conducting) plane. Solutions were obtained in closed form for $\Delta Y$ (change in probe admittance, Y, due to the presence of the dielectric). This allowed the calculation of the lift-off effect [1]. It was found that for (ad) > 1, the probe is mostly sensitive to lift-off and not to complex permittivity, for dielectric constants in the range $\epsilon \geq 10$. Here, a is the spatial frequency of the impressed potential and d is the lift-off distance. For the quantity ad of order unity, the probe is influenced by both $\epsilon$ and d.

A more realistic analysis was performed for the case of discrete electrode elements over a dielectric halfspace [2]. $\Delta Y$ was not exactly the same as for the sinusoidal potential case but displayed the same general trend. This analysis showed that a hybrid probe consisting of electrodes for which (ad) is much greater than one and electrodes for which (ad) is of order unity may be useful. The probe with large (ad) will be mostly sensitive to lift-off. This value can then be used in an inversion algorithm to obtain dielectric constants for measurements made with the probe for which (ad) is of order unity.

In collaboration with Professor B. A. Auld of Stanford University, a model was developed to determine the effect of inhomogeneities in dielectric properties on $\Delta Y$ [3]. This model could be used to estimate probe response to voids and cracks in dielectric substrates. It was found that

$$\Delta Y = j\omega\epsilon_0(\epsilon_i - \epsilon)(\nabla\phi) \cdot \mathbf{R} \cdot (\nabla\phi)V_f,$$

where $\omega$ = frequency, $\epsilon_o$ = permittivity of free space, $\epsilon_i$ is the permittivity of the inhomogeneity, $\epsilon$ is the complex permittivity of the homogeneous material, $\phi$ is the potential in the absence of the inhomogeneity, and $V_f$ is the volume of the inhomogeneity. The tensor, **R**, models the effect of the change in polarizability due to the presence of the inhomogeneity and is analogous to the demagnetization factor in magnetostatics [4].

Although it is not possible to evaluate the above equation in general, the form of the tensor, **R**, gives some physical insight. For example, in the case of cracks, **R** is a maximum when the electric field is perpendicular to the crack and a minimum when the electric field is parallel to the crack. Experiments described later show qualitative agreement with this theory.

## Theory for the Open-Circuit Probes

The coaxial probe consists of a coaxial line with an open-circuit termination [5,6]. A theory for this probe with lift-off was developed. The coaxial line has no low-frequency cutoff so, in principle, measurements can be made from near-DC to microwave frequencies. This probe generates both electric and magnetic field components and can be used to characterize both dielectric and conducting materials.

Since the fields are primarily reactive with little radiation, they are confined to a region near the probe end (approximately a distance equivalent to the outer conductor radius of the probe) and, therefore, lift-off produces a strong perturbation in the input admittance. Numerical solutions of a wave equation for the admittance of this probe as a function of air gap are in progress. These will be analyzed in the future to develop a method for separating the effects of lift-off from complex permittivity. In addition to the "field" model described above, the probe output was calculated using a lumped parameter equivalent circuit model to account for the probe fringing fields. The relations are valid at frequencies where the dimensions of the line are small compared with the wavelength and the open end of the line is not radiating. An uncertainty analysis was performed to estimate the accuracy in determining the real and imaginary components of the permittivity.

## Theory for Inhomogeneous Media

The above analysis assumes a material with a homogeneous dielectric constant. However, many materials are made of several constituents, each with its own dielectric constant. To determine the effective dielectric constant, "mixing rules" described in the literature were used. These mixing rules show a change in $\epsilon$ with varying degrees of compaction for greenware, since the volume fraction of the powder increases and the volume fraction of the voids decreases as the specimen is compacted. This change has been verified by experiment. Furthermore, the material can be dispersive. An expression was derived for the admittance of the capacitive probe, which indicates that for greensand the real part of probe admittance should depend linearly upon volume water fraction and also on frequency this was confirmed by measurements.

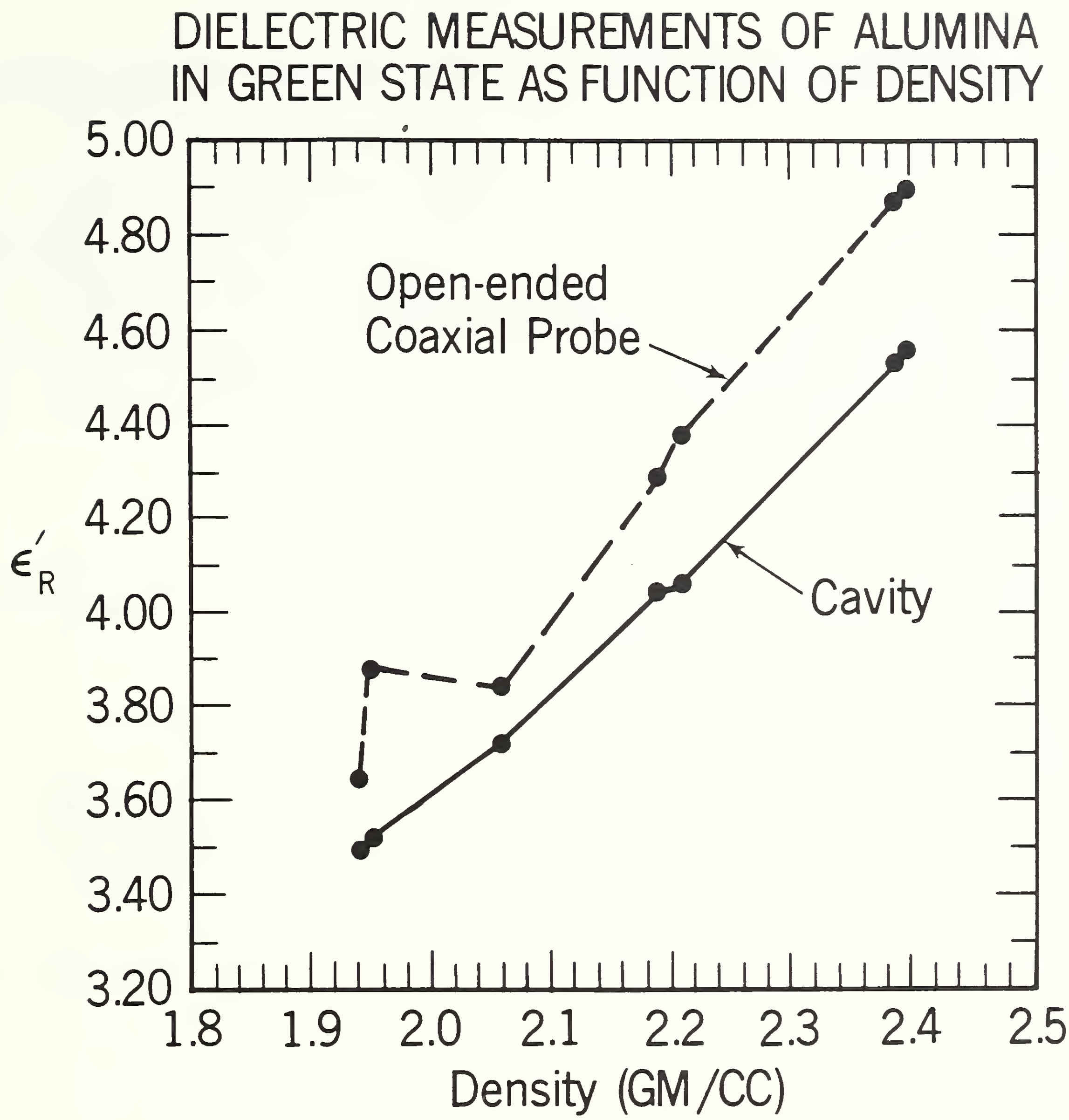


Fig. 1 Dielectric constant versus compaction of alumina in green state from measurements made with both X-band cavity, and with coaxial probe.

Experimental Results

The results of experiment agreed qualitatively with the theory given by Equation 1. For example, the maximum probe output was obtained when the electric field was perpendicular to the inhomogeneity (which was in the form of a buried slot). If the probe dimensions were to be reduced by a factor of ten, the signal should be sufficient to resolve a defect with a volume of approximately 1 $mm^3$. This assumes that the noise pickup with the present probe remains the same.

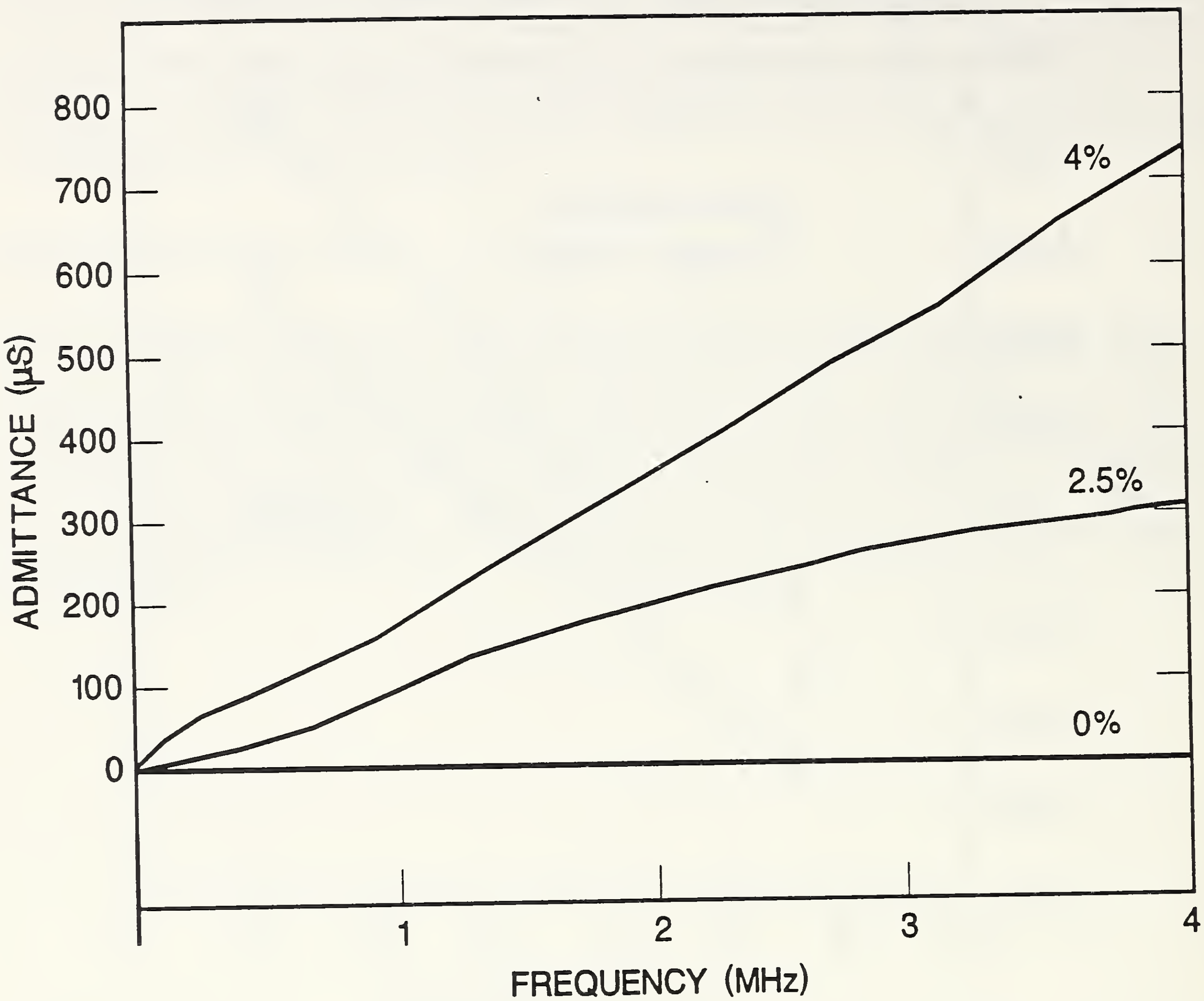


Fig. 2 Re(Y) (real part of capacitive probe admittance) versus frequency for several values of greensand moisture content.

In consultation with a major ceramics manufacturer, well-characterized compaction specimens were produced. Dies were designed and specimens with varying degrees of compaction were made. Measurements were performed both with microwave frequency probes and the capacitive probes. Both probe types showed a highly sensitive change in complex permittivity with degree of compaction. Typical data are shown in Figure 1 for both the open-ended coaxial probe and X-band cavity measurements. Although the latter is more accurate and is used as a reference, cavities cannot easily work in real-time for on-line process control. The open-ended coaxial probe, while at present not as accurate, can be operated on-line and shows essentially the same trend (lift-off effects) as the cavity results.

In another set of experiments, samples of greensand were obtained from a General Motors foundry. Test specimens were prepared with known moisture content. A mixing rule analysis indicated that at low frequencies (below the relaxation frequency of water), there should be a linear relation between the real part of the capacitive probe output and frequency. The slope of this line was predicted to depend on moisture content. This trend is clearly evident in the data shown in Figure 2. An open-circuit coaxial sample holder was used to make measurements on the real and imaginary parts of the dielectric constant of greensand at high frequencies. These measurements also show a strong correlation of moisture content as indicated in Figures 3 and 4. In the particular case of the greensand problem, there is a need to couple the probe output with an appropriate mixing rule to accurately predict moisture content. Its value would then be an input to an existing system which controls the amount of moisture added to greensand as it moves through the foundry. NIST and General Motors are currently exploring a collaborative effort in this area.

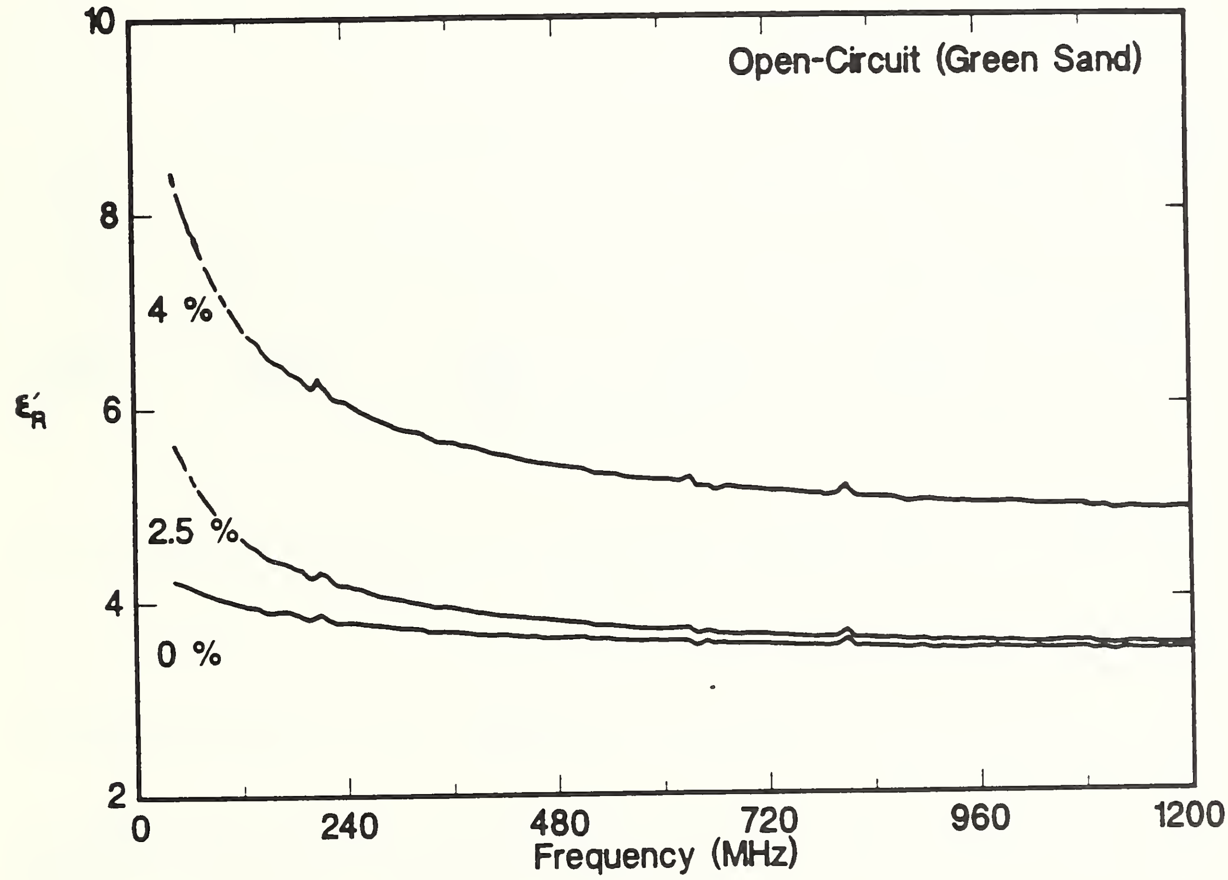


Fig. 3 $\epsilon_R'$ for versus frequency using an open-circuit sample holder in greensand samples prepared with 0, 2.5, and 4 percent moisture content.

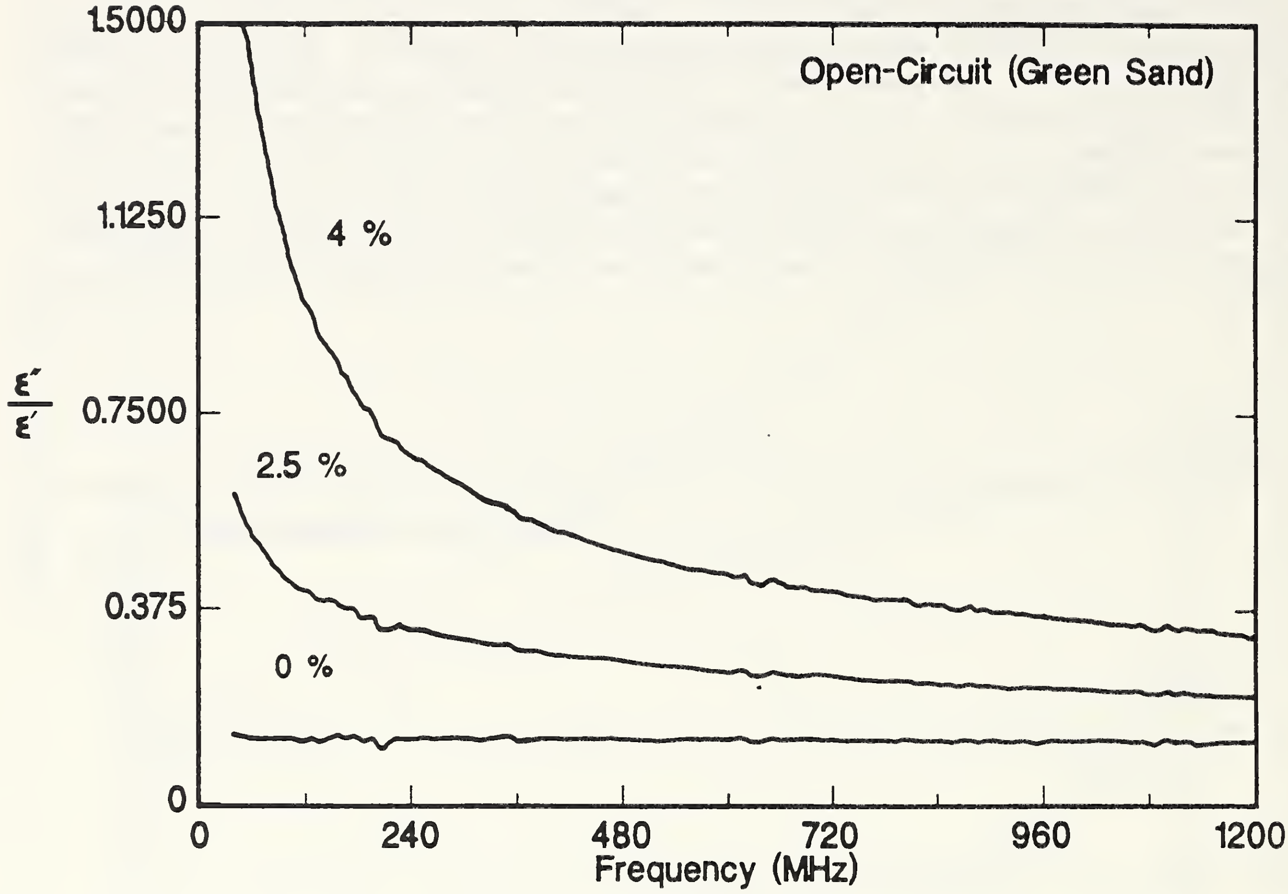


Fig. 4 Loss tangent, $\epsilon''/\epsilon'$, versus frequency using an open-circuit sample holder for greensand samples prepared with 0, 2.5, and 4 percent moisture content.

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## Transient Elastic Waves in Laminates

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The objective of this effort is development of techniques for the evaluation and process control of composite materials and other materials/structures where interfacial conditions are important. Derivation, development, refinement, and verification of the exact mechanical solution to transient wave propagation of a layer on a half space for various material properties and various interface (bonding) conditions is being accomplished. The most recent activities improved the computational speed and versatility of the coded solution, including the calculation of sample applications, and designing an experiment and interfacing instrumentation to verify the solution.

Pulsed ultrasonic testing techniques are increasingly having a greater role in the evaluation and quality control of composite materials. The understanding of the generation of ultrasound, its interaction with interfaces in the material, its detection and interpretation constitutes an important part of the development of the technology. This approach has been multi-faceted. The four domains of exact theory, numerical simulation, controlled laboratory experiment, and field applications are designed to have overlapping regions so that assumptions can be checked and solutions have immediate practical uses. The current project is to develop the ability to model exactly the generation, propagation, reflection and refraction, and detection of point source ultrasonic waves in layered media.

Previous research developed and made available to the public a Fortran code to compute the dynamic Green's function of an infinite plate [1]. The computation has many proven practical applications, among them calibration and design of transducers, design and verification of laser generation and detection of ultrasound, development of novel testing techniques, and acoustic emission source characterization. It has been utilized by federal labs, universities and industry. The code also serves as a check of the present program being developed.

The Fortran program to compute the transient Green's function of a plate layer on a half-space under various interface conditions was checked using two different integration paths and checked against the simple plate solution, and consistent results were obtained for all cases. The program is versatile and allows sources that are vector forces, couples, or dipoles with any orientation and with arbitrary time dependency. With this program, laser generated thermal-elastic and explosive sources and other distributed sources can be simulated by numerical integration.

During the past year, improvements in the selection of instants of time at which Green's-function values are iteratively computed reduced overall computational time to as little as 5% of the time previously required. The conventional scheme of using uniformly spaced instants was superseded by a coarse uniform mesh supplanted by brief high-density meshes coincident with the arrivals of the various wavefronts. Cubic spline interpolation was used to replace the data lost by adoption of the coarse uniform mesh. The routines were also expanded to allow each type of propagating wave (e.g., longitudinal or shear) to be associated with its own attenuation coefficient.

A new transient recorder with a 200 MHz sampling rate, long capture memory, and the capability to change sampling rates during the recording of a waveform was procured. Details of echo waveforms can be sampled at the maximum rate, while a slower rate is used when only low-frequency data are relevant. The transient recorder is being tested, and interface control programs are being written.

An example of possible applications is the calculation of the vertical displacement due to a pulsed laser thermal-elastic-expansion source (See Figure 1). Here, a plate layer of plastic on a semi-infinite glass substrate is considered under three different conditions of bonding. The thermal-elastic source and the displacement detector are both on the top surface of the specimen, separated by a distance of three times the thickness of the top layer. Shown in Figure 2 are the vertical displacements under interface conditions corresponding to the presence of a liquid, vacuum, or weld, respectively, between the plate and its substrate. From the graphs, it is easy to see that the reflected longitudinal wave ($2p_1$) and the reflected head wave ($s_1p_2^*p_1$) provide the most information about the interface conditions. Theoretical predictions will be compared with experimental results in future work.

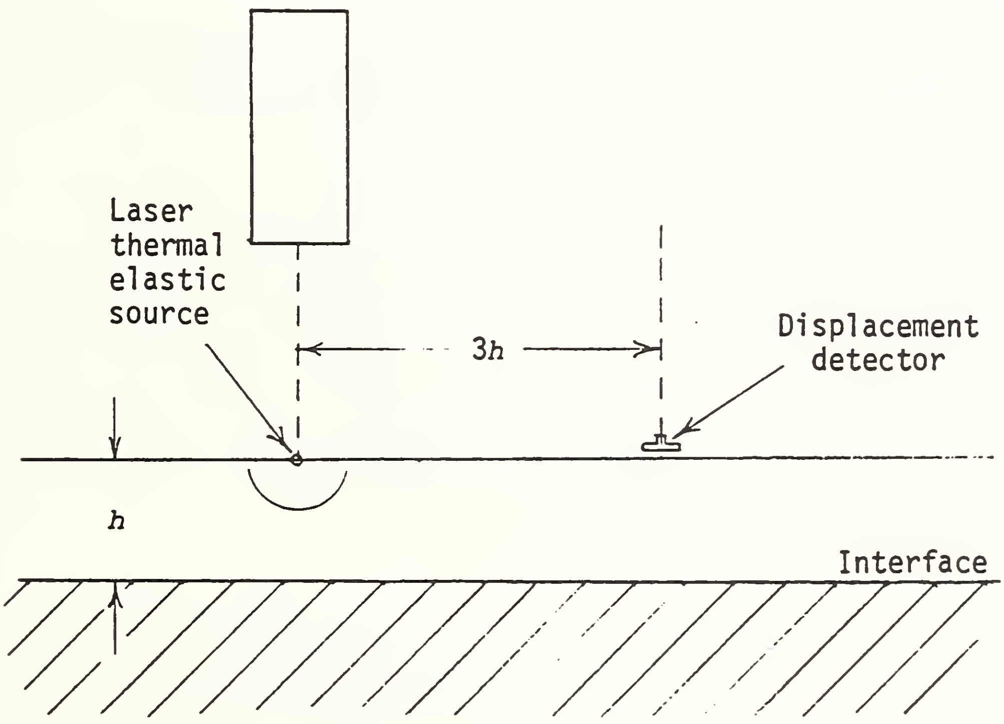


Fig. 1 Model configuration for a laser thermal-elastic source and a receiver located at a distance of 3 plate thicknesses. Layer is plastic; half space is glass.

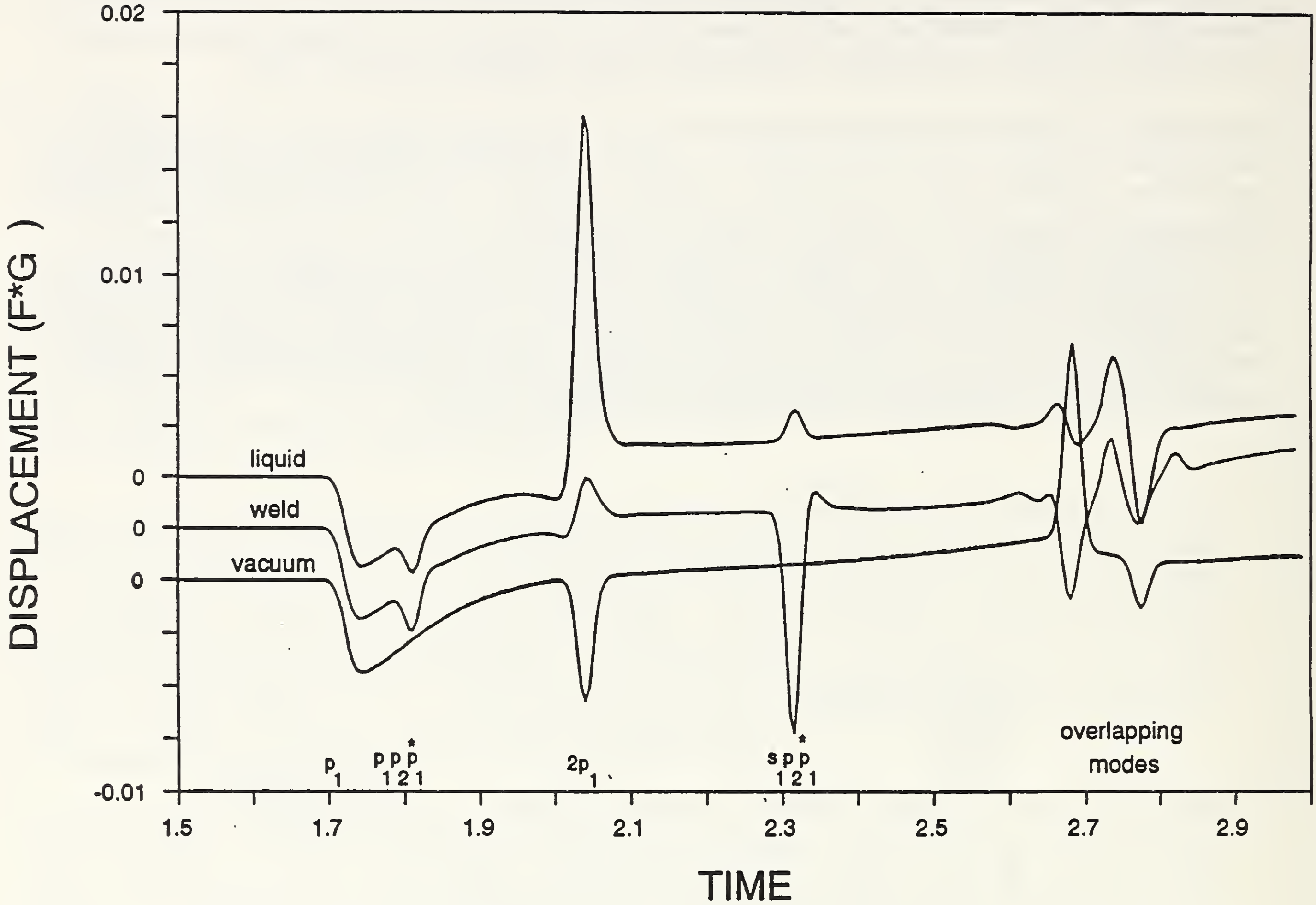


Fig. 2 Calculated vertical displacement waveform due to a pair of dipoles (laser source) for the configuration of Fig. 1: Liquid layer interface - transmits normal stress only. Vacuum interface - no interaction at interface. Welded interface - both stress and displacement are continuous.

We have begun discussions with several academic and industrial colleagues who have an immediate recognition of the power of this program for solving applications problems. They will soon be given the opportunity to exercise the algorithms and will provide feed back on any difficulties encountered and suggestions for making it more user friendly. Martin Marietta continues to express great interest and has provided encouragement. Office of Naval Research has provided modest financial support and great interest.

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## X-Ray Radioscopy Standards

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The objective of this investigation is to develop and distribute the standards and techniques required for the successful implementation of automated radioscopy inspection systems. X-ray radioscopy refers to x-ray inspection systems in which the conventional film imaging media has been replaced with an imaging screen, and also often have low-light-level cameras and video display monitors. Systems with this electronic viewing capability can be automated further with automatic inspection routines and motorized stages. Unfortunately, the standards and techniques necessary to confirm the proper operation of these systems are not yet available. This program supports efforts to develop the necessary standards and techniques so these efficient inspection technologies can replace older techniques with no loss of control.

Close collaboration with American Society for Materials and Testing (ASTM) Subcommittee E07.01 on Nondestructive Testing has been maintained. This committee contains representatives of the industrial developers and users of this technology, and is responsible for maintaining a wide variety of x-ray testing procedures and standards. Assistance in the preparation of new standards for radioscopy that parallels the older standards for film-based radiography has been provided.

The rest of the world is also quite interested in implementing this new x-ray inspection technology. International Institute of Welding (IIW) Commission V is developing a number of standards for this technique, which they will submit to the International Standards Organization (ISO) as draft standards. NIST is trying to serve as liaison between ASTM and IIW so U.S. standards will be compatible with the rest of the world.

At the January 1991 ASTM meeting and July IIW meeting, a twisted strip image quality indicator design for characterizing a microfocus x-ray tube beam was presented [1]. The concept is simply that of a thin strip with a half twist. The twist allows the long dimension of the strip cross section to be perfectly oriented with the x-ray beam at some location, without careful orientation of the strip itself. It was found that metal strip was readily available in thicknesses that provide a high-contrast image. Figure 1 shows the intensity gradient across an image of the strip. The 0.07-mm width demonstrates the good resolution possible with the technique. The concept is being evaluated in round robins within ASTM and IIW for measurement of image unsharpness and focal spot size.

Fiber-volume fraction and porosity level of polymer composites is of concern to those using these materials for high-performance structures. Since the constituents of a composite (fiber, matrix, and porosity) have differing x-ray attenuation coefficients and the energy dependence of the coefficients vary, it is possible to formulate an inversion algorithm for recovering the percentage of each constituent from attenuation measurements at three or more energies. An apparatus was assembled for making quantitative x-ray transmission measurements on composites. The system (Figure 2) utilizes an energy-sensitive NaI(T1) detector and a PC-based multichannel analyzer. Transmission spectra were collected for various samples of glass-epoxy composites and demonstrate the utility of the concept. The measurement method appears promising in cases where the x-ray attenuation characteristics of the fiber and matrix separately are known *a priori* or can be measured. The system stage will be automated next year for spatial mapping of the composite properties.

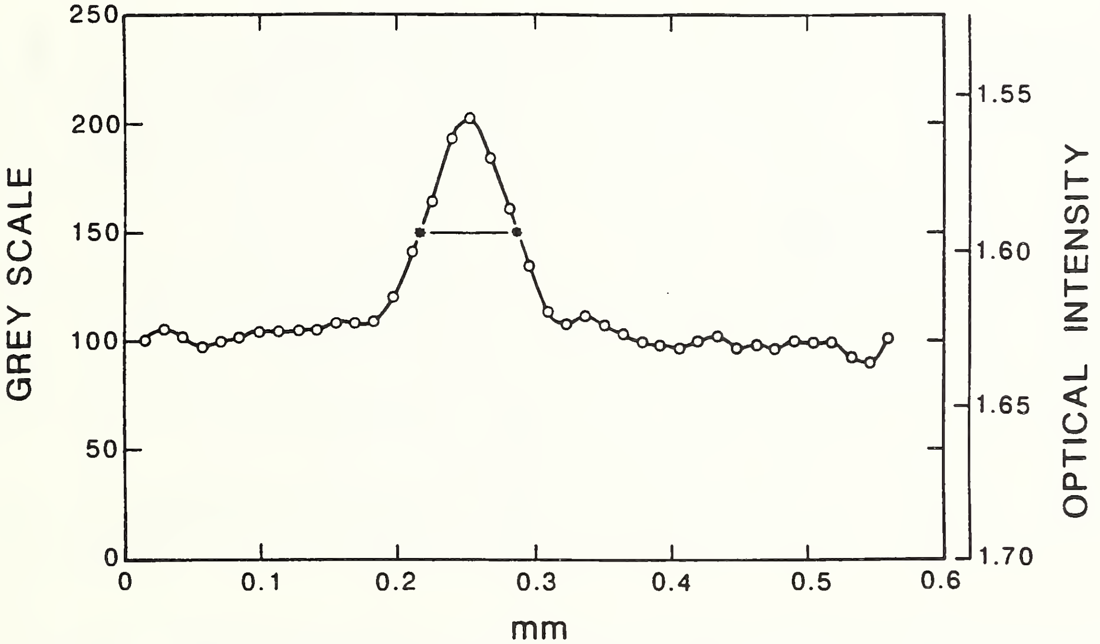


Fig. 1 Gray-scale intensity across the image of the twisted strip image quality indicator.

In an evaluation of new inspection technologies for advanced materials, we imaged a series of damaged composite panels with an x-ray laminography system. This system uses a scanned beam laminography design to emphasize various planes of focus within a three-dimensional structure. The damaged panels were treated* with a high atomic number material that strongly absorbed the x-ray beam. It was found that the laminography system was able to reveal branching of the crack paths as the plane of focus was scanned through the panels.

For the past two years, a summary of the IIW nondestructive evaluation activities were published in Materials Evaluation. In those reports, the various documents that are being producing are described and a request for U.S. contributions to the standards making task forces. When IIW began to develop standards for radioscopy systems this year, NIST circulated the documents from each of the committees so the U.S. standards would be as similar to those of IIW (and ISO) as possible.

NIST has been working with the General Electric Company, the Naval Air Engineering Center, and the U.S. Army Materials Technology Laboratory in developing a military standard for radioscopic inspection. The standard will be a supplement to a general ASTM standard (E1255) on radioscopic inspection. The fifth draft of this standard has been circulated for comment.

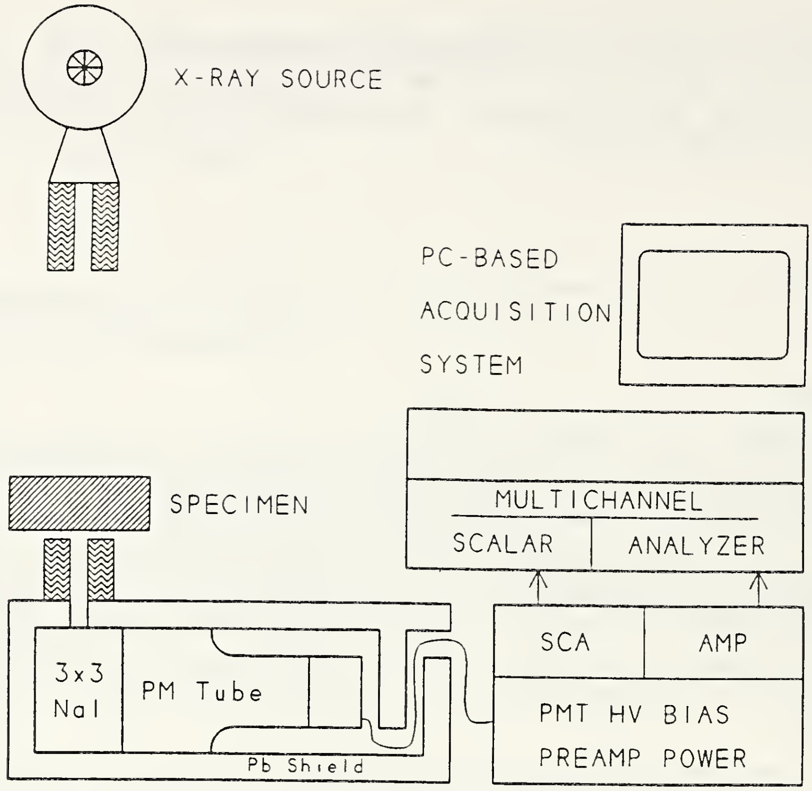


Fig. 2 Schematic of NIST prototype system for in-situ measurement of fiber-volume in composite materials.

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## Advancement of Documentary Standards for Nondestructive Evaluation

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This project is a continuing activity that responds to NIST's basic mission in standardization as it relates to nondestructive evaluation (NDE). The objective of the project is twofold: to promote the development of national standards that reflect the latest results of NIST research, and to pursue the adoption of international standards that are consistent with the best practices of American industry. These activities were integral parts of the NDE Program from its inception.

The approach to achieving these objectives is based upon the personal involvement and influence of the author in American and international standards-writing organizations. The author serves on the Executive Council of the ASTM committee on NDE, and chairs the section on infrared methods and the subcommittee on residual stress measurements. In ISO he heads the U.S. delegation to the committee on NDE and he chairs the U.S. Technical Advisory Group to this committee.

All of this work is performed in close collaboration with the private sector which is well represented in the ASTM committees, the U.S. delegation to ISO, and the Technical Advisory Group. Following are some of the principal accomplishments of this project in FY 1991.

An ASTM subcommittee letter ballot on "New Standard Test Method for Noise Equivalent Temperature Difference of Thermal Imaging Systems", which was developed in the NDE Program, was carried out. This is the third of three standards which, taken together, define the important performance characteristics of thermographic systems for NDE applications. Negative votes submitted by General Electric (GE) resulted in revisions to the document, which will be reballoted before the end of the fiscal year.

Another ASTM subcommittee letter ballot on "New Standard Recommended Practice for Construction of a Stepped Block and its Use to Estimate Errors Produced by Speed-of-Sound Measurement Systems for Solids", which was also developed in the NDE Program, was also carried out. This document is an essential precursor to a standard test method for ultrasonic measurements of residual stresses. The ballot was successful in that no negative votes were cast. However, several comments provided by experts merit consideration before the document is advanced to a committee ballot.

A three-day symposium on NDE standards was held at NIST under joint sponsorship of NIST and ASTM. Four of the papers were by NIST personnel; two of these papers were based on work carried out under this project. The proceedings of the symposium--a volume containing 23 peer-reviewed and carefully edited papers--is expected to be an important contribution to the literature.

The biennial meeting of the ISO technical committee on NDE, which was held in May 1991, produced numerous significant accomplishments for the U.S. Two NDE documents based upon U.S. standards are ready for registration as Draft International Standards and at least four others will soon advance to Committee Draft status.

As part of its concerted effort to promulgate internationally acceptable standards before the implementation of the European Community in 1992, the U.S. delegation had submitted proposals for five new work items since the previous meeting in 1989. The international ballots of these proposals all produced favorable votes, but the proposals failed nevertheless, because ISO rules require at least five countries to indicate a desire to work on a project. Reballot of the proposals is expected to be successful.

## Eddy Current Standards and Methods

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This program focused on factors affecting the performance of probes used in eddy current nondestructive evaluation inspections and the development of techniques and standards for measuring that performance [1].

Eddy current inspections are widely used in both civilian and military applications for finding failure-producing defects in metal parts before they reach critical size. The eddy current technique is a very sensitive method but is affected by many variables. Often these effects are known but not quantified, making the design of equipment or procedures more of an art than a science. Empirical and computer-based models of eddy current probe performance were developed and tested so that probe design and selection can be optimized in a predictable fashion.

In FY 1991, efforts were focused on producing an empirical model for predicting probe sensitivity. This model was developed by winding coils with very specific construction parameters and using a statistical analysis to determine the functional dependance of probe sensitivity on these parameters [2,3]. Experimental work has relied on NIST expertise in probe construction to produce a new type of eddy current probe and develop a new application for an existing probe design.

### Recording Head Probe

The new eddy current probe which was produced was made using a ferrite head from computer hard drive. Other novel probe designs were made using magnetic recording heads from floppy disk drives [4,5]. The field bubble produced by a hard drive head is considerably smaller than that of a floppy drive head and thus produces better spatial resolution than either the floppy drive probe or a conventional probe. Measurements of the recording head probe showed that this new probe can detect very small flaws in low conductivity alloys that are missed by conventional eddy current probes. Figure 1 shows a plot of the impedance response of the recording head probe on an electrical-discharge machined (EDM) notch in Inconel 718 (In 718) measuring 0.11 mm long by 0.04 mm wide. The recording head probe sensitivity is also directional. This feature, when used in conjunction with the fine spatial resolution can be used to map the extent and orientation of a flaw.

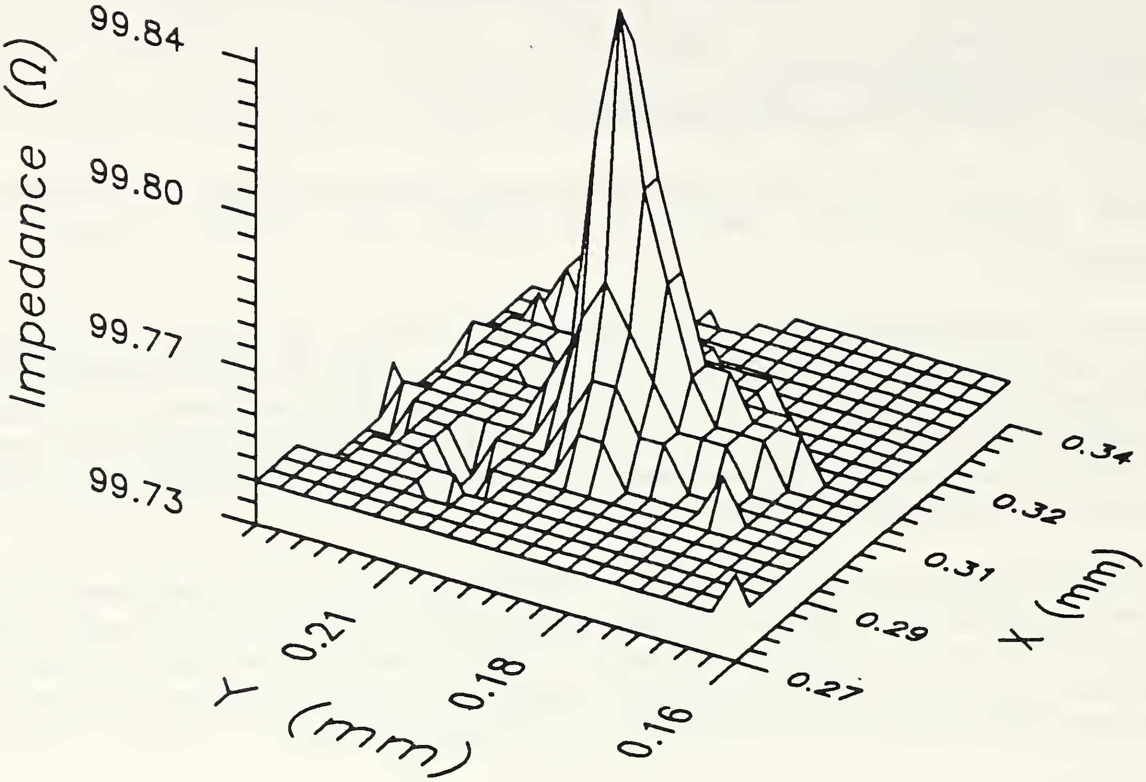


Fig. 1 Recording head impedance response on an EDM notch in In 718.

Fatigue Crack Growth Monitor

A technique using a differential eddy current sensor that can be used to monitor the formation and growth of fatigue cracks was developed. In this experiment, the sensor on flat plate specimens was used in a 4-point bend apparatus, although there is no reason why the technique could not be extended to more complex part geometries. These specimens were made from 7075-T6 aluminum and a stress riser was formed in the top surface of the plates by drilling a 0.34 mm diameter hole approximately 0.2 mm deep. The sensor was secured to the specimen with an electrical insulating varnish. The varnish is easily softened using acetone which makes sensor removal a benign process and the sensors reusable.

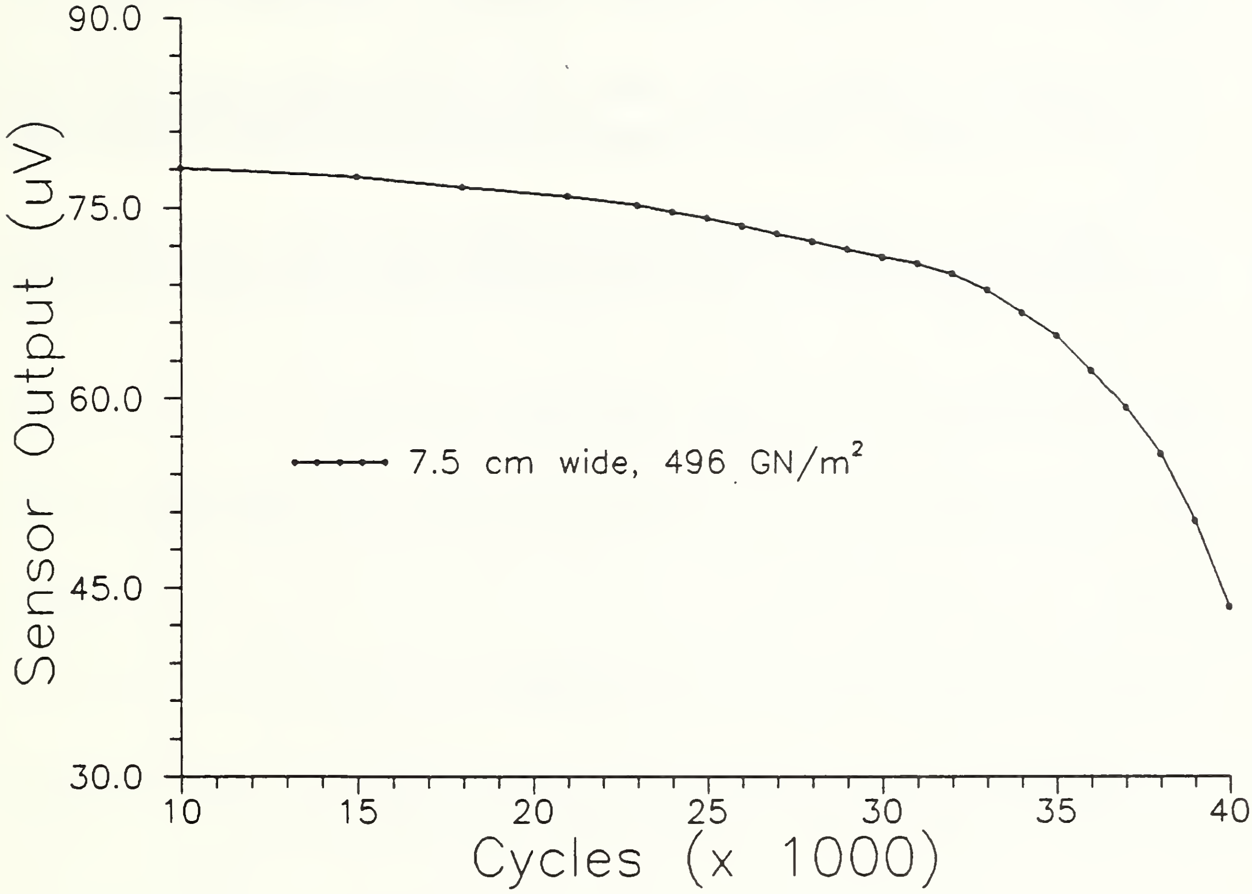


Fig. 2 Sensor output vs load cycles for 7075-T6 aluminum four-point bend specimen containing a stress concentration.

A 0.25 mV signal at 100 kHz was used to drive the outer coil and the output of the pickup coils was measured with a lock-in amplifier. The sensors were positioned on the aluminum specimens so that the stress riser was located under one of the pickup coils. Also, the sensors were oriented in such a way that a line joining the centers of the pickup coils would be perpendicular to the direction of the crack growth. This orientation results in a linear sensor output over a wide range of flaw sizes [6]. Figure 2 shows a plot of sensor output versus number of load cycles. The output decreases because the stress riser was positioned under the negative polarity coil and the sensor had a positive unbalance when mounted.

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2. T. E. Capobianco, "Real-Time Eddy Current Monitoring of Fatigue Crack Growth," The Review of Progress in Quantitative NDE, Brunswick, ME, 1991, to be published.

## Ultrasonics and Acoustic Emission Standards and Methods

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Automated Production Technology Division

The objectives of this project are to develop new or improved methods for acoustic emission (AE) and ultrasonic inspection and process monitoring techniques, develop and disseminate artifact and documentary standards, and develop and maintain measurement services.

Virtually all of the considerable NIST document standards work on AE and ultrasonics has been partially supported by this project. The project has also resulted in virtually all of the NIST output in alternative standards delivery mechanisms (measurement services, calibrations, and SRM's). The output of this project has resulted in part or all of the methodology for nine ASTM standards one DOT practice, one ISO CD, three MIL STDs, and two ASME standards. The methodologies, for example the Hsu acoustic emission simulation source and the NBS conical transducer, are used worldwide. The value of the technology has been recognized by awards from national (ASTM) and foreign organizations (Japanese Society for NDI). Of equal importance, this project relates directly to all of the other Office of Intelligent Processing of Materials (OIPM) thrust areas by providing the tools and expertise to address these areas.

The documentary standards work this year was smaller but productive. The ultrasonic velocity standard made rapid progress on its way through ASTM committee E28. The ISO subcommittee on Acoustic Methods has four documents ready to be advanced to Committee Draft status, all resulting from NIST research or technology. A report detailing the technology for secondary calibration of AE transducers based on the NIST Conical Transducer and the NIST primary calibration was submitted to E7.04 subcommittee on AE. A group member was selected as the General Chairman of the IEEE Ultrasonics Symposium for 1995.

### Primary Calibration of Acoustic Emission Transducers

The uncertainty of the frequency response data produced by the primary calibration system has several causes: electronic noise in the front end amplifiers (especially in the standard transducer channel), the quantitization process of the transient recorder, and aliasing of frequencies above the Nyquist frequency (10 MHz). Noise from the front-end amplifier of the standard channel was reduced by the installation of a different amplifier, and aliasing noise was reduced by insertion of low pass filters in both channels. These improvements in the past year reduce the variation of calibration results on non-reseated transducers from about 1 dB to about 0.5 dB within the frequency range of the calibration.

Unlike the voltage amplifier previously used, the new low noise front-end amplifier is a charge amplifier. Therefore, the standard capacitive transducer acts as a charge source working into a nominally zero-impedance load, instead of a voltage source working into a nominally infinite-impedance load. In the new system, stray capacitance of the transducer does not affect its sensitivity. The calibration calculations were recast in terms of the charge sensitivity of the transducer. Calibrations of several transducers using both the old and the new systems were compared. The results indicate negligible bias of calibration data resulting from the changeover.

A gain-monitoring system was incorporated into the calibration facility. To characterize the system, a fast voltage step (the height of which can be easily determined) was applied to the inputs of both calibration channels. Captured data are then processed to determine the gains of both channels. By determining system gains before each calibration run, drift is compensated and overall accuracy is improved.

Secondary Calibration of Acoustic Emission Transducers

The prototype secondary calibration system consists of a steel block 40 cm by 40 cm by 20 cm with source, reference transducer and transducer under test mounted on one of the square block faces. It was previously demonstrated that using a capillary-break source and capturing waveforms from both reference and test transducers, transfer calibrations can be carried out with an uncertainty of about 3 dB [1].

A method using a conical transducer source driven by a gated sine wave was also been tested. Frequency spectra of the reference and test transducer outputs were compared frequency by frequency. The results compared well with those of the primary calibration. The gated sine wave method produces calibration data that appear less noisy than those of the transient capture method. Presently constructed circuitry does not allow for phase measurement, but modified electronics that would provide the phase information is being designed.

A report was submitted to ASTM E7.04 Subcommittee on Acoustic Emission describing progress on the development of a recommended procedure for secondary calibration of transducers.

Experimental Determination of Source Force Waveforms

Experiments with elastic impacts of steel balls on a glass plate (described in the Summary Report for FY 1990) were continued. Careful analysis of photographically determined coefficients of restitution of the impacts showed that measurements of source force waveforms with less than 5% uncertainty can be accomplished [2].

Work on experimentally determining the force-time waveform of various other sources also continued. The uncertainty in some cases could be estimated to be as small as 2%. The experimentally determined waveforms for a capacitive source and for a small quantity of high explosive (nitrogen iodide detonated by heat) are shown in Figure 1. The waveforms both have very good frequency content and are very close to an ideal step function and impulse, respectively [2].

Conical Transducer Optimization

The theory developed by Greenspan [3] explaining the action of the NIST conical transducer was used in computer modeling experiments. Various piezo-element shapes and sizes were tried with various backing materials, and some very rough guiding principles emerged. It is desirable to make the piezo-element as small as is practicable. It is also desirable to use a backing material with a specific mechanical impedance close to either that of the piezo-element or that of the material on which the transducer is to be used.

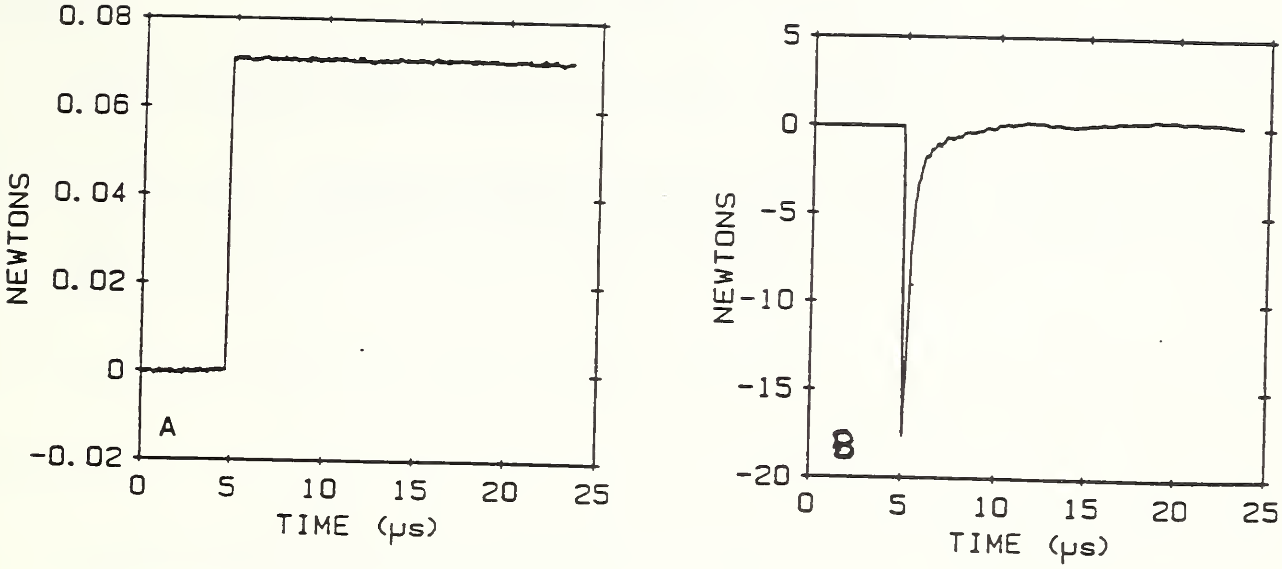


Fig. 1 Deconvolved force-time waveform: A. From a standard capacitive source transducer driven with a 49.7 V step. B. From a small charge of high explosive detonated by heat.

A particularly fortunate combination for use on steel was a modeled transducer utilizing a 1 mm diameter by 1 mm long PZT element with a steel backing. The computed response of this transducer is shown in Figure 2. As the curve shows, the variation with frequency is smooth enough to be readily corrected by electrical equalization. Plans call for construction of a transducer of this design for testing. Further work needs to be done to design optimal transducers for plastics and composites.

Retractable Source

A principal objective in designing this source was to combine the rapid unloading characteristics of the breaking capillary and the repetitive triggering capability of conventional piezoelectric transducers. In this design, a conical horn is driven through an acoustic lens by a piezo-ceramic element. With the appropriate electrical drive waveform, the horn tip retracts from the surface of a test block and rapidly unloads the static force originally imparted by contact of the horn tip and test block.

Tests of a prototype revealed that unloading proceeded far less rapidly than expected. The force waveforms exhibit details suggesting the influence of the asperities unavoidably present on the surfaces of both the test block and the horn tip. Further work based on this design was abandoned.

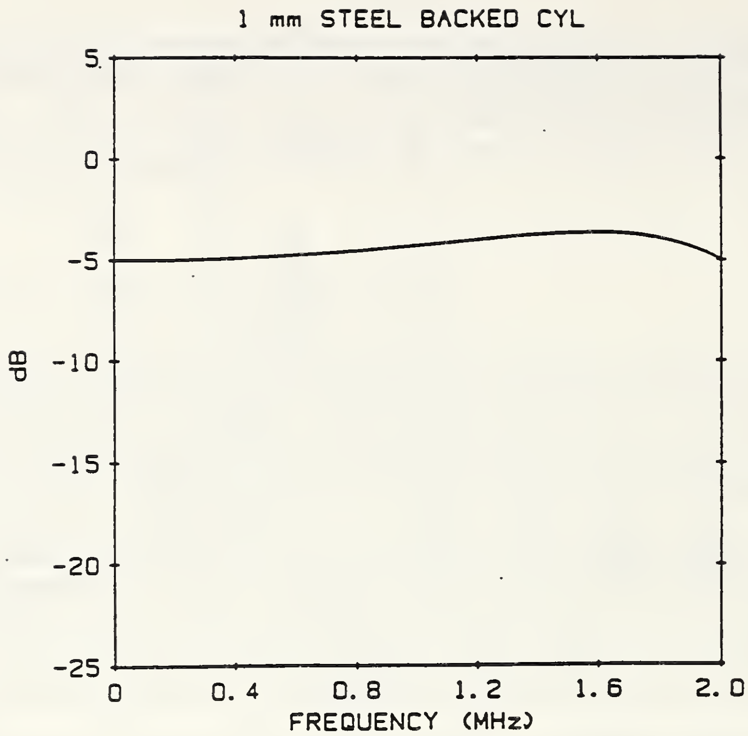


Fig. 2 Calculated response of a transducer having PZT element 1 mm in diameter and 1 mm long with a steel backing.

Intercomparison of Transducer Calibrations

A three way cooperative arrangement has been made to intercompare the calibrations of acoustic emission transducers. Dr. Satoshi Nagai of the National Laboratory of Metrology in Tsukuba, Japan, has developed a calibration method employing a laser source and a laser interferometer as standard receiver. Dr. Hajime Hatano of the Science University of Tokyo uses a reciprocity technique to calibrate acoustic emission transducers. NIST has agreed to intercompare results of these types of calibrations on transducers of several types including some of Japanese manufacture and on an NBS conical transducer. The work is to be carried out on both steel and aluminum blocks. Aluminum blocks of two sizes have been obtained, machined and polished for some of the NIST calibrations.

This project has also received some support from ONR, the Army MTL, and the FDA. Detailed consultation with many academic and industrial technologists has resulted in wide dissemination and, in some cases, commercialization of the technology. For example, the NIST conical transducer is now available from at least three independent sources. The documentary standards work is an organic collaboration with primary metals producers, the aerospace community, transducer houses, and large research and applications laboratories.

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A. Wolfenden, M. R. Harmouche, G. V. Blessing, Y. T. Chen, P. Terranova, V. K. Kinra, V. Dayal, and R. Phillips, "Dynamic Shear Modulus Measurements in Two Nickel-Based Alloys (to be published).

## Nondestructive Evaluation Using Magnetic Particles

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Materials Science and Engineering Laboratory

The objective of this investigation is to provide standards and methods for magnetic measurements used in the nondestructive evaluation (NDE) of steel parts. Current emphasis has been on flaw detection using magnetic particle methods.

Magnetic methods for flaw detection and property characterization of ferritic steels are widely used both for production and for maintenance. Methods for characterization rely on relationships between magnetic properties such as coercive force and mechanical properties such as yield strength. Methods for flaw detection, including the magnetic particle method, detect magnetic leakage fields that occur at defects in a magnetized material. Reliable use of these methods requires standardization based on a sound knowledge of the relationship of magnetic properties with observed leakage fields and mechanical properties.

The mechanisms and science underlying the use of magnetic methods in nondestructive evaluation and materials processing is under study. Working primarily with voluntary standards organizations such as ASTM, the results are applied to the development of improved standards and methods for the application of magnetic NDE in industry. Recent work has focused on test rings used for qualification of magnetic particles and the rating of artificial defects in the form of shims for use in process control of magnetic particle inspection.

Cooperative work on standards with ASTM committee E07 and committee K of the SAE has continued during the current year. A revision of a draft standard for a magnetic particle test ring was prepared. Cooperative measurements on rings of this design were made by NIST, Magnaflux Corp., and McDonnell Douglas. Based on their results, a redesigned ring was produced and tested at NIST and submitted to these industrial organizations for further testing. The results of these tests will be a new magnetic particle test ring. A number of these rings will be produced at NIST as a Standard Reference Material. As a result, the qualification of magnetic particles used in magnetic particle testing will be more reliable and reproducible. Equipment and software for measuring and analyzing the magnetic leakage field from defects in these ring were developed and tested.

A leakage field pattern from a test ring produced at NIST is shown in Figure 1. Rather than displaying the measured leakage field directly, the derivative of this field is plotted. This is desirable because it is the gradient of the leakage field that determines its ability to attract magnetic particles and form indications that reveal the defect. In addition to the leakage field gradient there are two other important factors that determine deductibility by magnetic particle inspection. That these are of considerable importance has not been previously recognized. These factors are the coercive force and the field gradient noise.

The inset in Figure 1 shows some of the leakage field gradient data from the ring on an expanded scale. The noise in this data is evident. This does not represent measurement noise because it is reproducible to a high degree. The noise arises from variations in the microstructure of the material on the scale of the measurement (which is approximately 0.1 mm). Experience with several different steels indicated that the magnitude of the noise is smaller for material with finer grain size. It is the magnitude of this noise that determines whether or not an interfering background of particles will stick to the surface of a material being tested and mask indications from the defects being sought. Experience with this noise indicates that it might be possible to use it as a measure of grain size uniformity or other properties of interest in steel.

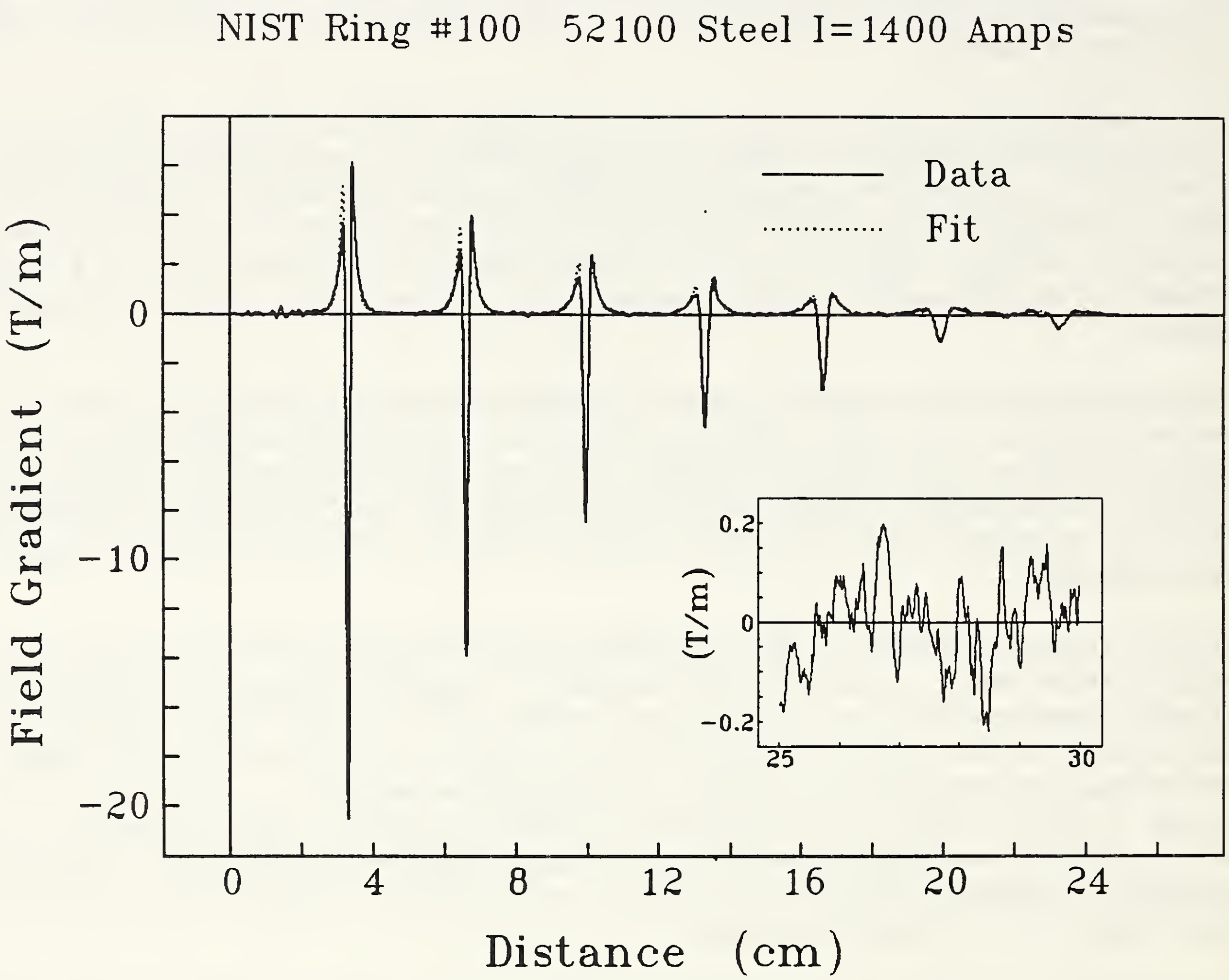



Fig. 1 Magnetic leakage field gradient from a magnetic particle test ring where the solid line represents the measured values and the dotted line represents a least squares fit to the data

The major reason that inconsistency in the use of magnetic particle test rings has plagued the industry for many years can be seen in Figure 2. This figure shows the peak value of gradient of the leakage field vs. current used for two rings fabricated form AISI 0101 tool steel (commonly called Ketos rings). These two rings were from the same manufacturer, had contiguous serial numbers, and were reportedly fabricated from the same lot of material. It can be seen, for example, that at 1500 amperes the peak field gradient for ring 90239 can be almost twice as large as that for ring 90238. Further, the residual field gradient (gradient at 0 current) is considerably larger for ring 90239. Hence, particles which would form a strong indication on ring 90239 might not form any indication on ring 90238.

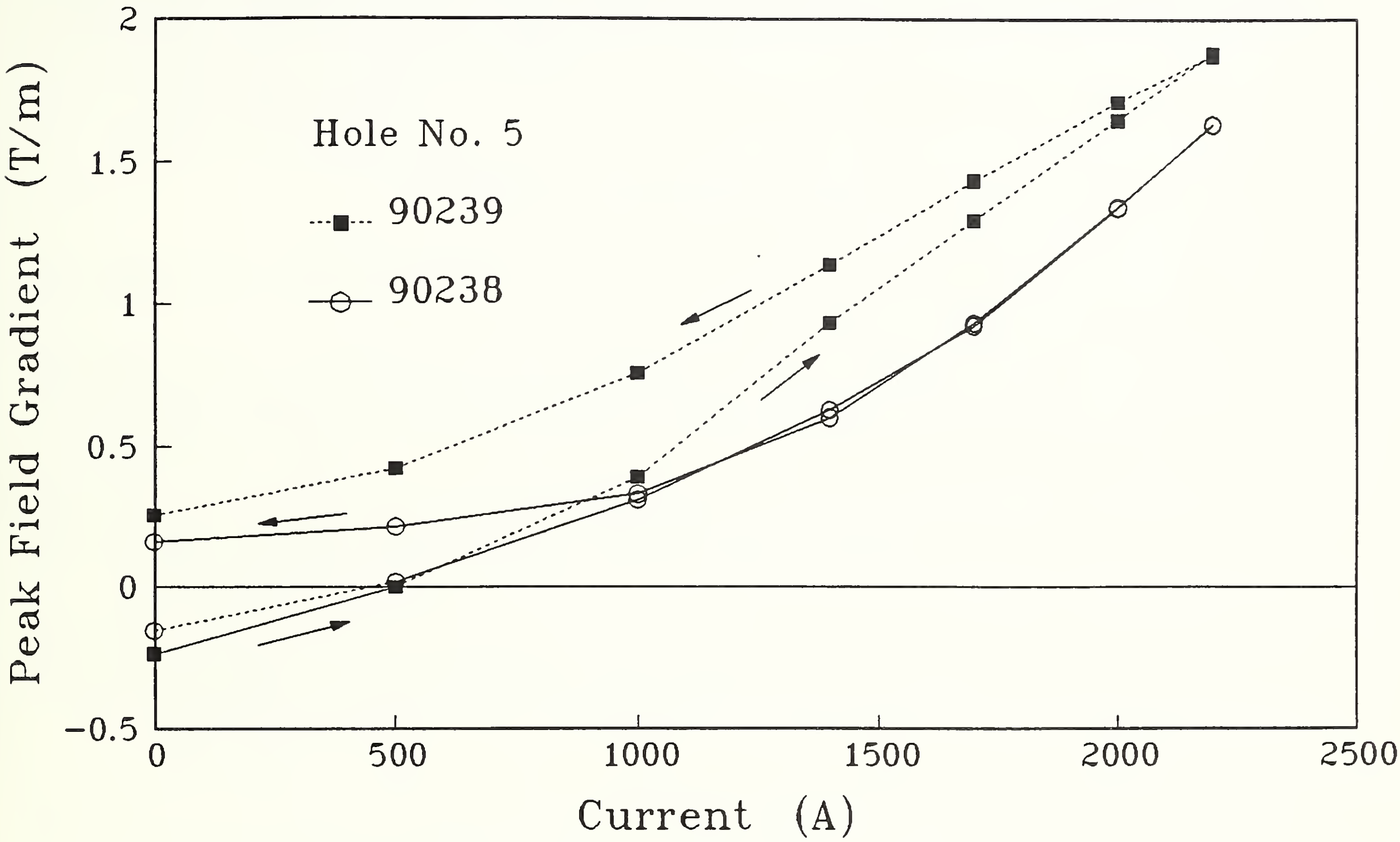



Fig. 2 Peak field gradient vs. central conductor current for two rings fabricated from Ketos steel (AISI 0101 tool steel)

The use of shims to qualify magnetic particle testing procedures and methods has recently been increasing. Shims are small pieces of a thin foil containing an artificial defect. The shims are placed on the surface of a part to be tested and the test and equipment being used are verified when a magnetic particle indication forms due to the presence of the artificial defect in the shim. There has been considerable controversy about the efficacy and repeatability of shims and a growing need for standards and specifications governing their production and use. The preparation of a standard based on performance rather than on design has presented a considerable challenge. A possible technique for the performance certification of such shims was developed. This procedure involves the use of the magnetic particle test ring described above. Working in conjunction with ASTM committee E 07, several drafts of a proposed standard were prepared and a number of tests were conducted to set the parameters to be used in the standard. A round robin test based on the standard should be underway within a year. The incorporation of shim use into existing standards governing magnetic particle NDE could result in a considerable increase in the reliability and efficiency with which magnetic particle tests are conducted.

The work described here was in collaboration primarily with Jim Chase of Magnaflux Corp., Chicago, IL., John A. Patsey of United States Steel, Pittsburgh, PA, Paul L. Ristuccia, Boeing Commercial Airplane Group, Seattle, WA, D. J. Hagemaier, McDonnell Douglas Aircraft Company, Long Beach, CA, J. S. Borucki of Ardrox Corp., La Miranda, CA, Richard Gaydos of Richard Gaydos & Associates, Westlake, OH, and John Fenton of LTV Aerospace, Dallas, TX.

# PERSONNEL

The permanent staff of the Office of Intelligent Processing of Materials is listed below.

## OIPM PERMANENT STAFF IN FISCAL YEAR 1991

*H. Thomas Yolken*, Chief
*John Gudas*, Deputy Chief
*George Birnbaum*, Senior Scientist
*Patty Salpino*, Administrative Officer
*Ellen Altman*, Secretary
*Linda Souders*, Secretary

## OUTPUTS AND INTERACTIONS

A. SEMINARS AT NIST

Christopher Scruby, AEA Industrial Technology, Harwell Lab., Oxfordshire, UK
"Laser Ultrasonic and Magnetic Sensors for Monitoring Materials"
October 4, 1990

Nelson N. Hsu, Automated Production Technology Division, NIST, Gaithersburg
"R&D Activities in Ultrasonics and Acoustic Emission in Japan"
February 26, 1991

Dr. Dale E. Chimenti, Johns Hopkins University, Baltimore
"Ultrasonic Guided Waves in Fluid-Loaded Plates with Applications to NDE of Composites"
July 11, 1991

Professor Julian Szekely, Massachusetts Institute of Technology, Cambridge
"The Role of Mathematical Modelling in the Intelligent Processing of Materials"
February 15, 1991

JOINT IPM/METALLURGY SEMINAR

Professor B. R. Tittmann, Pennsylvania State University, University Park, PA
"Advanced Sensors for Process Monitoring and Control of Polymer Composites"
November 20, 1990

B. INVITED TALKS BY OIPM STAFF

"Molecular Interactions and the Shape of Pressure Broadened Bands in the Far Wings", Goerge Birnbaum, University of Florence, Dept. of Physics, Firenze, Italy, June 3, 1991.

"Recent Developments in NDE Standards at NIST", Symposium NDT Standards II:, sponsored by ASTM Committee E-7 on NDT and NIST, April 9, 1991.

"Physics of Pressure Broadening in Infrared Spectroscopy", George Birnbaum, Institüt Für Experimental Physik, Vienna, Austria, June 13, 1991.

"The Modeling of Molecular Line Wing Absorption", George Birnbaum, Air Force Geophysical Directorate, Hanscom Air Force Base, MA, September 19, 1991.

"Intelligent Processing of Materials", H. Thomas Yolken, Department of Energy, Kansas City, Kansas, May 14, 1991.

"Electronic Solder Research at NIST", H. Thomas Yolken, COSMOS Club, September 20, 1991.

## C. AWARDS AND APPOINTMENTS

John P. Gudas, Office of Intelligent Processing of Materials, received the Federal Laboratory Consortium 1991 Award for Excellence in Technology Transfer.

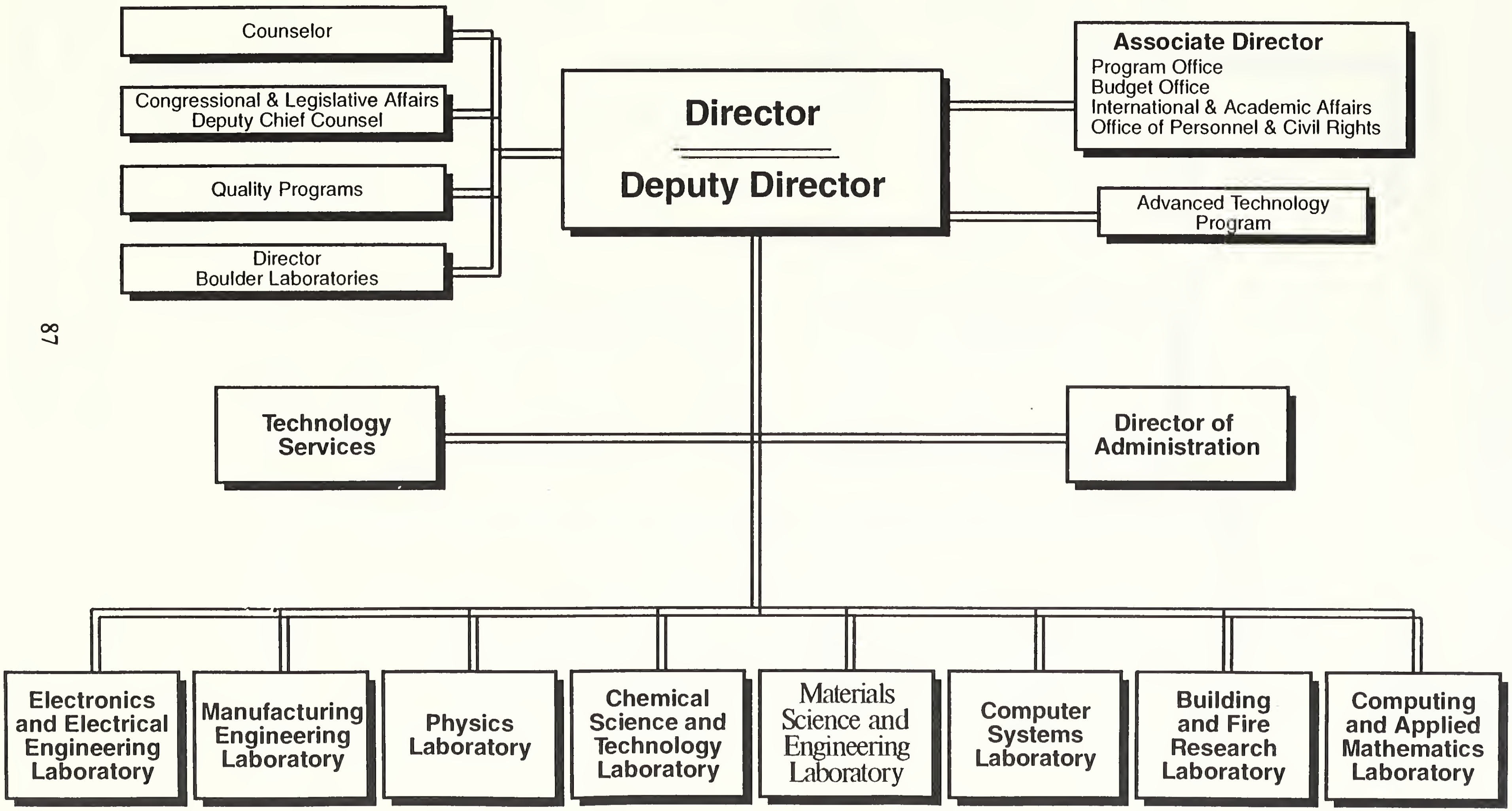
H. Thomas Yolken, Office of Intellligent Processing of Materials, was elected to the Board of Directors for the American Society for Nondestructive Testing.

H. Thomas Yolken, Office of Intelligent Processing of Materials, is Editor-in-Chief of the Research in Nondestructive Evaluation, A Journal of the American Society for Nondestructive Testing.

H. Thomas Yolken, Office of Intelligent Processing of Materials, was elected Vice Chairman of the Research Council for the American Society for Nondestructive Testing

H. Thomas Yolken, Office of Intelligent Processing of Materials, was appointed to chairman, working group on Intelligent Processing of Materials, Committee on Materials, White House, Office of Science and Technology Policy.

# National Institute of Standards and Technology



## MATERIALS SCIENCE AND ENGINEERING LABORATORY

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H.L. Rook, Deputy Director

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J.P. Gudas, Deputy

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**Polymers**

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B.M. Fanconi, Deputy

**Ceramics**

S.W. Freiman, Acting Chief
S.J. Dapkunas, Deputy

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H.I. McHenry, Chief
C.M. Fortunko, Deputy

**Reactor Radiation**

J.M. Rowe, Chief
T.M. Raby, Deputy

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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 Intelligent Processing of Materials Program at NIST for fiscal year 1991 is presented in this annual report.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

intelligent processing of materials; materials science and engineering; nondestructive evaluation; nondestructive evaluation standards; on-line sensing; sensors for process control

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