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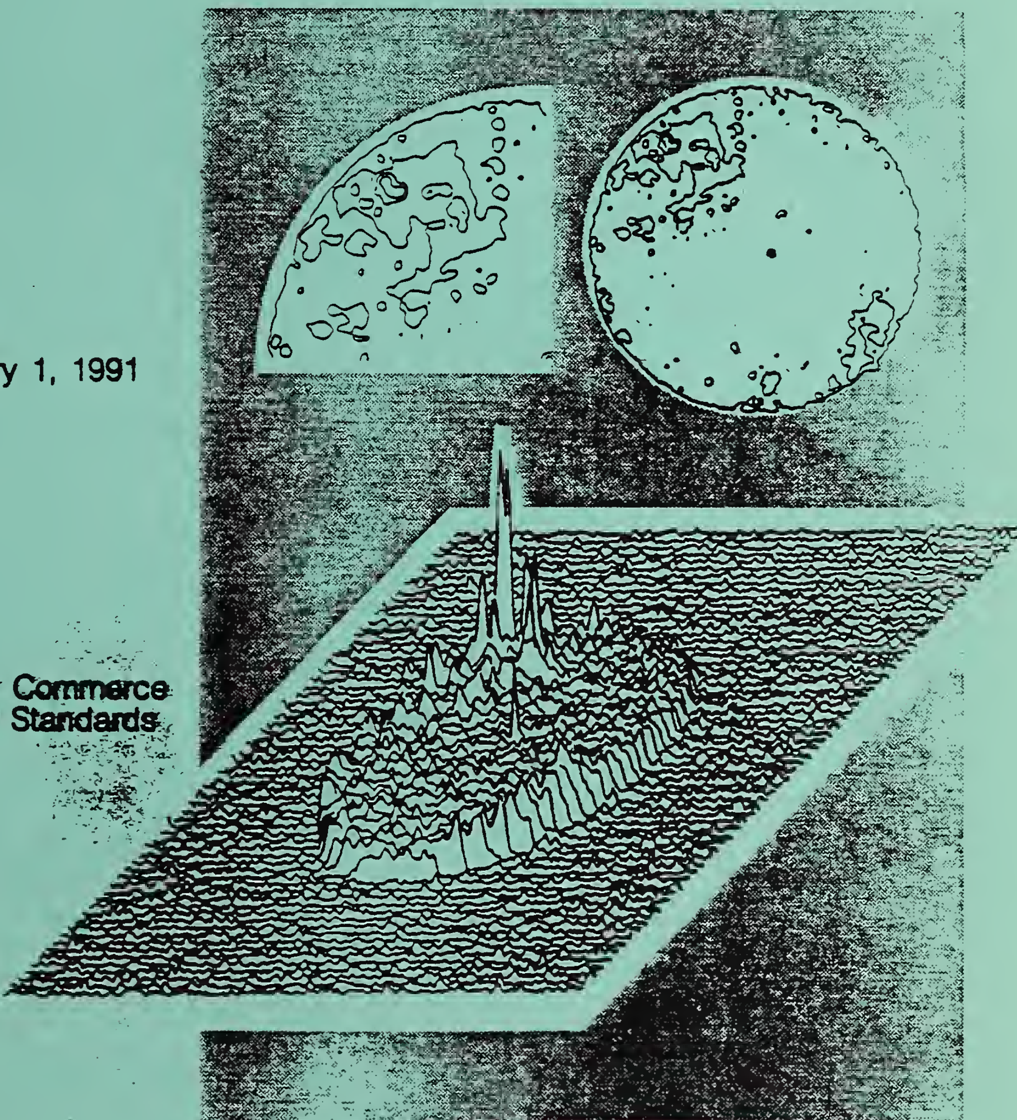
NONDESTRUCTIVE EVALUATION



NAS-NRC
Assessment Panel
January 31-February 1, 1991

NISTIR 4393
U.S. Department of Commerce
National Institute of Standards and Technology

Technical Activities
1990

Magnetic resonance image or stack plot of water distribution in a silicon nitride water slurry. The x and y plane represent the cross section plane of the sample and z direction (out of the cover) represents water concentration. Research by Dr. Pu Sen Wang, Ceramics.

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H.T. Yolken, Chief
J.P. Gudas, Deputy

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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

Fiscal Year 1990, the start of a new decade, saw substantial progress on the proposed major expansion of our activities into the broader arena of intelligent processing of materials as stated in our strategic plan for the 1990s. This plan broadens the scope of the NDE program to include the major focus on research for an integrated approach to intelligent processing of materials. This approach utilizes NDE for process control coupled with computerized decision-makers based upon process models, data, and expert systems. This approach is intended to provide closed-loop control of the evolution of microstructure during materials processing. Current efforts on intelligent processing at NIST include research on high pressure gas atomization of metal powders, hot isostatic pressing of titanium aluminides (with DARPA support), and electronic solder connections (with U.S. Army-Harry Diamond Laboratory support). Planning is also underway with the American Iron and Steel Institute (AISI) for NIST to take part in their program on intelligent processing of steel.

Our research efforts were focused this year, as they were the previous year on four high-priority activities: NDE for Ceramic and Metal Powder Production and Consolidation; NDE for Formability of Metals; NDE for Composites Processing and Interfaces; and NDE Standards and Methods. This focused approach provides enhanced synergism between our NDE research efforts and NIST efforts in materials science and engineering, and provides greater interactions with industry.

The researchers working on the NIST NDE Program have made a number of significant scientific and technical advances. Although this report contains descriptions of these advances, I would like to highlight a few of the more significant of these.

A consortium, consisting of Crucible Materials, General Electric, Hoeganaes (for the first two years), and NIST successfully completed a three-year program to develop the science base and technology for intelligent processing of rapidly solidified metal powders. The NIST principal investigators were Steve Ridder and Frank Biancaniello of the Metallurgy Division; George Mattingly, Cary Presser, Pedro Espino, and Serge Huzarewicz of the Center for Chemical Engineering; and Steve Osella of the Center for Manufacturing Engineering. This group, working with researchers from our industry partners, developed a fundamental understanding of the liquid jet break-up leading to metal droplet formation. This understanding was based on utilizing a variety of in-situ measurement techniques such as Schlieren and high speed photography, optical holography, and pressure surveys, along with fluid dynamics modeling techniques. They also succeeded in applying in-situ particle sizing techniques to determine droplet size. In addition, they designed and implemented an intelligent control system including acquisition of hardware and development of software. As requested by our industrial partners, planning for a Phase II consortium to develop a second generation intelligent processing system for metal powder atomization is currently underway.

Last year, 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 DoD effort on the subject. Non-intrusive sensors for the soldering process are being evaluated and calibration techniques developed, computer programs for analyzing and evaluating the sensor signal are being formulated, and a data base on the physical properties of solder joint components is being generated. In addition, fundamental research activities are underway to develop a better understanding of the solder process, in order that a meaningful process model can be formulated. NIST researchers from the Metallurgy Division, the Materials Reliability Division, and the Center for Manufacturing Engineering are involved in this major project that has made significant progress this year.

Several new projects were started this year. An effort was initiated to develop eddy current methods for monitoring the electrical conductivity of carbon-carbon composite materials during processing. 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. If successful, this technique will prove useful as an element in a process control system. This project is being jointly carried out by General Dynamics Corporation, Fort Worth, Texas, and Arnold Kahn (Metallurgy Division) at NIST.

Another new project on nuclear magnetic resonance imaging (MRI) was initiated this year. Carried out by Pu Sen Wang of the Ceramics Division, this research is aimed at providing data for process modeling of ceramic and ceramic composite processes.

The prospects look bright for our Program over the next few years, particularly, for our expanded focus on intelligent processing of materials. I believe that our Office is moving toward having a major impact on American industry by fostering the intelligent processing concept. 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 and the ceramics industry. Technology transfer will be a major component of the cooperative NIST/industry research ventures in intelligent processing of materials.

H. Thomas Yolken, Chief
Office of Nondestructive Evaluation

For more information on projects in this report, kindly call 301/975-5727 or write to the Office of Nondestructive Evaluation, Materials Building, Room B342, National Institute of Standards and Technology, Gaithersburg, Maryland 20899.

TECHNICAL ACTIVITIES

A. NDE FOR CERAMIC AND METAL POWDER PRODUCTION AND CONSOLIDATION

This activity is mainly concerned with developing approaches, sensors, and procedures for nondestructively determining those properties of ceramic and metal powders and consolidated materials that relate to the quality and performance of the materials and manufactured parts. The emphasis is primarily on measurements that can be made during the manufacturing process to sense the pertinent properties of the product during critical stages of its formation and to provide the data required to control the process. The activity includes the intelligent processing of rapidly solidified metal powders, the use of capacitative array probes for characterizing ceramic materials, and a new project on nuclear magnetic resonance imaging for ceramic processing.

- The project on metal powders is aimed at developing an intelligent processing system that involves the development of a process model, the investigation of a variety of sensors and their performance in an actual inert gas/metal atomization facility, and an expert system for the process control. This project is viewed as a model system to obtain experience and insight on how to approach the complete problem of process sensing and control. This year, a three-year NIST/U.S. industry consortium project was completed with an impressive list of accomplishments which are summarized at the end of the report on Intelligent Processing of Rapidly Solidified Metal Powders. The approach taken to realize this goal was to develop (1) a fundamental understanding of the liquid jet breakup leading to a process model of droplet formation; (2) real time techniques for measuring droplet size and its distribution; and (3) an expert system for control of the atomization process based in part on the process model.

  A proposal to form a research consortium by the National Institute of Standards and Technology entitled "Intelligent Processing of Rapidly Solidified Metal Powders by Inert Gas Atomization-Phase II" (9/17/90) was prepared. The objective was to organize a consortium to plan, financially support, and conduct research toward a second generation integrated system for this purpose.

- The capacitative array probe, a set of electrodes with a prescribed alternating potential and used in a differential mode, was used to scan a plexiglass slab with side-drilled holes that simulate defects. A perturbation theory is being developed to model the effect of small inhomogeneities on probe operation, and to aid in the interpretation of such measurements. The differential mode is useful in the inspection of materials which have inhomogeneous properties. However, if voids are uniformly distributed, it is necessary to use a probe that measures the absolute value of the dielectric constant, ϵ, rather than its changes. Two theories are being studied to solve the problem of measuring absolute ϵ.

To calculate the fields generated by the probe and to simulate the probe response to various flaws, a two-dimensional solution of Laplace's equation was obtained by finite element analysis. With such results, the validity of two theories of probe performance can be assessed.

- A new project on nuclear magnetic resonance (NMR) imaging for material applications was initiated this year. In particular, the distribution of material properties can be obtained by detecting the NMR active nuclei such such as the hydrogen proton in the component or material. This was demonstrated by obtaining a contour plot of water in a slice of a tube containing Si_3N_4/H_2O slurry. The intensity of the water, which was readily mapped, indicates the absence of the ceramic material. This capability provides a way of producing a three dimensional image by stacking the plots of successive slices. The distribution of water and ceramic in the slurry is an important factor to control in ceramic processing.

Intelligent Processing of Rapidly Solidified Metal Powders

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The objective of the Phase I consortium effort was to develop an intelligent processing system for controlling particle size which would be applicable to a broad range of metal atomization systems currently used or under development by U.S. industry (e.g. free-fall or confined gas atomization and spray forming). The specific research facility used during this three year study was the NIST gas atomizing system. The research program focused on the modeling of droplet formation, advanced sensing techniques for on-line particle size measurement, and development of an intelligent control system. Toward this end, four research groups were assembled into a team, each with specific goals and timetables:

(1) the Fluid Flow Group studied fluid dynamics as related to gas atomization;

(2) the Optical Diagnostics Group developed particle size sensing capabilities;

(3) the Intelligent Control System Group provided computer control software and hardware;

(4) the Metal Atomization Group operated the atomization system and coordinated the activities of the other groups.

By the completion of the third year of this research program, the following accomplishments were realized [see refs. 1-13]:

o Successful high speed cinematography and laser holography studies that revealed the operative liquid disruption sequences and intermittent characteristics of metal atomization;

o Development of mathematical models of supersonic jets which describe the aspiration effect seen in gas atomizers and provide a means to evaluate alternate atomizing geometries;

o Gas flow measurements which quantified the gas jet produced in the NIST atomizing system and identified the important gas pressure control strategies;

o Evaluaton of performance of several particle size measurement techniques followed by installation of a Fraunhofer diffraction device in the NIST metal atomization system to obtain real-time particle size measurement data;

o Construction and use of a continuous dry powder flow facility for particle size sensor development;

o Development of innovative pattern recognition software which resulted in dramatically improved time resolution of the particle size sensor (from .05 hz to >2 hz);

o Design, installation and successful operation of an expert system controller and user interface to run the atomization system. A key element of this program is an expert control system shell with which an operator is capable of rapidly prototyping control system architectures and strategies;

o Completion of parameterization studies of particle size distribution versus various processing conditions to evaluate the hardware modifications and assess the expert control system.

During the past year, the following accomplishments contributed to the successful demonstration of the potential to control powder particle size through intelligent processing.

Process and Model

Efforts continued in the study of droplet formation mechanisms within the specific "consortium" atomization die (see refs. 2-7 for schematic drawings). This die produces a circular arrangement of supersonic gas jets that impinge on a central, downward flowing, molten metal stream that is aspirated through a delivery tube attached to the bottom of a crucible. A series of N_2 gas atomization experiments provided several interesting results relevant to droplet formation in gas atomizers. Previous microscopic data has shown the presence of hollow spheres in Ar atomized metals, especially in sizes larger than 80 μm. When N_2 was substituted for the atomizing gas this porosity was effectively eliminated as shown in Figure 1. This phenomenon was observed in 304SS, René 95 and high alloy tool steels and may be the result of surface tension or viscosity changes associated with the formation of various metal nitrides. Another common occurrence in powder micrographs is small particles occupying the interior of larger particles. The small particles appear to have solidified first before the larger droplet forms around them [10]. An explanation of these particle microstructures will require further investigation.

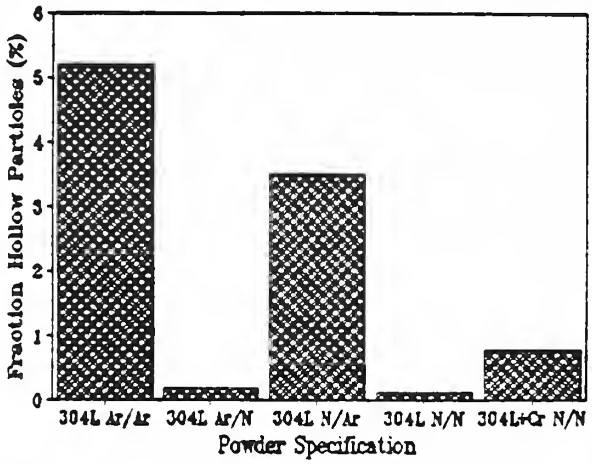

Figure 1. Frequency of hollow particles observed in polished cross sections of five powder types

Process Parameterization

The liquid disruption study discussed was part of an extensive investigation aimed at determining the system identification and control function of the Supersonic Inert Gas Metal Atomizatizer system. This investigation has included several atomization runs on various alloys and pure metals. (e.g., 304 SS, René 95, Vascomax 350, pure Cu, and several Cu, Al, Sn and Ni based alloys.) Figure 2 shows how three computed data quantities (the die pressure, gas/metal mass flow ratio and P_e/P_r) from numerous runs of argon atomized René 95 affect the particle size distribution (shown here as DSA = Dimensionless Surface Area = S_p/S_T where S_p is the total surface area of 1 g powder mass and S_T is the surface area of a 1 g mass sphere). The data shown in Figure 2 indicates that maximum surface area (largest quantity of smaller particle sizes) is produced for this alloy when this atomization system is operated at a die pressure ≈ 1040 psig (7.2 MPa), gas/metal ratio ≈ 3 and P_e/P_r ≈ 30.

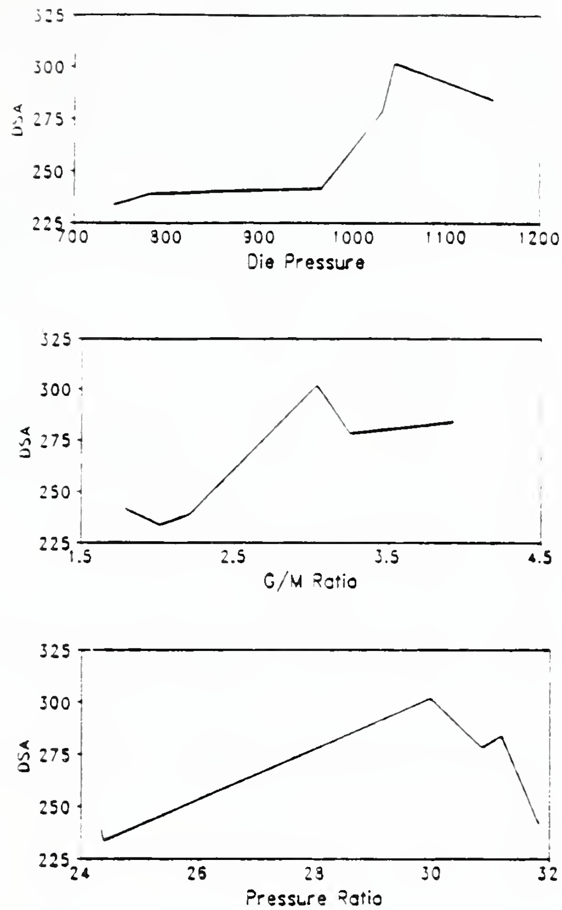

Figure 2. Dimensionless Surface Area (DSA) versus die pressure, Gas/Metal ratio and gas jet pressure ratio (p_e/P_r)

This study also showed how die pressure, gas/metal ratio and P_e/P_r are "related". Figure 3 contains three plots of the various combinations of these quantities. Correlation would be indicated if any of these plots had monotonically increasing lines connecting the data values. This figure shows how metal flow rate and die pressure (or P_e/P_r) are interrelated and therefore interfere with the correlation between gas pressure (both die pressure and P_e/P_r) and the gas/metal ratio for lower pressure values. As gas pressure increases beyond the point where maximum aspiration is achieved, the gas/metal ratio rises with rising gas pressure. The plot of P_e/P_r vs. die pressure displays the effects of a closed atomizing chamber with fixed gas exhaust rate. Atomizing systems with these characteristics will not show good correlation between die pressure and pressure ratio because P_r is strongly affected by P_e. (P_r is also dependent on gas temperature and, therefore, the metal pour temperature, heat capacity, heat of fusion and flow rate.)

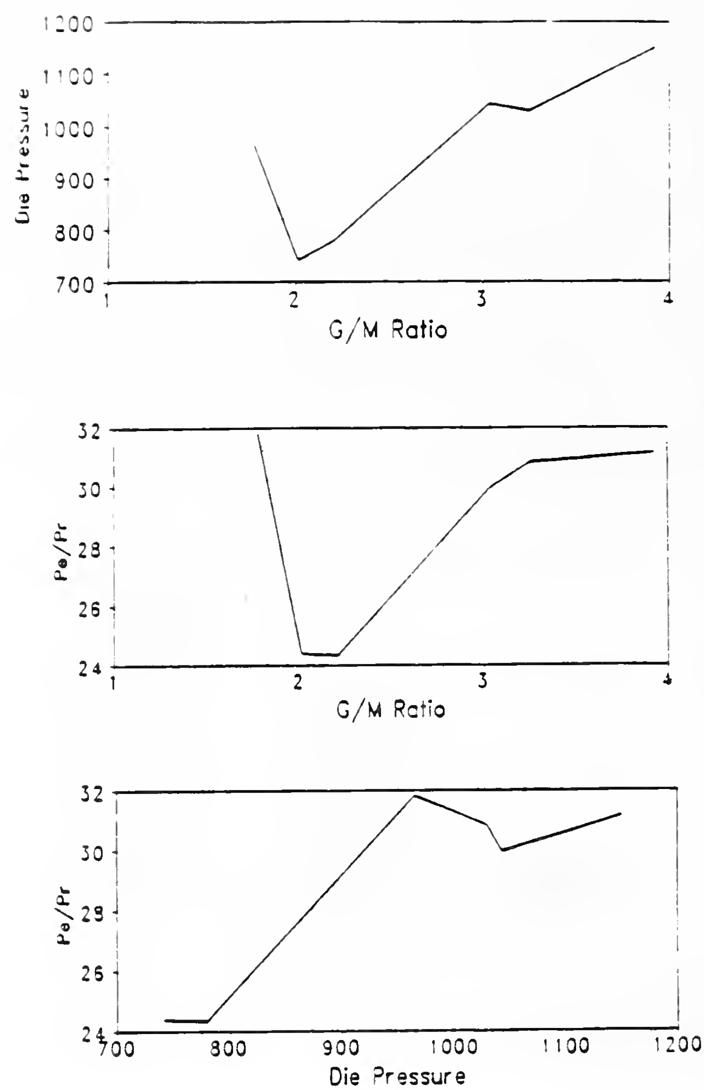

Figure 3. Die pressure and (P_e/P_r) versus Gas/Metal ratio and (P_e/P_r) versus die pressure

Intelligent Control System

A new Data Acquisition and Control (DAC) system (determined to be vital to the integration of an advanced control system for the atomization process) was installed. This system includes a digital I/O interface board, a micro-computer based DAC, and a low-voltage switchboard for manual control of the atomizer's process actuators (this switchboard simplifies the implementation of automatic control by TTL-level signals from the computer). A serial interface was installed for communication between the DAC and the Intelligent Control System (ICS).

Figure 4 diagrams the architecture of the atomization ICS. The high-level controller refers to a physically remote microprocessor with which an operator can interact via the keyboard and graphics monitor of the operator interface. The world model is a database describing the performance characteristics of the atomizer as provided by knowledgeable experts and by continuous updates during atomizer operation. The local low-level controller is provided as a "stand-alone" device capable of maintaining status quo during the operation of the atomizer as well as providing the necessary process feedback. The product sensor is the previously described in-situ particle sizer. This sensor is connected to the high-level controller directly, bypassing the local controller, so as to improve response of the control system to changes in particle size distribution.

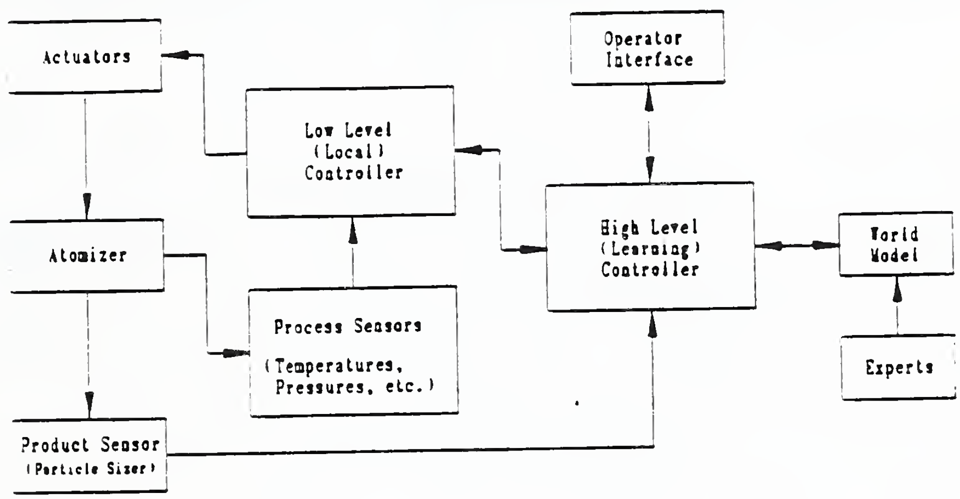

Figure 4. Atomizer control system

The run supervisor, via the operator interface, can plan and execute atomization runs, monitor process conditions during runs, and analyze process data subsequent to the runs. The ICS is responsible for reconciling user requests with current process conditions in order to meet production objectives. The reconciliation is accomplished via heuristic process control knowledge provided by the operator(s) as well as learned knowledge acquired inductively during atomization experiments.

The high-level controller's decision-making strategy is based on the principle of Hierarchical Control (17). In this approach, the strategy is considered to be goal-directed. The controller's highest level goal, the objective, is decomposed into a number of more elementary sub-goals. All of the sub-goals have to be achieved in order to accomplish the objective.

To carry out the necessary goal-decomposition, the controller is divided into a number of control modules. The control modules are related in such a way that a next lower level module is responsible for the accomplishment of one or more of the sub-goals. Each sub-goal becomes the objective for that lower level module resulting in a recursive definition of the overall objective.

The knowledge required to decompose a module's objective is called the control knowledge and is stored in its knowledge-base in the form of facts and rules. A fact in the controller's knowledge-base has the following form:

(relation item-1 [item-2 ... item-N])

The items in the fact are associated by the given relationship. An example of an asserted fact is

(equal-to Die-Pressure 1000.00)

which could mean that the measured pressure in the die plenum is 1000.00 psi. The exact meaning of a fact depends on its use in a rule.

A rule specifies the action that is to take place when a given condition is encountered. As a result, a rule has the following form:

IF condition THEN action

The condition, also called stimulus or state, is made up of a number of facts. When all of the facts which make up a rule's condition have been asserted (entered in the knowledge-base), the rule is said to be triggered. More than one rule can be triggered simultaneously. A conflict-resolution policy is used to select from among the triggered rules. When a rule is selected, it is said to be fired. When a rule is fired, the rule's action, also called response, is performed.

If only one rule can be triggered at any one time, the rules in the knowledge-base make up what is called a state-table. If the policy used to select a rule is variable, the controller is considered to be adaptive. If the manner in which the selection policy is varied leads to a better performance, the controller is said to be learning. Note that because in a state-table each condition only has one associated action, no learning can take place. All of these methods make up the intelligent decision-making of the atomizer's process controller.

As currently designed, most of the controller's knowledge is of the preprogrammed type which is acquired from an understanding (albeit incomplete) of the atomization process. For example, the interrelationships between process variables as learned in the parameterization study (described previously) have been incorporated in the current knowledge base for gas pressure management.

The gas pressure feeding the atomizing die is dynamically controlled to maintain constant P_a/P_r provided P_r stays below a predetermined safety setpoint (124 kPa ≈ 18 psia) at which point gas pressure is reduced until P_r drops below the safety setpoint. The controller's software is designed to permit easy development and modification of the control knowledge and architecture used to execute atomization runs. As new knowledge is acquired, it can quickly be added to the knowledge-base. Similarly, as new sensors or actuators are added to the atomizer system, additional control modules can be incorporated in the controller.

The use of learning techniques to control a process is predicated on the ability to perform a number of trial-and-error experiments. This requirement, in general, necessitates the use of process models on which to train the controller off-line before the controller is allowed to fine-tune its control on-line. In this regard, plans have been made for experiments involving learning control of a surrogate model of the atomization process.

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Capacitive Array Probe for Characterization of Ceramic Materials

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The capacitive array probe is a set of electrodes having a prescribed potential, and operated at low frequencies where the resulting field is electrostatic. By varying the probe potential pattern, the shape of the field can be varied, resulting in potentially attractive features for the quantitative characterization of dielectric materials.

The use of this device is being investigated for rapid, inexpensive, nondestructive evaluation of ceramic materials. It is especially important to detect defective materials in the green state (prior to sintering). Examples of defects which may be detected include voids, inclusions, excess (or deficiency) of organic binders, and improper densification.

Differential Mode/Through Transmission Probe

In past years, the use of a one-sided capacitive probe (all electrodes in same plane, above the specimen) was studied. This probe suffers from one potential drawback. When used with high-dielectric materials, the field generated by the probe may be excluded from the specimen. The high polarizability of the surface effectively generates a surface charge which shields the interior of the specimen from the probe's electric field. To overcome this problem, the system shown in Figure 1 is being developed. The probe uses four electrodes above the specimen which couple to a conducting (grounded) plate below the specimen. The electrodes above the specimen are at the same non-zero potential, which causes the electric field to penetrate the specimen. The inner electrodes are "active", and the outer two are "guards" which confine the fringing of the electric field and force more of it to penetrate the specimen in the direction normal to the specimen surface.

The probe is operated in the differential mode. We have, in effect, constructed an amplifier consisting of the three operational amplifiers (shown by the symbol ▷ in Figure 1). Amplifiers U1 and U2 detect the voltage drops across precision resistors. These drops are caused by current through the resistors, which is influenced by the capacitance between the electrode and ground plate. This capacitance will change if, for example, the dielectric material directly beneath the electrode has voids or porosities. Hence, the differential amplifier U3 will generate an output voltage related to a change in dielectric properties of the specimen. In effect, the probe output is related to $\Delta\epsilon/\Delta x$, where $\Delta\epsilon$ = difference in dielectric constant, ϵ, under the inner electrodes, and Δx = separation between inner electrodes.

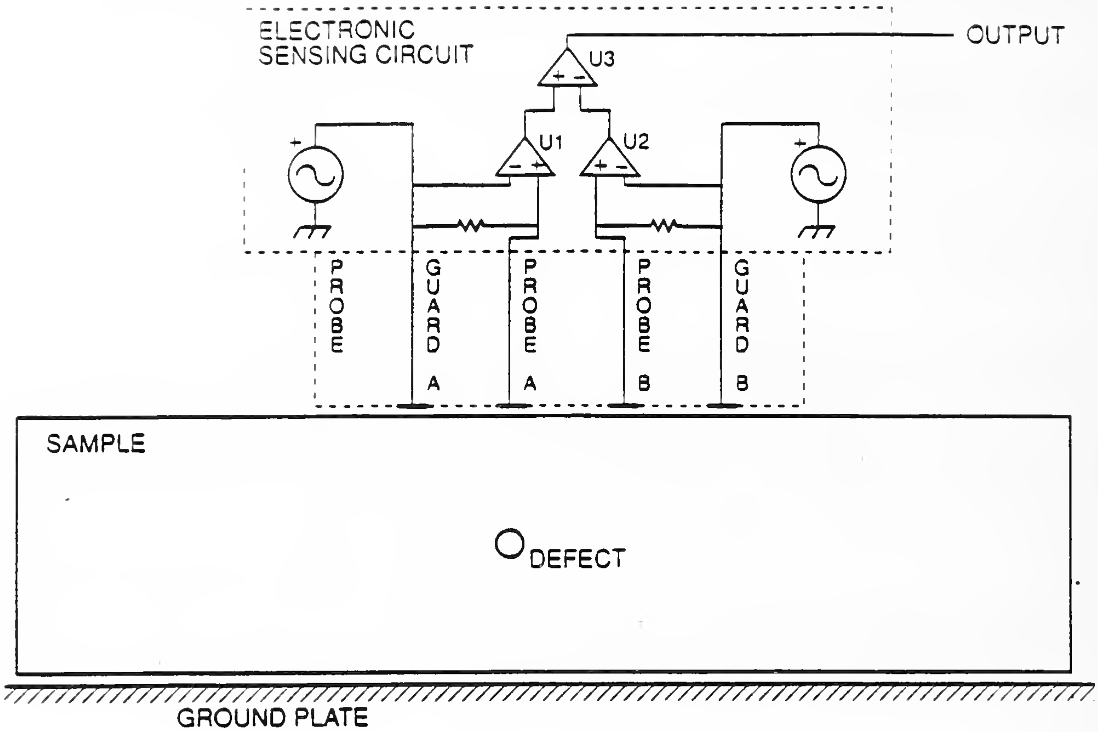

Figure 1. Schematic of experimental setup for through-transmission experiment. Dotted line encloses electronics and probe electrodes, which are housed in one enclosure. Probe A and Probe B are active electrodes, with guards A and B to confine fringing field.

Successive versions of the electronics were constructed and improved resulting in repeatable results. Scans were made over dielectric material with artificial defects. The result of one such scan is shown in Figure 2. The defects here were side-drilled holes in a plexiglass slab ($\epsilon = 3.7$). The slab was 12 mm thick, and the holes (of diameter 4.7, 3.2, and 1.6 mm) were drilled in the mid-plane (6 mm depth). The smallest detectable hole (diameter = 1.6 mm) corresponds roughly to a void volume fraction of 2.5%. An approximate analysis shows that our current system should detect voids having volume fractions in the range 1–2% for common ceramic materials such as alumina ($\epsilon = 10$). Note that the amplitude and phase are approximately mirror images of each other in Figure 2. An analysis of this effect is being performed, which may reveal that phase measurement may be the most sensitive means of detecting small defects. Our studies have shown that system sensitivity can be improved by: increasing probe voltage; increasing operating frequency; and increasing probe impedance. The electronics are being upgraded (e.g., by increasing the system bandwidth) to incorporate these potential improvements.

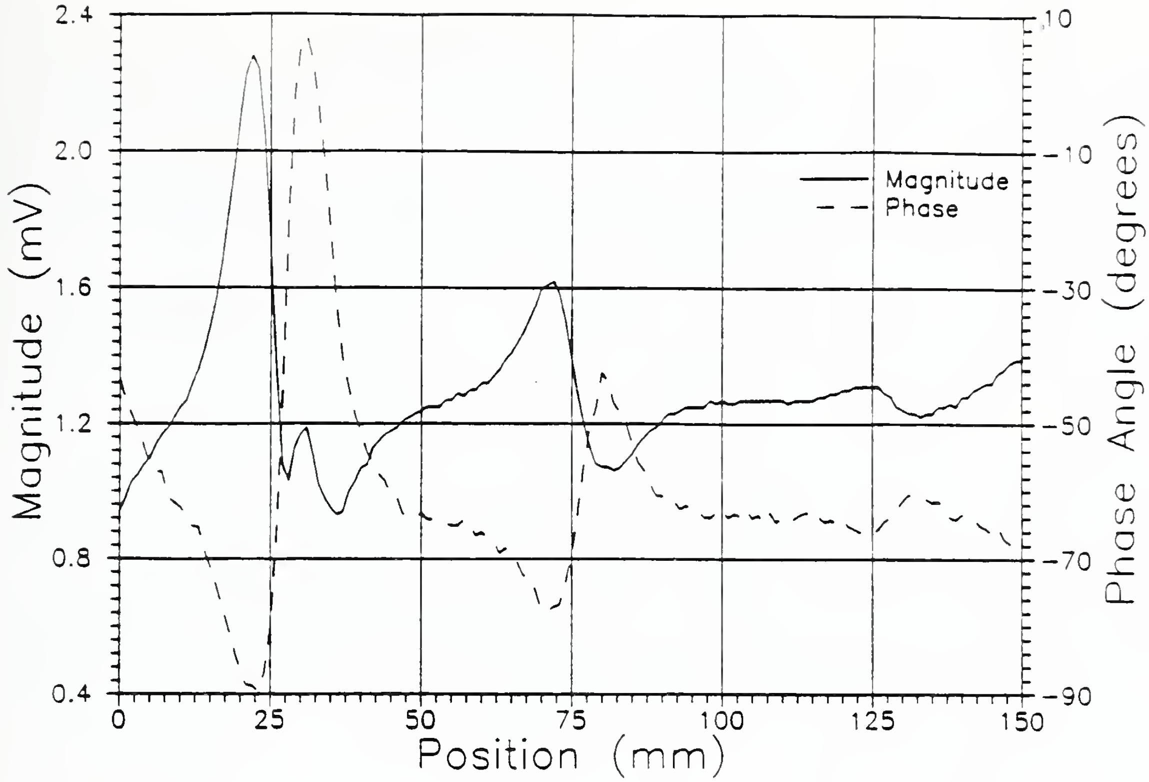

Figure 2. Differential voltage output (magnitude and phase) from scan of plexiglass sample with side-drilled holes. Holes are located at 25 mm, 76 mm, and 128 mm; they are 4.7 mm, 3.2 mm, and 1.6 mm in diameter, respectively.

A perturbation theory is currently being developed (in collaboration with Professor B. Auld, Stanford University), to model the effect of small inhomogeneities on probe operation. This theory will be used not only to interpret our experimental results in a quantitative fashion; it will also be used to invert the probe measurements. That is, the output of the differential amplifier U3 will be measured as a function of probe position and used to determine the amount of material inhomogeneity. A potential application is characterization of green-state ceramics to reject defective specimens (e.g., with voids, improper densification, etc.) prior to sintering. Another possible application is in processing of printed circuit boards, where variations in ϵ are to be avoided.

More realistic specimens were designed, to more closely model actual defects in ceramics. These specimens, when fabricated, will have voids or inclusions of known volume fraction and dielectric constant. The effect of these defects on specimen dielectric constant will be calculated, as well as the probe response to the defects. Measurements of probe response will be made and compared to theoretical predictions.

Absolute Mode/Inversion Problem

The differential mode is useful for the inspection of materials which have inhomogeneous properties. However, material may still be defective, even if homogeneous. For example, if voids are uniformly distributed, a differential probe will be unable to detect this. To overcome this, it is necessary to use a probe which measures the absolute value of the dielectric constant ϵ, rather than changes in ϵ. A first-generation probe of this type was previously developed, and shown to give results in good agreement with theory.

Preliminary studies evaluated methods of solving the inverse problem. At present, two theories, based on Refs. 1 and 2, are being investigated. The theory of Ref. 1, a first approximation, assumes a sinusoidal potential in the probe plane, which is also assumed of infinite extent. The exact theory of Ref. 2 models the case of a potential prescribed only over the probe electrodes. In both theories, the probe is over a dielectric half-space (no edge effects). The approximate theory gives a closed form solution for $\Delta Y(d, \epsilon)$, the change in admittance, and hence using it for inversion is somewhat simpler. The exact theory requires a more lengthy calculation to obtain ΔY, which must be evaluated on a computer. A comparison between the predictions of the two theories, normalized to agree at liftoff, near 0.2 mm, is shown in

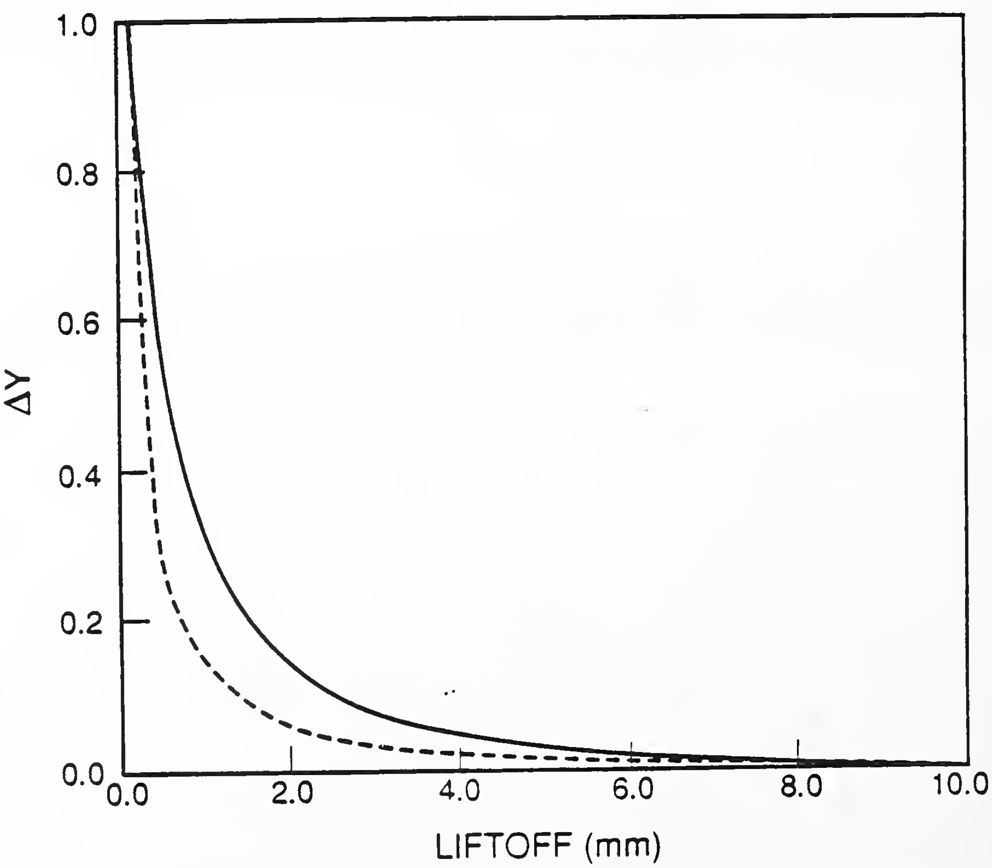

Figure 3. Comparison of approximate and exact theories for the case of a typical probe (4 electrodes 14 mm apart) above a halfspace with dielectric constant = 10. Approximate theory results are indicated by solid line; exact theory results are indicated by dotted line.

Fig. 3, for $\epsilon = 10$ (a common value for ceramics). For small values of liftoff (near 0.2 mm) the theories initially agree, and then diverge. For large liftoff, where $\Delta Y = 0$, the theories again agree, since the probe no longer senses the specimen.

In the simplest case, where the probe can contact the specimen, d will be known and ϵ can be directly obtained from the theory. Voids or inclusions cause a small perturbation about the nominal value, 7ϵ, and both theories give results which vary linearly with ϵ. The maximum difference in void fractions predicted by the two theories is about 1%. For unknown liftoff, it may be possible to linearize $\Delta Y(f,\epsilon,d)$ (where f = probe spatial frequency), for both theories.

Finite-Element Modelling

A two-dimensional solution of the Laplace equation, which is solved using the finite element method, was used to calculate the fields generated by the probe and to simulate the probe response to various flaws. The purposes of this are to evaluate the validity of the theories of Refs. 1 and 2 and also to evaluate probe sensitivity to buried inhomogeneities. For the differential probe, the program will determine the admittance change which results from inhomogeneities. It can be also used as a design tool to optimize probe geometry, since it will show how electrode configuration and voltage concentrates the electric field. Figure 4 shows the through-transmission probe configuration considered by the numerical model for the case of three (large) buried inclusions. Each of the probe fingers is fixed at a potential of 1.0 for this case. The figure shows the potential contours for the buried inclusion problem. The large value of ϵ ($\epsilon = 100$) for the inhomogeneities causes the potential field to be disturbed.

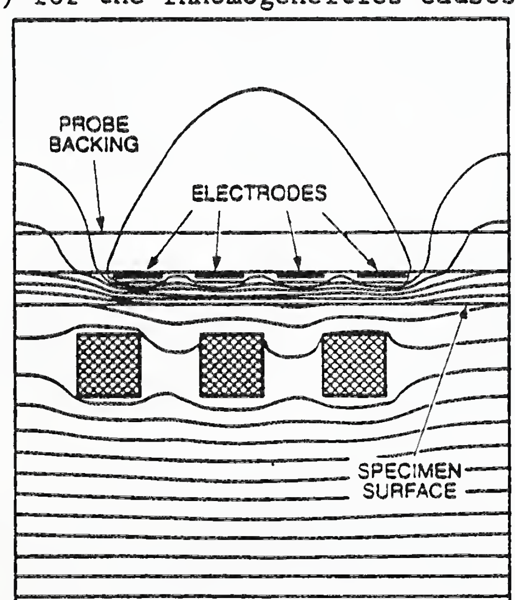

Figure 4. Finite element modelling of typical probe configuration in through-transmission mode. Probe is mounted on material with dielectric constant of 3.9. Sample dielectric constant is 3.7. Three square inclusions (dielectric constant = 100) are embedded in sample. Inclusions are indicted by cross-hatching. Contour intervals are equal to 0.05 volts.

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Nuclear Magnetic Resonance Imaging For Ceramic Materials

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Nuclear magnetic resonance (NMR) imaging provides a unique, in-situ material diagnostic technique for internal mapping of multiphase distribution and profiling the chemical and physical characteristics of advanced materials. In a composite, for example, the binder distribution is a critical parameter that predicts its mechanical properties, thermal conductivity, material compatibility, reliability, and thus its applicability. The objective of this program is to establish a solid state NMR imager at NIST for ceramic process modeling and other NDE uses.

The distribution of materials properties can be monitored by detecting the relaxation times of the NMR-active nuclei in the component. Due to the nuclear spin dipole-dipole interaction in the solid state, the NMR spectroscopic signal will be very broad. To perform NMR imaging based on these unresolved broad lines is extremely difficult. Hence, performing NMR imaging on a high resolution liquid NMR signal will be a good starting point for such a task. A liquid imaging facility will also find valuable applications in determining diffusion, slurry characterization, etc.

To construct a picture from the NMR spectroscopic signals, a set of field gradient coils around the resonant probe coil are used to control the magnetic field strength and frequencies along the X-, Y-, and Z-coordinate so that NMR signals can be accumulated along these coordinates. The power and the numbers of selected excitations along X- and Y-coordinates determine the spatial resolution of the picture and Z-coordinate controls the thickness of this internal slicing.

During the past year, the imaging accessary was built into a Bruker MSL-400 NMR spectrometer. It includes a ^{1}H resonant probe with gradient coils, a selective excitation unit, and associated electronic equipment. The selective excitation unit generates pulses to detect the NMR signal in the resonant coil and the field gradient coil to profile the signal intensities along X-, Y-, and Z-coordinate. The pulse shapes are preset to offset the distortion produced by the eddy current. The pictures were constructed by an array processor of the Bruker ASPECT 3000 computer system.

Figure 1 shows a contour plot of water distribution in an internal slice across an NMR tube containing Si_3N_4/H_2O slurry. The distribution of water and ceramic in a slurry has a substantial effect on processing and ultimate properties of ceramic materials processed in this manner. The picture was taken at the proton frequency (400.130MHz). In a Si_3O_4/H_2O slurry, silicon nitride powders exist in solution in colloidal dispersion and the intensity of water represents the negative of the powder distribution in the solution.

Figure 1 thus represents a picture of Si_3N_4 distribution in the slurry of 9.7 mm diameter, cross-sectioned at a specific point. Each isle represents an area in which the water concentration is different from its neighbors. Note that at the upper left quadrant the structure is very complex. This suggests a heterogeneous distribution of the silicon nitride in the slurry. The plot of this quadrant is shown on the top separately for clarity. At any point in the picture, the water intensity along the horizontal or vertical direction can also be profiled. This capability provides a methodology to construct a three dimensional picture by stacking the plots of successive slices. Figure 2 is an example of such a plot constructed from 72 profiles. Note the high water intensity regions at the upper left quadrant agrees very well with the contour plot in Figure 1. A stack plot up to several hundred profiles can be easily performed to examine the internal material distribution at any desired section. A multiple slicing technique, which will be able to take several slices at various parts of the sample simultaneously, is under development.

Figure 1. Contour plot of water distribution in a silicon nitride/water slurry.

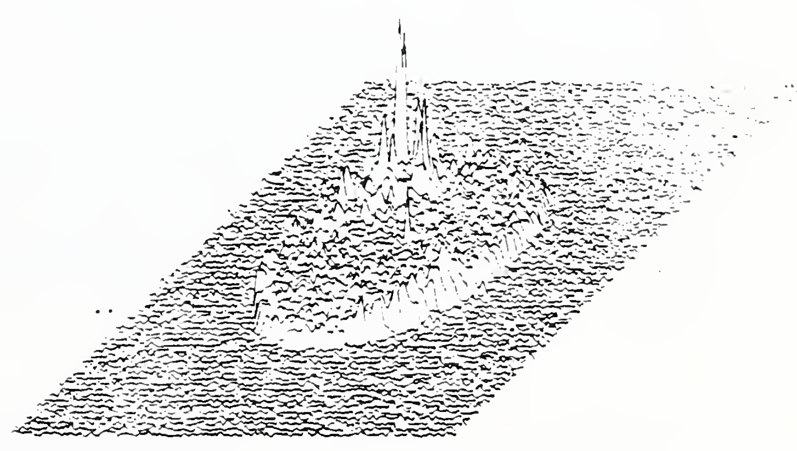

Figure 2. Stack plot of water distribution in a silicon nitride/water slurry.

B. NDE FOR FORMABILITY OF METALS

The goal of this activity in the Nondestructive Evaluation Program is to develop generic approaches, sensors, and procedures for quantitative NDE of metals during the forming process. The emphasis is on measurements that can be made on the production line to improve process control rather than developing inspection techniques for post-manufacturing inspection. Current efforts in this activity include the development of NDE temperature sensors based on eddy current techniques for determining the internal temperature distribution in hot aluminum during processing, development of an in-process ultrasonic monitor for metal grain texture in manufacturing of sheet metal products, and utilization of magnetic NDE techniques to characterize steels being processed. Examples of accomplishments this year include:

- During manufacture, the determination of internal temperature distribution of aluminum during various processes for process control has been a troublesome problem for the aluminum industry. In a joint effort between NIST and The Aluminum Association, past years' theoretical and experimental results showed that a multifrequency eddy current measurement approach in the 100 kHz to 1 MHz range provided an NDE temperature sensor for process control. This year the feasibility of the approach was demonstrated for moving sheet and plate. However, additional problems associated with chemical composition variations still need to be resolved.

- The formability of sheet metal, which in large part depends on grain texture or grain orientation, is a significant process variable in the manufacture of such diverse items as beverage cans, appliances, and automotive panels. In order to improve process control, a noncontacting ultrasonic method was developed over the past few years. This year's research in collaboration with Ford successfully developed and demonstrated a prototype ultrasonic measurement device for determining texture in thin ferritic steel sheet.

- The measurement of Barkhausen noise is used to characterize the metallurgical and stress state of materials. Research at NIST has produced a new method for characterizing this noise (named the jumpsum method). The method uses only a single hysteresis cycle to collect the data, which can lead to a significant time saving in a field or production environment. This method clearly showed different responses for different aging treatments of austenized steel, and the markedly different responses from the surface and unhardened core of a case hardened sample of 1050 steel obtained from a production run of automobile axles at a General Motors plant.

Eddy Current Temperature Sensing

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The joint NIST-Aluminum Association project on temperature measurement in extrusion processing and sheet and plate rolling had as its objectives in 1989-1990: (1) the analysis of the velocity effect in through-transmission eddy current sensing; (2) the analysis of alloy composition effects on the temperature measurement; and (3) the construction of a wheel-mounted sensor for measuring temperatures along the length of a billet. The first and third objectives have been accomplished. Measurements relating to composition effects have been made, and analysis is currently being performed with the assistance of the Statistical Engineering Division.

The through-transmission sensor is based on the induction of AC eddy currents in the moving extrusion or sheet by AC driving currents in the transmitting coil. The signal received in the secondary coil is sensitive to the temperature dependent electrical conductivity of the flat aluminum product, facilitating a temperature measurement. Calibration against thermocouples was performed in the laboratory using extrusions and flat-rolled sheets heated in a furnace, removed, and placed in the sensor to cool in air during the measurements.

Velocity effects observed in a previous plant test (see ref. 1) have been studied theoretically and the model verified empirically by rotating an aluminum lithium alloy disc in a lathe, simulating linear velocities attained in high-speed rolling. (The aluminum lithium alloy had a room temperature conductivity comparable to that of rolled can-stock aluminum alloy 3004 at elevated rolling temperatures.) For non-zero velocity, a family of impedance curves was generated, similar to the zero velocity curve, but reduced in height at the low frequency limit. The laboratory sensor is shown in Figure 1. Figure 2 shows theoretical impedance curves for the tangential velocities at the coil centerline and the corresponding measured points. Discrepancies between the calculated and measured values were due to a simplified theoretical model based on a dipole model for the transmitter and receiver coils. However, we consider the data and theory to be sufficiently close to verify the mechanism of the effect and to warrant more accurate calculation for practical cases.

Variation in alloying constituents allowable under specific alloy designations will give rise to apparent temperature shifts due to the corresponding changes in the electrical conductivity of the product. To determine the effects of such alloying constituents as Mn, Mg, Cu, etc. on the eddy current temperature determination, we have performed measurements on sheets of eight aluminum compositions allowable under the 3004 designation, prepared by Alcan International Ltd., Kingston R & D Centre. These samples were formulated so

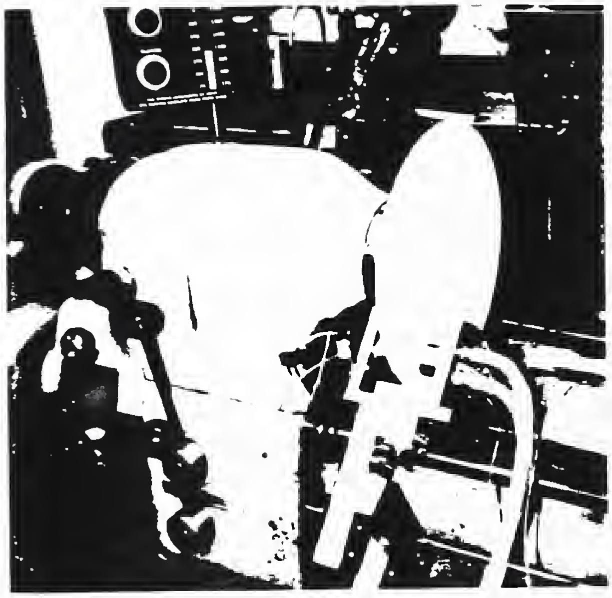

Figure 1. Sensor coils mounted on lathe bed for measuring velocity effects in through-transmission impedance measurements. Sample disc is of aluminum-lithium alloy.

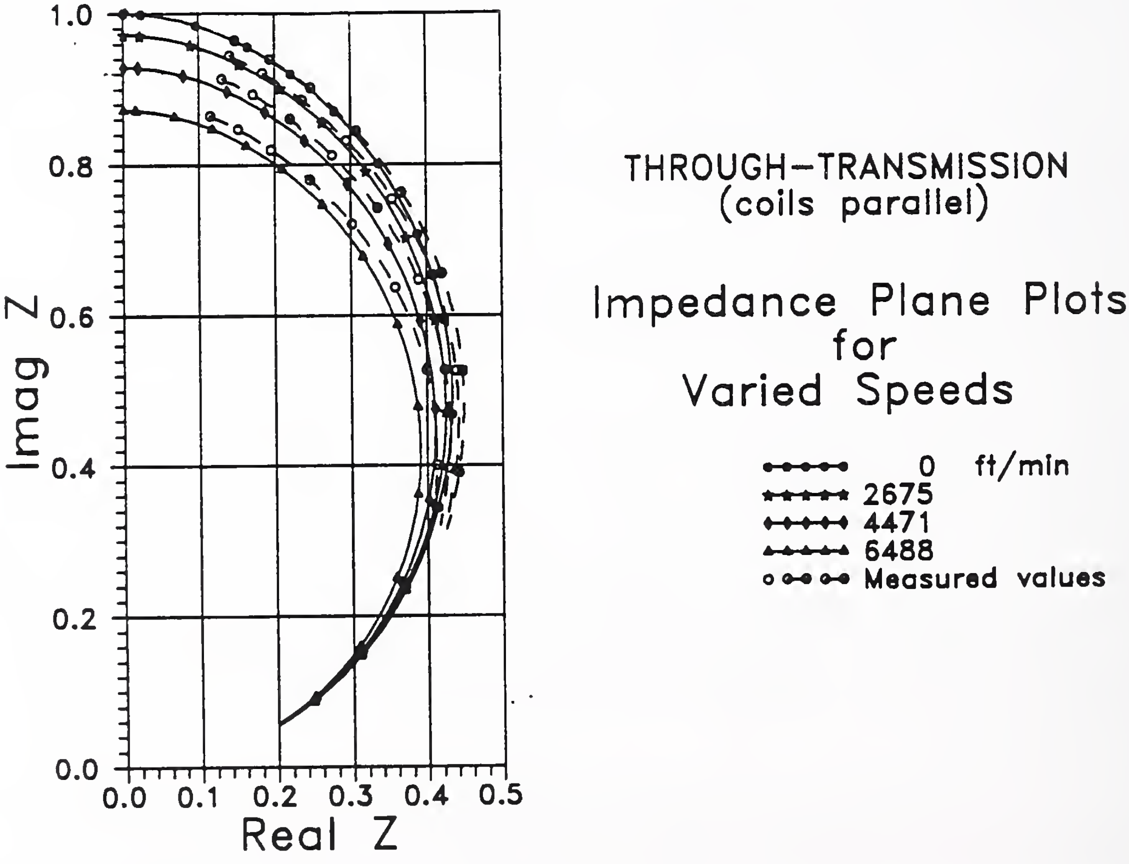

Figure 2. Through-transmission curves for the aluminum alloy disc of Figure 1, showing the effects of velocity. Solid lines are calculated using a dipole model for the coils; dashed curves are corresponding measured values. Speeds are tangential velocities of the disc at the center-line of the coils.

as to facilitate the statistical determination of composition effects, i.e., high and low Mg, Mn, etc. The Statistical Engineering Division of NIST designed a two day test and analyzed the resultant data, performing a linear regression analysis relating the eddy current impedance measurements to original melt compositions and sample thickness. If all the constituents were in solid solution during the measurements then the theoretical model would provide an accurate estimate for the temperature correction of 3004 sheet when thickness and composition are known. Because of wide variations of thickness within the Alcan R & D sheets, we repeated the test with the more uniform commercially rolled 3004 sheet supplied by Alcan, The Aluminum Company of America (ALCOA), and Commonwealth Aluminum Co. The Alcan commercial sheets fitted very well with the predictions of the statistical model. However, the Commonwealth and ALCOA sheets did not correlate well; the differences are likely due to differences in the amount of Mn in solution, arising from differences in thermal histories. Table 1 shows the variabilities (estimated standard deviations) of the temperature measurements for specimens of similar composition and for repeated measurements on the same sample.

Table 1. Similar composition specimen variability and same specimen repeatability for the two experiments. Variabilities shown are expected standard deviations in temperature determinations by The Eddy Current System.

	Similar composition variability	Same specimen variability
Laboratory samples	8.6 °C	2.3 °C
Commercial samples	6.6	3.5

The present project activity is concerned with quantifying the sources of variability and finding methods to control or correct for them. The previous measurements will be repeated on samples of alloy 1350, which is of 99.5% pure aluminum. These samples will have minimal variation of dissolved alloy components and it is expected that sample to sample variations in sensor temperature readings will be comparable to that of repeated measurements on the same sample.

We are grateful to Dr. Eric S. Lagergren of Statistical Engineering for his work on the analysis of data.

References:

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Ultrasonic Measurement of Sheet Metal Formability

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Steel sheet is an important component of many consumer goods. For example, the automotive industry is itself a prime consumer of steel sheet, which is used for body parts. This continues to be true, in spite of great improvements in component materials technology. In fact, the steel industry is now beginning to produce new types of sheet which has improved formability, and thus, can reduce vehicle weight and improve fuel economy.

Formability is characterized by the so-called plastic strain ratio, or r-value. The r-value traditionally has been measured by cutting tensile specimens from a rolled sheet at different angles to the sheet rolling fracture. The specimens are plastically deformed, and the (true) strains in the width (ϵ_w) and thickness (ϵ_t) directions measured. The r-value is then given by: $r = \epsilon_w/\epsilon_t$.

In forming operations (such as making hoods and door panels), the sheet is plastically deformed by a series of presses in a stamping line. As the sheet flows, its thickness will be reduced. Too much thickness reduction can result in a defective part (tearing, pin-holing, etc.). On the other hand, if too little plastic flow (in the plane of the sheet) occurs, the part may not fit when assembling the automobile body. Hence, not only is it desirable for the automobile manufacturer to know the r-value from a simple test; it is also desirable to know if any variability in the material occurs from place to place in the sheet.

For several years, NIST researchers have been working towards the goal of a totally nondestructive, on-line system for sheet formability measurement. With increased resources available in FY '90, progress towards this goal has been greatly accelerated.

It is known from theory that the velocity of ultrasonic waves, propagating in the plane of the sheet, can be related to the r-value. Preliminary measurements at NIST had shown that a particular type of guided mode was more promising than other modes; i.e., it showed a better correlation with mechanical measurements of r-value.

An improved correlation was found when account was taken for the effect of any coating (applied to the sheet for corrosion resistance). For zinc-coated steels of U.S. manufacturers, the coating does not seem to affect the r-value; however, it changes the effective density and stiffness of the sheet. Using the (known) coating thickness, it is possible to infer the velocity in the steel from the measured velocity in the zinc-coating/steel sheet "composite". (This method for doing this was developed by Professor M. Hirao, Osaka University, Japan.) The results are shown in Figure 1, which indicates that $\bar{r}$ can be resolved to about 0.1 or better.

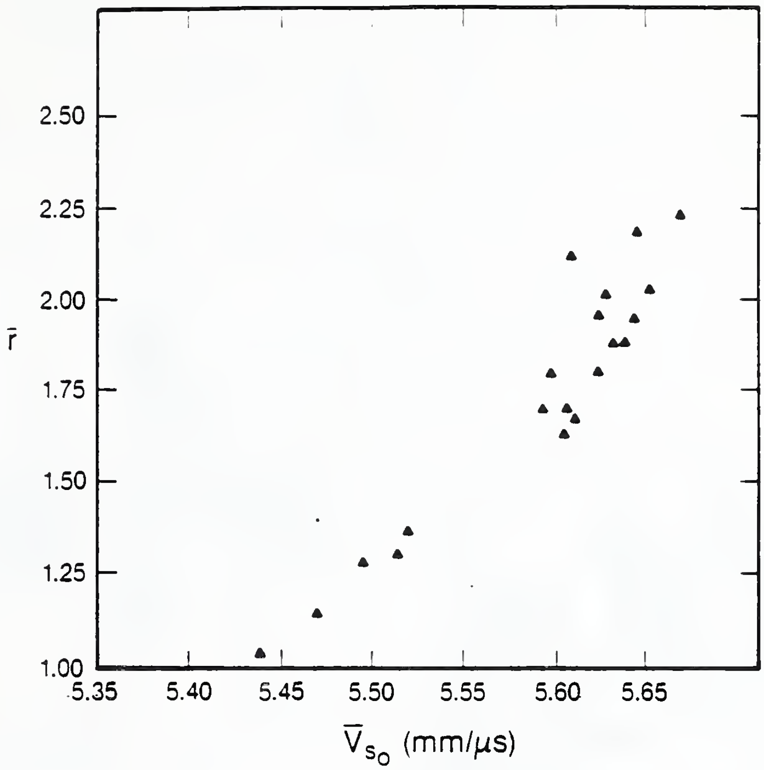

Figure 1. Correlation between average r-value, $\bar{r}$, and the average in-plane velocity, V_{s_o}, of a guided wave.

At present, NIST researchers are collaborating with Professor Hirao (currently a guest researcher at NIST) on improved methods for r-value prediction with a goal of resolution 0.05 in r-value. Various metallurgical theories are being explored with the ultimate goal being a model which will have as its input measured ultrasonic velocities, and have as its output the r-values in three different directions in the sheet. The model should be implemented on a PC and make a prediction in a matter of seconds.

NIST researchers have also collaborated with Ford Motor Company in the successful development of a prototype r-value measurement device. This instrument uses noncontacting ultrasonic transducers called EMATs (for ElectroMagnetic Acoustic Transducers). EMATs require no couplant or intervening medium to couple sound from transducer to specimen (or vice versa). In the prototype device, a transmitting EMAT generates a guided wave travelling at a selected angle relative to sheet rolling direction. A receiving EMAT detects the wave, which is amplified and its arrival time measured. Measurements are made at 0°, ±45°, and 90° to the sheet rolling direction. The average velocity is calculated, and a correlation based on Figure 1 is used to predict the average r-value, $\bar{r}$.

The actual device is pictured in Figure 2. On the right is a rack of electronics developed entirely at NIST (in collaboration with Professor D.V. Mitrakovic, a guest researcher from the University of Belgrade). The electronics are modular, so that any defective unit can be removed and replaced by a spare. The electronics generate a high-current pulse to drive the transmitting EMAT, amplify the (low-level) signal detected by the receiving EMAT, and generate a signal to stop a counter (used for velocity measurements). The counter is shown sitting atop the electronics rack, and in the only piece of commercial gear in the system.

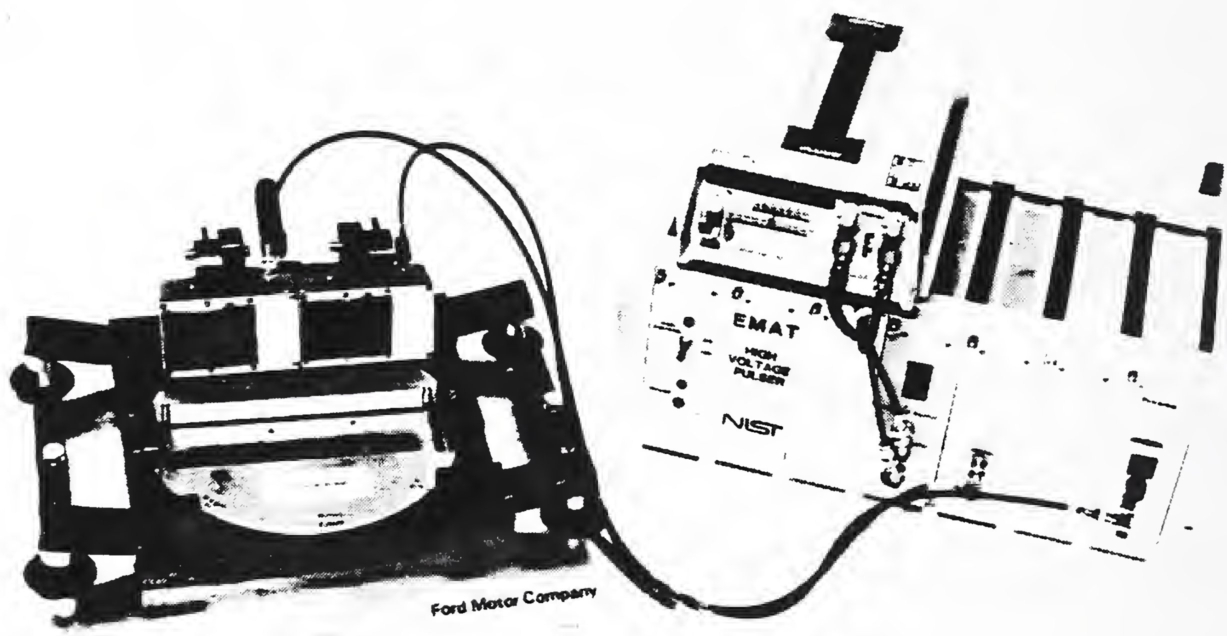

Figure 2. Prototype r-value measurement jointly developed by Ford Motor Co. and NIST.

The unit on the left-hand side of the picture consists of two EMATs, which can be lowered through a slot cut in a turntable. The turntable (lighter-colored area) is supported by a frame held on the sheet by four suction feet. The EMATs were designed and built by NIST. The turntable was developed and supplied by Ford Company and one of their vendors.

In operation, the EMATs (shown lifted off the plate in the photo) are lowered, using a handle, through the slot in the turntable. When the EMATs are in proximity to the sheet, sound will travel through the sheet from one EMAT to the other, and the counter will record the arrival time. The EMATs are then lifted off the sheet, the turntable is rotated 45°, and the measurement updated. When the four measurements have been recorded, they are then entered into a short program which runs on a PC, and calculates the value of $\bar{r}$ based on the correlation of Figure 1. The computer program, an operators manual, and a training video were provided to Ford by NIST as part of the technology transfer. At present, the device is being used in a research lab at Ford. It will be used to gather more data, some of which will be used to enlarge the data base of Figure 1. Then the device will be transferred to the quality assurance shop of the Dearborn stamping plant for further trials and evaluation.

In a further collaboration aimed at technology transfer, Ford and NIST are cost-sharing on the design and construction of a device to move sheet metal at known and controlled speeds. This will allow EMATs to be used to measure velocities on moving sheet metal at speeds approaching those found in a continuous annealing line in a steel mill. Several design concepts for the moving sheet device have been explored and a final design is currently under construction. When delivered (estimated date August 1990), the device will give NIST a unique measurement facility. Various combinations of EMATs will be tested, with different clearances between EMATs and sheet. Measurements will be made at a variety of speeds to determine what measurement artifacts were due to sheet motion. This test program is anticipated to commence in FY 1991.

Reference

"Ultrasonic Measurement of Sheet Metal Texture and Formability: Comparison with Neutron Diffraction and Mechanical Measurements," submitted to Research in Nondestructive Evaluation.

Barkhausen NDE

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Magnetic methods of nondestructive testing are heavily used in industry, primarily on ferritic steels. They are used both to detect defects and to characterize the metallurgical and stress state of materials. All currently used magnetic methods of flaw detection, including the magnetic particle method, rely in some way on the detection and measurement of leakage fields. Characterization methods presently in use rely primarily on the measurement of bulk hysteretic properties or related phenomena such as Barkhausen noise. Reliable use of these methods requires standardization and a fundamental knowledge of the relationship between microstructure and magnetic properties.

There are several methods for the characterization of Barkhausen noise. These methods may be based on, for example, the power density spectrum, the total noise power, the total number of Barkhausen jumps, or the first or second moments of the jump amplitude spectrum. We developed instrumentation which obtains a complete cycle of noise in digitized form. This information can then be analyzed by digital techniques to obtain all the important noise properties. Properties which have been used are: (1) the noise pulse amplitude distribution profile, (2) the power spectral density, (3) the pulse number spectrum, and (4) the total noise power. Having the digitized data allows these properties to be readily obtained as a function of applied field during a hysteresis loop. This digitized data was used to show the statistical independence of Barkhausen noise pulses arising from a variety of materials[1].

In addition to the traditional properties of the Barkhausen noise spectrum, we developed a method for characterizing the noise which we term the "jumpsum" method. In this method an algorithm is used which essentially performs a pulse height analysis of the jumps as a function of field. The height of these jumps are then summed as a function of applied field. Using this procedure, the noise is turned into a relatively smooth curve after a single hysteresis cycle. The use of a single cycle to collect the data can lead to significant time savings, which is important in a field or production environment, because the hysteresis cycle times used are usually very low (less than 1 Hz).

The use of the jumpsum method is illustrated in Figures 1 and 2. Figure 1 shows the jumpsum versus applied field for samples of ASTM A710 precipitation hardening steel given different heat treatments. In each case the steel was austenized for 90 minutes at 1650 F, quenched, and then aged at various temperatures for 90 minutes. The aging condition is readily distinguished. For example, the Barkhausen jumpsum response is quite different for a sample given no aging treatment (as quenched) and a sample overaged at 1100 F, even though these two samples have almost the same hardness. The jumpsum response of SRM 493 (a NIST standard reference material containing spherodized cementite in ferrite), taken under the same experimental conditions (i.e. the same detector, amplifier bandpass, field sweep frequency, field sweep amplitude, field sweep waveform, noise discriminator, etc.) as the A710 steel, is also shown in Figure 1.

Figure 2 shows the jumpsum obtained from a case hardened sample of 1050 steel. This material was taken from a production run of automobile axles at a General Motors plant in Saginaw, Michigan. The case of the axle was hardened to approximately HRC 50. Note the large difference in jumpsum response between the unhardened core and the hardened case. Again, results are compared with the response of SRM 493 taken under the same experimental conditions.

Figures 1 and 2 illustrate the value of a reference material in Barkhausen measurements. The jumpsum response is similar to most other properties of the Barkhausen noise in that its magnitude depends on the details of the equipment being used (some of the variables have been noted above). However, the relationship between the response of a piece under test and the reference will remain constant. In addition, the reference provides a check for the reproducible output of the equipment. A total of four representative samples of SRM 493 were tested for their Barkhausen noise properties. Some of these properties are quite sensitive to the experimental conditions under which the noise was obtained. However, when tested under the same conditions, the measured characteristics have only small variations. The data are being analyzed to assign numbers to the expected variations.

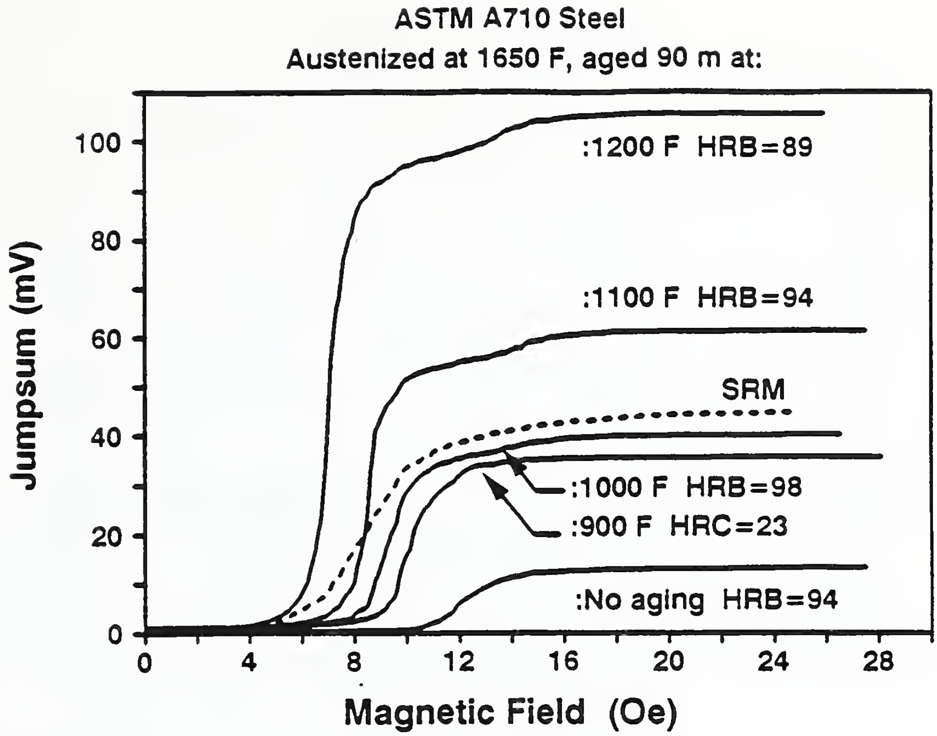

Figure 1. Barkhausen jumpsum vs. applied magnetic field for a sample of A710 precipitation hardening steel given an austenizing treatment followed by an aging treatment of various temperatures. The dotted curve is the response of SRM 493 taken under exactly the same conditions.

Cooperative work on standards with ASTM committee E07 and committee K of the SAE has continued during the current year. The magnetic particle inspection standard E709 is being revised and updated, based in part on work performed on this and other[2] projects. A preliminary new ring standard for use in magnetic particle inspection was designed and fabricated from a special run of 52100 steel provided by Lucas Aerospace, Maple Heights, Ohio. Two of the rings were produced. The magnetic leakage fields from the artificial hole pattern in these rings was measured. These rings were then sent for testing to the Nondestructive Test Lab of LTV, Dallas Texas. Based on our measurements and the results from LTV, a new hole pattern will be designed. A new magnetic particle inspection method, in which we are playing a leading role, is being developed based on the use of shim standards to determine the adequacy of the applied field. The purpose of the new standard is to simplify the use of the magnetic particle method by removing the considerable confusion arising from attempting to apply formulas designed for simple geometries to the many complex geometries currently being tested by this method.

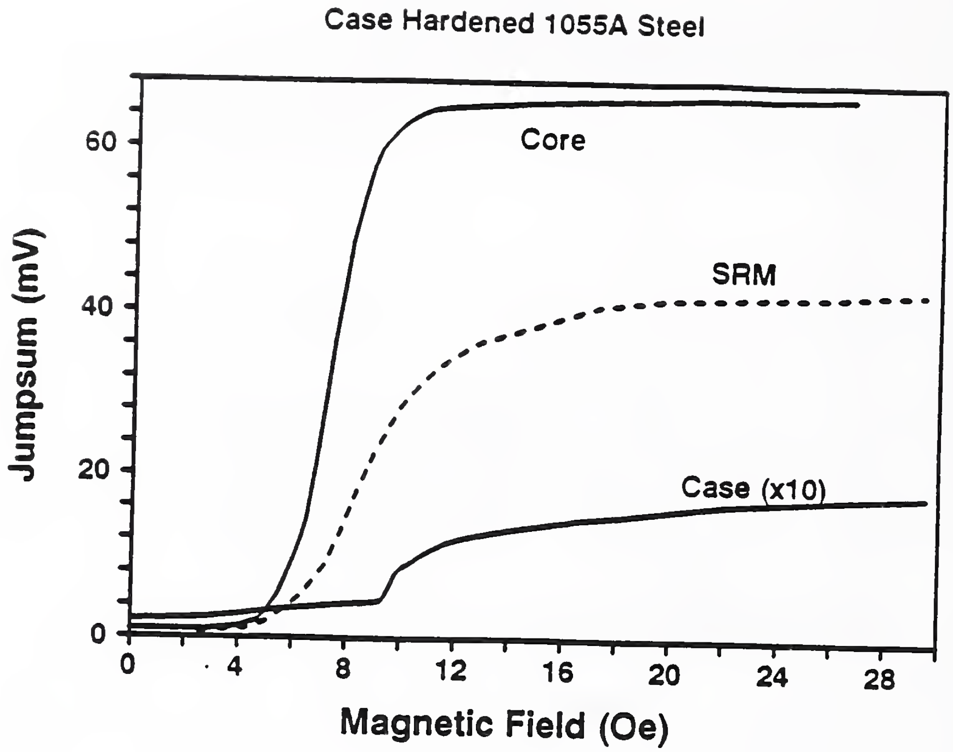

Figure 2. Barkhausen jumpsum from a case hardened rod of 1050 steel. The curve labeled 'core' is from the unhardened center. The curve from the hardened case has had its amplitude multiplied by a factor of 10. The dotted line was obtained from SRM 493 under the same conditions.

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C. NDE FOR COMPOSITES PROCESSING AND INTERFACES

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

This activity includes research on utilizing fluorescence spectroscopy to monitor the processing of polymer matrix composites, developing eddy current sensing techniques to monitor the high temperature processing of carbon-carbon composites, applying ultrasonic techniques to improve the ability to monitor interfaces and understand their performance, intelligent processing of electronic solder connections (which involves characterizing and understanding several metal-metal interfaces), and developing ultrasonic sensors for characterizing liquid-solid metal interfaces.

- A major barrier to the implementation of advanced polymer composite materials in many applications is that the processing lacks the desired reliability. To improve the reliability, NDE measurement techniques need to be developed to improve process monitoring and control. Previous NIST research showed that fluorescence spectroscopy is feasible for polymer process monitoring of non-Newtonian shear viscosity, molecular orientation, velocity, and flow characteristics. These parameters play a key role in determining the properties of composites. During the past year significant progress has been attained in a number of areas in this effort including the measurement of shear stress, non-Newtonian viscosity, quality-of-mix of ingredients, and molecular orientation.

- Initial results have confirmed the feasibility of utilizing on-line eddy current conductivity sensors to monitor the high temperature baking and pyrolyzes of carbon-carbon composites. The electrical conductivity data obtained by the eddy current sensor during carbon-carbon processing will be used to adjust the time-temperature cycles used for processing and help increase the yield of high quality products. This research is being carried out jointly by the General Dynamic Corporation and NIST researchers in the Metallurgy Division.

- The need to greatly reduce size in electronic assemblies has substantially increased the difficulty of achieving reliability in solder joint connections. Last year, the Office of Nondestructive Evaluation established a NIST-wide project to develop intelligent processing which is being funded by the U.S. Army Harry Diamond Laboratories. Non-intrusive sensors for the soldering process have been evaluated and calibration techniques developed. Computer programs for analyzing and evaluating the sensor signal are being formulated, and a data base on the physical properties of solder joint components is being generated. In addition, fundamental research activities are underway to develop a better understanding of the solder process in order to formulate a meaningful process model. NIST researchers from the Metallurgy Division, the Materials Reliability Division, and the Center for Manufacturing Engineering are involved in this major project.

Measurements for Control of Polymer Processing Parameters Using Fluorescence Spectroscopy

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In this program, fluorescence spectroscopy is being employed as a tool to monitor polymer processing parameters which are important for understanding process behavior. The measurements involve 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. We have concentrated on developing concepts and methods to measure molecular orientation, shear stress, shear rate, non-Newtonian viscosity, velocity, flow instabilities, quality-of-mix of ingredients, residence time distribution, intersegmental mixing, and the onset of the glass transition. Work on quality-of-mix and residence time distribution, carried out in collaboration with the Naval Surface Warfare Center (NSWC), was completed in FY90. In collaboration with Drexel University, a fluorescent probe is being used to monitor the onset of the glass transition in polystyrene during injection molding processing. Significant progress has been made at NIST in the measurement of shear stress, non-Newtonian viscosity, and molecular orientation.

Residence Time Distribution, Quality-of-Mix, and Flow Instabilities

During the past year, we achieved the stated goals of a collaborative project with NSWC to make on-line measurements of quality-of-mix and residence time distribution of twin screw processing of polybutadiene mixed with $CaCO_3$ particulate. For these experiments, an optical fiber probe was installed at the exit die of the extruder and monitored the fluorescence intensity from a dopant dye. By applying the results of previous work, (see ref. 1) we were able to interpret the fluorescence in terms of the concentration of $CaCO_3$ in the mix. Figure 1 shows fluorescence intensity versus time for time after a step function change has been made in the operating set points of the process. At time $t = 0$, the feed ratio of $CaCO_3$ to polybutadiene was changed from 63/37 to 60/40 by weight and the subsequent change in the product was monitored via the fluorescence. Note that the sensitivity of this measurement is approximately 1% in composition. In this case the smooth transition from one state to the other took approximately seven minutes. The flat regions at either end of the curve are typical of the response from a well mixed product which does not change more than 1% in composition. Having an on-line measurement of transition time and quality-of-mix is a valuable tool for maintaining the quality of the product. By continuously monitoring the output, it was possible to maintain constant surveillance on the product and on changes in the processing conditions.

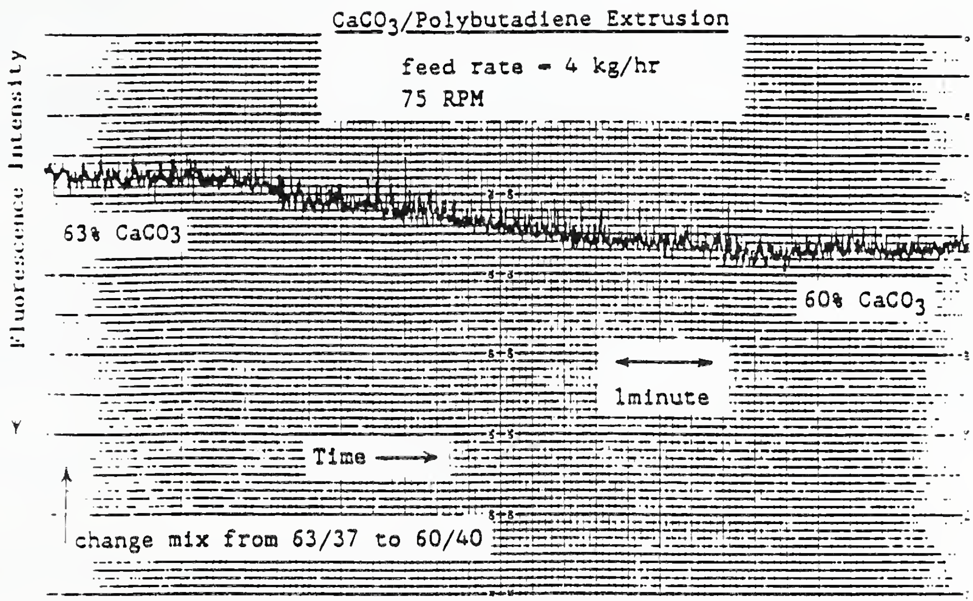

Figure 1. Fluorescence intensity at the exit of a twin screw extruder is plotted vs time for polybutadiene filled with $CaCO_3$ particulate. Here, we monitor the concentration of particulate after the feed rate of $CaCO_3$ is changed from 63% to 60% by weight. The monitoring technique is used to measure the transition time from one operating state to the another.

The fluorescence probe was also used to observe residence time distribution. In these experiments, a fluorescent dye was "instantaneously" injected into the extruder at a point which was 62 cm upstream from the exit die. The dispersion of the dye in the mixture was detected at the exit die of the extruder. The delta function distribution at the time of dye injection was transformed into a dispersion which extended over several minutes, indicating the extent of spatial mixing and residence time of the dye in the extruder. A series of residence time experiments carried out for different operating conditions demonstrated the applicability of this technique to monitor the transit time of the ingredients through the machine. This information is being used by NSWC to optimize productivity and product quality.

Shear Stress and Non-Newtonian Viscosity

The measurement of fluorescence anisotropy can be used to determine shear stress, molecular orientation, and non-Newtonian viscosity. The measurement involves the use of polarized light to determine the orientation of the fluorescent dye. Laboratory confirmation of this measurement concept and an extensive experimental study of anisotropy using a polymeric fluorescent dye have been carried out. The polymeric dye is polybutadiene tagged with anthracene which was synthesized in our laboratory. The dye was doped into a polybutadiene matrix and anisotropy was measured as a function of the applied shear stress using a optically instrumented rheometer. It was found that anisotropy decreased as a function of the applied shear stress when the fluorescent probe was doped into polybutadiene plasticized with (5%) cetane. For polybutadiene plasticized with 50% cetane, anisotropy was nearly independent of the applied shear stress. These data, shown in Figure 2, were interpreted in terms of a molecular model which depicts the polymeric probe participating in the orientation of the matrix polymer at 5% plasticization but not participating at 50% because the concentration of molecular entanglements has become too dilute compared to the size of the probe molecule. The model also yielded molecular orientation functions which showed that, for an applied shear stress, the orientation of the probe molecule in the 5% plasticized specimen is small and can be described as a slightly perturbed random orientation. This knowledge can be applied to polymer processing measurement technology by using these data along with shear rate and viscosity data to characterize the rheology of polymer materials undergoing process flow.

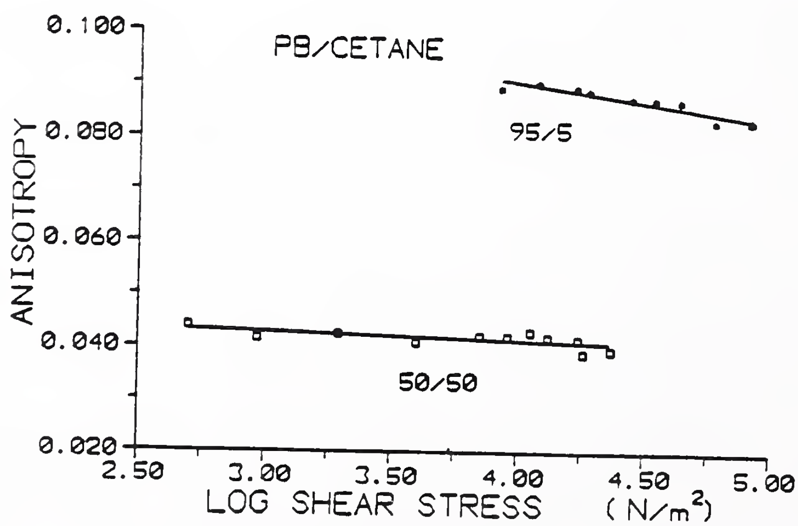

Figure 2. Anisotropy is plotted vs shear stress for the 5 and 50% cetane plasticized polybutadiene specimens, designated 95/5 and 50/50 respectively. Each specimen has been doped at 0.1% concentration by weight with the anthracene tagged probe molecule.

Fluorescence Monitoring of the Glass Transition

For this work, we are employing 1,3-bis-(pyrene)propane, a fluorescent probe molecule which has been used in the past to monitor the curing of epoxies. The fluorescence radiation from this probe occurs in three prominent bands which change in relative intensity in response to changes in viscosity of the probe neighborhood. Using this probe doped into polystyrene, the fluorescence intensity was monitored as the temperature was lowered from above the glass transition (105°C) to room temperature. At the onset of the glass transition temperature, fluorescence intensities display a distinct change in slope on the intensity vs temperature curve. This phenomenon will be used to monitor injection molding of polystyrene; by measuring fluorescence from the polymer as it resides in the mold, we will be able to control the time at which the mold is opened.

Reference

1. A. J. Bur, J. Shibata, T. K. Trout, F. W. Wang, and C. L. Thomas, Polymer Eng. Sci. 29, 1759 (1989).

Eddy Current Testing of Carbon-Carbon Composites

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The objective of this new effort is the evaluation of eddy current methods for monitoring the electrical conductivity of carbon-carbon composite materials during processing. Carbon-carbon composites are produced by baking and pyrolyzing a prepared structure of graphite fibers in a matrix of organic resins. Processing temperatures are as high as 800° C. Large changes in electrical conductivity occur in the material between the initial stages and the final product. Use of an on-line eddy current sensor will correlate conductivity changes with various stages of the pyrolysis and will prove useful as an element in a process control system. This project is being carried out jointly by the General Dynamics Corp., Fort Worth, Texas and NIST, with partial support by DARPA.

In the first half year of the project ending October 1, 1990, the objectives were to arrive at the design of an eddy current sensor to be placed in the processing reactor. A room temperature sensor model was constructed consisting of two flat spiral coils of outer diameter 4 inches, separated by a distance of 0.5 inch. Measurements were made on a sample of the composite before pyrolysis, a one foot square sheet of thickness 0.2 inch. The electronic equipment used was the same as for the monitoring of hot isostatic pressing[1]. A plot of the real part of the transfer impedance between the primary and secondary coils with a conductive sample in place shows a maximum at a frequency determined by the resistivity of the sample. The real part of the impedance represents the ohmic loss associated with the eddy currents induced in the sample. At low frequencies the loss increases with frequency since the induced emf is proportional to the rate of change of magnetic flux which penetrates the sample. At high frequencies the skin effect dominates, flux is excluded, and the loss decreases with increasing frequency. The peak loss, representing the transition between these limits, may be used to obtain a measurement of the resistivity of the test sample.

Figure 1 shows the experimental points and the theoretical curve, calculated by the methods of Dodd and Deeds[2], which we have applied to a thin conductive sheet. Variation of resistivity shifts the theoretical curve along the frequency axis. We adjusted the maximum of the theoretical curve to match the experimental data, which yielded a resistivity of 4000 microhm-cm for the sample before pyrolysis. A typical carbon-carbon composite, after pyrolysis, has a resistivity of 900 microhm-cm. These values are in a range well within observability with the existing electronic equipment.

The initial experimental results are encouraging. A high-temperature coil system is now being constructed and tests at reactor temperatures are being planned.

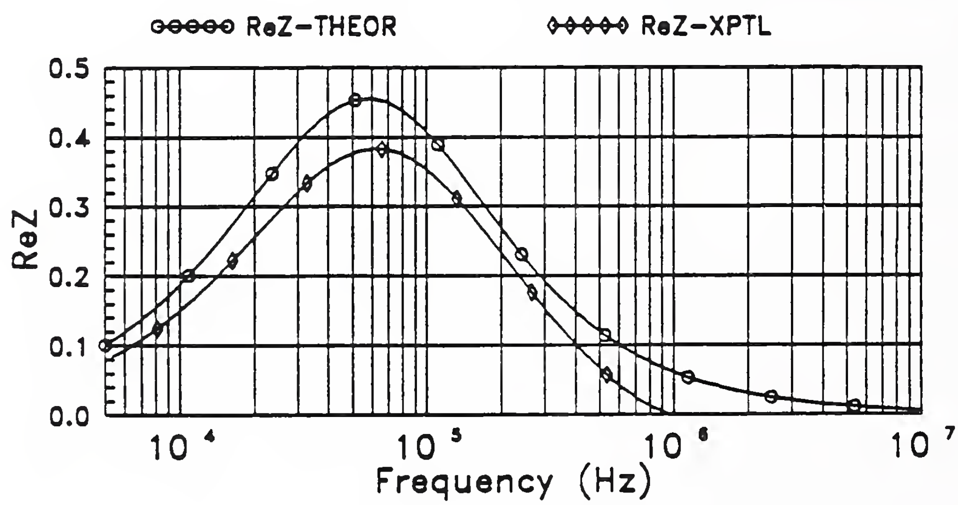

Figure 1. Real part of the two-coil transfer impedance with a carbon-carbon sample in place. The theoretical curve has been adjusted to match the measured absorption peak.

References

1. Eddy Current Techniques for Sample Dimension Measurements During Hot Isostatic Pressing, A. H. Kahn, M. L. Mester, and H. N. G. Wadley, Second International Conference on Hot Isostatic Pressing (1989); to be published.

2. Analytical Solutions to Eddy-Current Probe-Coil Problems, C. V. Dodd and

W. E. Deeds, J. Appl. Phys. 39, 2829 (1968).

Transient Elastic Waves in Laminates

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Pulsed ultrasonic testing techniques have been proposed for 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. Our approach has been a multi-faceted one. 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 usages.

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. Specifically, it was proposed to derive and design algorithms to compute the exact dynamic Green's function of a layer of uniform thickness on a half-space for various material properties and various interface (bonding) conditions.

In the past we developed and made available to the public a Fortran code to compute the dynamic Green's function of an infinite plate. 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 industry, universities and federal labs. The code also serves as a check of the present program being developed.

During the current year we successfully completed, carefully debugged, and verified the full code for the transient behavior of a layer with various interface conditions on a half space of different material. Varification was accomplished by comparison with the plate theory, published continuous wave formulations, and arrival time estimates.

Earlier the exact fundamental solution for this structure was explicitly derived from three dimensional equations of motion. The final solution was a series. Each term of the series corresponds to a successive arrival of a "generalized ray" and each is a definite line integral around a fixed path which can be easily computed numerically. Willis's method was used in the derivation. A new scheme of automatic generation of the arrivals and ray paths, and the summation of the corresponding products of reflection coefficients was developed. Now the FORTRAN code for computation of the Green's tensor when both the source and the detector are located on the top surface is complete and runs on a PC. Green's tensor is being used to simulate displacements due to pulsed sources of known time waveform.

Additionally, several other tasks were completed during the current year. We completed a report[1] detailing the derivation, algorithms, and code development including sample results for several mathematical sources (delta, step, damped sine) for a layer on a half space with various interface conditions. We also performed detailed runs of the exact solution to begin investigating the production and evaluation of boundary leaky waves. In addition, we designed the experiments for providing transient waveforms with which to compare the exact theory. And finally, we developed the algorithms for a multi-layer structure based upon J. Willis' method.

Figure 1 in last year's annual report is somewhat in error. (These were preliminary results prior to additional error checks and debugging.) However, the conclusions remain that early wave arrivals depend on and reflect the condition of the bond at the interface and can be used to deduce the conditions of the bond; some of the first few head waves and regular reflected rays change their polarities and amplitudes depending on interface condition.

From a preliminary study of optimal source locations, it appears that sub surface sources near or on the interface show a lot of promise. This at first seems not amenable to practice but in fact suggests sources on the edge of real (finite) parts or the use of a source focused in such a way as to produce a virtual source at the interface.

We next plan to use this exact solution to develop methods for optimally injecting the best boundary probing waves which can be received by a surface receiver, and determining what type (e.g., out of plane or tangential) reciever is optimum. Composites will be given particular emphasis. An experiment using a plexiglass plate on an aluminum block will be conducted. We will also try to resolve the seeming failure of reciprocity for an elastic layer on a half space.

Reference

1. S. Ren, N. Hsu, and D. Eitzen, "Transient Green's Tensor for a Layered Solid Half-Space with Different Interface Conditions," to be submitted to J. Acoust. Soc. Am.

Intelligent Processing of Solder Joint Connections for Printed Wiring Assemblies

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U.S. Army Laboratory Command

The need to reduce size and weight in military hardware, and specifically in electronic assemblies, has greatly increased the difficulty of achieving reliability in solder connections. Individual solder connections in state-of-the-art weapon systems are only fractions of a millimeter in size, and the current dependence on surface-mount technology for printed wiring boards has eliminated the support that had previously been afforded by through-hole designs. Nevertheless, these connections are required to function dependably under temperature extremes, mechanical shock and vibration, corrosive environments, and other adverse conditions associated with military operations.

In a multi-year contract from the U.S. Army Harry Diamond Laboratories, the Office of Nondestructive Evaluation established a NIST-wide project to develop intelligent processing technology for electronic solder connections in order to increase their reliability. Intelligent processing of materials is an approach which utilizes non-intrusive sensors to monitor critical characteristics of materials while they are is being processed. The sensor signals feed into a computer that maintains continuous control of the process parameters such that the product always satisfies its requirements. The control decisions are made on the basis of process models, expert systems, and databases.

The NIST project, which represents only part of a larger DoD effort on the subject, addresses many of the important features of the problem. Non-intrusive sensors for the soldering process are being evaluated, computer programs for analyzing and evaluating the sensor signals are being formulated, and a database is being generated. Despite the fact that soldering is a centuries-old technology, it is only poorly understood and, more importantly, it is clouded by accumulations of false concepts and gross misinformation. Thus, fundamental research activities are being carried out to develop a better understanding of the soldering process, in order that a meaningful process model can eventually be formulated.

Three commercial sensor systems are being evaluated, and calibration methodology is being developed. The first is an instrument for inspecting solder connections by heating them with short laser pulses and then monitoring the thermal radiation from their surfaces as they cool down immediately thereafter. These thermal signatures are sensitive to critical characteristics of the solder connections. During the past year this system was studied by T. R. Lettieri of the Precision Engineering Division, with assistance from H. Frederikse, a consultant. The study centered principally upon the effects of laser noise on the subsequent thermal signatures. A heat-flow model was developed which was used to show that high-frequency noise with amplitudes up to 20 percent of the total laser energy has only a negligible effect on the peak value of the thermal signature. However, low-frequency noise can create sizable nonreproducibilities in solder-joint measurements when its period is much longer than the laser pulse. Based upon a suggestion by the NIST researchers, the manufacturer of the inspection system added a stabilization circuit to the instrument, which appears to have eliminated the noise problem.

Lettieri formulated a work plan which is being used by the Naval Weapons Support Center to evaluate the thermal inspection system. The plan calls for testing the system using a combination of soldering pastes, test coupons, and paste bake times. Laser parameters to be varied include pulse length, laser power, and spot size. NIST personnel will participate in the analysis of the data.

The second sensor system is a laser-based machine vision instrument that scans circuit boards according to a pre-programmed raster pattern, using a light scattering technique to characterize the shape of each solder connection.

T. Doiron of the Precision Engineering Division is testing the basic metrology of the instrument. A test object consisting of an aluminum plate with measurement artifacts mounted near each of the four corners was developed. Each artifact has three pseudo-solder joints, also of aluminum, which serve as the test structures for the system. A test program, to be implemented by a DoD contractor using the test object, is intended to evaluate the positioning accuracy of the instrument and its solder joint parameter measurement characteristics. The final phase of the task will be to correlate the test data and arrive at the accuracy level of the instrument.

The third sensor system under consideration is based upon x-ray laminography. This is a radiologic imaging technique that can capture two-dimensional slices through a three-dimensional solder connection. Its advantage over a conventional radiographic system is its ability to image individual planes, say, under a surface-mounted device or on one side of a multilayer board. T. A. Siewert and M. W. Austin of the Materials Reliability Division are assessing the advantages and disadvantages of the system in a quantitative manner based on fundamental scientific parameters. The first task was to procure a laminography system, which was first installed in a DoD contractor's plant for a production evaluation on complex circuit boards. It will be delivered to NIST in January 1991 for measurement of its resolution and response. Meanwhile, the NIST researchers assembled a test object consisting of a circuit board populated with various image quality indicators. Preliminary tests have confirmed the ability of the object to yield meaningful data on the inspection system's resolution and positioning accuracy. Siewert and Austin are presently developing a procedure to evaluate the system's reproducibility in terms of line-pair resolution.

D. T. Read of the Materials Reliability Division is developing computer algorithms which will enable the mechanical integrity of solder connections to be evaluated from the sensory information in near real-time as they are being inspected. The stresses that arise in surface-mount solder connections due to differential thermal expansion, circuit board flexure, and accelerations can be significant, and can produce failures if the connections are not properly formed. The evaluation will be based upon a comparison of the actual mechanical stresses, which are anticipated in service, with the strengths and fracture toughnesses of the materials that comprise the solder connections. Present efforts are concentrating on the development of an automated procedure for modeling individual solder connections by generating finite element meshes from the sensory data provided by the machine vision and the x-ray laminography inspection systems.

A considerable portion of the work on this project is directed toward the establishment of a database of the mechanical and physical properties of the various materials involved in solder connections. The determination of these properties required, first of all, careful identification of the materials, and then their synthesis in forms that would enable their properties to be measured. When solder connections are made, thin layers or dispersions of brittle intermetallic compounds are nucleated near the interfaces between the solder and the base metal. In the case of conventional tin-lead solder used on copper leads, the principal intermetallics formed are Cu_6Sn_5 and Cu_3Sn.

If the leads are nickel plated, Ni_3Sn_4 is also formed. These intermetallic regions grow in size even at room temperature and strongly affect the performance of the solder connections. However, many properties of these intermetallic compounds had never been measured prior to this project because bulk samples of the materials were not available for test.

During the past year, scientists in the Metallurgy Division's Metallurgical Processing Group (F. S. Biancaniello, W. J. Boettinger and R. J. Schaefer) prepared fine-grained single-phase bulk samples of these compounds for the first time anywhere. The process involved the use of advanced rapid solidification techniques to produce powders of the intermetallics, and then hot isostatic pressing to convert the powders to bulk samples. This important development has resulted in numerous requests from other laboratories for samples on which to make measurements.

R. J. Fields, S. R. Low, III, and D. E. Harne of the Metallurgy Division measured the mechanical properties of high-purity 63 Sn - 37 Pb solder as well as the intermetallic compounds. The tensile properties of the solder material were evaluated at room temperature over a wide range of displacement rates. The results showed Young's modulus to be relatively constant while the strength values increased significantly with displacement rate. Elongation showed a strong inverse relationship to displacement rate.

Torsion tests of the solder were also carried out. Comparison of the yield strengths in tension with those in pure shear, for equivalent strain rates, suggests that the yield strength of solder under multiaxial stress conditions is determined more closely by the octahedral shear stress (the von Mises criterion) than the maximum shear stress (the Tresca criterion).

In related work by R. B. Clough and R. H. Patel of the Metallurgy Division, a configuration for a lap joint specimen was developed in which the solder is stressed in shear with relatively little bending. The interface was modeled using an elastoplastic crack solution. This specimen configuration can be tested in a conventional tensile testing machine. At the suggestion of the project sponsor, a composite solder was prepared by introducing a fine dispersion of Cu_6Sn_5 powder, approximately 15 percent by volume, into a melt of eutectic lead/tin solder. Copper lap joint specimens made with this composite solder were fabricated and tested for comparison with the strengths of joints made with conventional liquid solder. The results have been encouraging and appear to represent a novel approach to improving the performance of solder connections.

To measure the hardness and the fracture toughness of the intermetallic compounds at various temperatures, R.J. Fields and co-workers designed and constructed a unique device. It consists of a diamond Vickers hardness indenter mounted on a screw-driven crosshead capable of a wide range of displacement rates. The lower anvil, on which the disk-shaped specimen rests, is rotatable so that a series of indentations can be made over the surface of the specimen without moving it relative to the anvil. The indenter and the anvil are enclosed in a chamber that can be heated or cooled to test temperatures of interest.

This device was used to study the three intermetallic compounds over a temperature range extending from -55 to +125 C and above. Under some conditions of load and temperature, the indentations produced cracks which were analyzed to determine the fracture toughness. The test results show that these compounds are harder than high-strength steel at room temperature, but only twice as tough as glass. Raising the temperature produces some softening but very little toughening.

An ultrasonic approach is being used to evaluate the elastic properties of the intermetallic compounds at various temperatures. This approach is based upon the well known relationship between Young's modulus, density, and the speed of sound. Measurements on Cu_6Sn_5, carried out by E. Drescher-Krasicka in the Metallurgy Division, have thus far shown a linear dependence of sound speed on temperature from about -67 to +80° C. Changes in the slope of the velocity/ temperature curve above and below this range suggest the possibility of phase changes at those temperatures.

Physical property data for the intermetallic compounds are also being determined. Density measurements made it possible to infer information about the filling of interstitial sites in the crystal lattices. Thermal diffusivity measurements were made with a thermal wave technique, and thermal expansion measurements were carried out over the range -55 to 200 C using a standard dilatometric method. The results show that the expansion behavior of Cu_6Sn_5 is almost identical to that of pure copper, so that stresses are unlikely to develop between these two materials due to temperature changes. The thermal expansion of Ni_3Sn_4, on the other hand, is slightly higher than that of pure nickel and slightly lower than that of pure copper, so that low values of thermal stress could be developed in solder connections to nickel-plated copper.

A significantly greater portion of the work on this project in the coming year will be devoted to studies to elucidate certain aspects of the soldering process that must be understood in order to permit process models to be formulated. W. J. Boettinger and C. A. Handwerker have been planning this effort and, one of the important phenomena that will be investigated is solderability. Research will be carried out to clarify the factors that influence solderability and, if possible, to identify some characteristic of this quantity which could be measured on-line in a soldering workstation. Interactions with industry are currently underway to establish an industry/NIST/HDL consortium on solderability. Another research task to be undertaken by E. Drescher-Krasicka will use an acoustic microscope to study the growth of the intermetallic compounds in solder joints and the degree of adhesion between the solder and the base metal.

Ultrasonic Sensing of the Liquid-Solid Interface in Metals

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This project focuses on the development of ultrasonic techniques for monitoring the liquid/solid interface position and structure in metals and alloys. The goal is to address a need in the metals processing industries for improved process sensing and modeling of directional solidification. In the continuous casting of steel, for example, the position of the liquid/solid interface determines the maximum viable rate at which the process may proceed. At the same time, the interface microstructure, which is a function of temperature gradients and solidification rate, determines the final mechanical properties. Therefore, without information on the liquid/solid interface, either through direct on-line sensing or indirect experimentally verified models, the process cannot be optimized and controlled in terms of either economics or product quality.

In order to use ultrasonics to obtain real-time information on interface microstructure, sound waves must be reflected off the interface from the liquid side. If the waves were to originate in the solid, grain scattering effects would obscure the details of scattering from the interface. Although a liquid is a simple medium in which to perform scattering measurements, the construction of an ultrasonic sensor which can be immersed in liquid metal is a formidable technical challenge. Two basic sensor designs are being explored. One design uses refractory fiber acoustic waveguides surrounded by gas within a tube which isolates the fibers from the liquid metal except at the ends of the fibers. This configuration provides complete internal reflection of ultrasonic waves travelling along the fibers and, hence, produces relatively long useful propagation distances. A second design uses lasers to generate and detect the ultrasonic wave. A transparent rod would be immersed in the liquid metal and the wave generated and detected at the surface of the liquid which presses against the end of the rod. Initial tests of this sensor configuration are being planned in collaboration with Dr. James Wagner at Johns Hopkins University.

Dendritic liquid/solid interfaces display a certain degree of periodicity which is usually described in terms of primary arm spacing (the average distance between the "trunks" which are roughly normal to the overall interface plane) and secondary arm spacing (the distance between the smaller, more closely spaced, branches off the primary arms). In order to determine the effect of dendritic structure on ultrasonic backscatter, a series of modeling studies have been initiated. An array of polystyrene wafers was constructed, immersed in a water bath, and ultrasonic waves were reflected off the structure at normal incidence. The amplitude of the reflected pulse was measured while the frequency was scanned under computer control. This experiment was designed to provide insight into basic secondary arm scattering effects. Figure 1 (solid line) shows a scan from 0.3 Mhz to 1.9 Mhz with 10 wafers having a periodicity of 0.65 mm. and a thickness of 0.33 mm. The amplitude has been normalized with respect to the sensitivity of the transducer. Although the broad plateau in the scan is fairly reproducible, the smaller peaks vary with slight repositioning of the transducer, probably because of edge effects (the array of wafers was smaller than the width of the ultrasonic beam at the higher frequencies). The dashed line in Figure 1 shows a theoretical solution derived under the assumption that the sound is a normally-incident planewave. In this theoretical model, the media and fields depend only on depth, and the propagation obeys the one-dimensional wave equation. The exact solution, which fully accounts for multiple scattering, is derived by matching boundary conditions at each interface. The problem is then formulated in matrix propagator form, in which the back-scattered or forward-scattered ultrasonic wave is numerically propagated from one layer to the next by a series of two-by-two matrix multiplications, one multiplication per layer. The method allows any combination of layer impedances and thicknesses. A second modeling study has explored the effect of primary dendrite arm spacing with the wavefront nonparallel to the interface. A flat periodic array of parallel wires was constructed and immersed in water. A single transducer was positioned such that the wave-vector was perpendicular to the individual wires, but at an angle, θ, to the normal of the plane of the array. With this geometry, constructive interference of reflections from the wires is expected at frequencies, f, such that

$$f = nv/2d\sin\theta ,$$

where v is the velocity in water, d is the wire spacing, and n is a positive integer. Figure 2 shows frequency scans at incident angles of 60° and 75° with an array of 0.20 mm. diameter copper wires spaced 1.27 mm. apart. The frequency response is relatively simple, with well defined peaks at the expected frequencies. Figure 3 shows the calculated and measured peak positions with θ ranging from 35° and 85°.

These initial modeling studies suggest that efforts with dendritic interfaces should focus on obtaining information on primary arm spacing. Secondary arm effects are probably more complicated and occur at higher, less readily accessible, frequencies. Work in the immediate future will explore model arrays with periodicity in two dimensions and varying degrees of randomness in the spacing.

One persistent obstacle in evaluating the feasibility of any ultrasonic technique for probing liquid/solid interfaces in alloys is the lack of information relating to the intrinsic attenuation in mixed phase regions. It is generally assumed that attenuation is quite large in alloys undergoing solidification, but no reliable information is available on the subject because of technical difficulties involved in performing ultrasonic measurements on a solidifying sample. A unique electromagnetic acoustic transduction (EMAT) technique has been designed to address this need for fundamental materials data. The technique uses a small coil between the poles of two permanent magnets inside a vacuum chamber. A small (1-2 mm. diameter) spherical sample rests on a non-wettable platform inside the coil. Eddy currents are generated in the sphere by passing a high-frequency pulsed current through the coil. Lorentz forces on the charge carriers in the sample then generate ultrasonic standing waves. After shutting off the driving pulse, detection of the decaying oscillations is accomplished with the same coil by the reverse mechanism. Measurements can be performed continuously during melting because surface tension keeps the sample almost spherical. A unique signal analysis

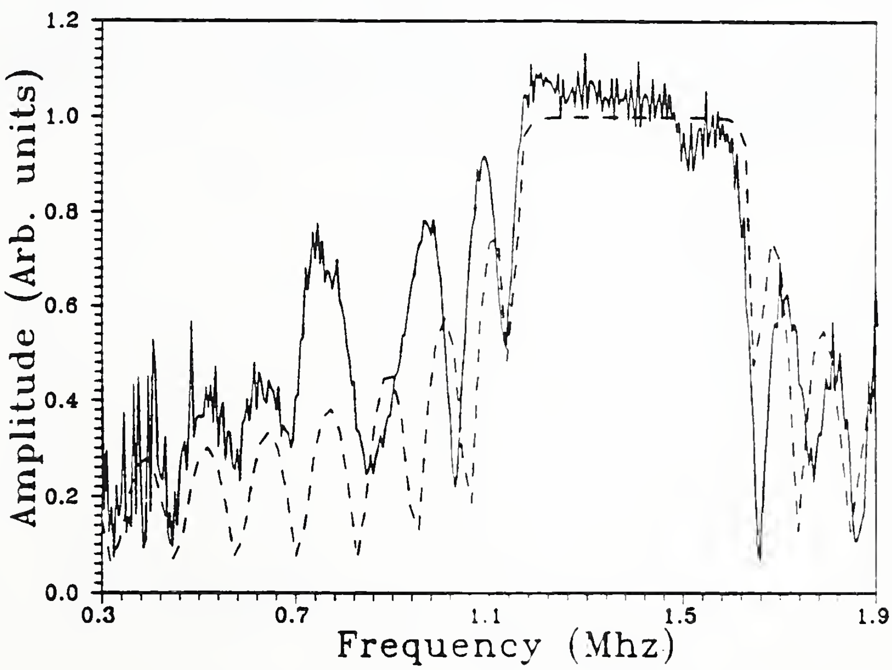

Figure 1. Amplitude versus frequency of ultrasonic waves reflected off a polystyrene/water one-dimensional periodic structure. The polystyrene wafers are 0.33 mm. thick and spaced 0.65 mm. apart (with water between them). The ultrasonic waves (generated by a 1 Mhz broadband transducer) are at normal incidence. The amplitude has been normalized with respect to the transducer sensitivity.

technique has been developed which allows the attenuation and frequency of a resonant peak to be simultaneously measured under computer control with a response time of about 2 seconds. Initial development of this technique has employed 1/16" diameter solid steel balls as samples. The resonant frequency spectrum shows sharp peaks which agree exactly with those obtained from numerically solving the wavequations. Future studies in this area will focus on performing measurements on low-melting-point metals and alloys undergoing solidification.

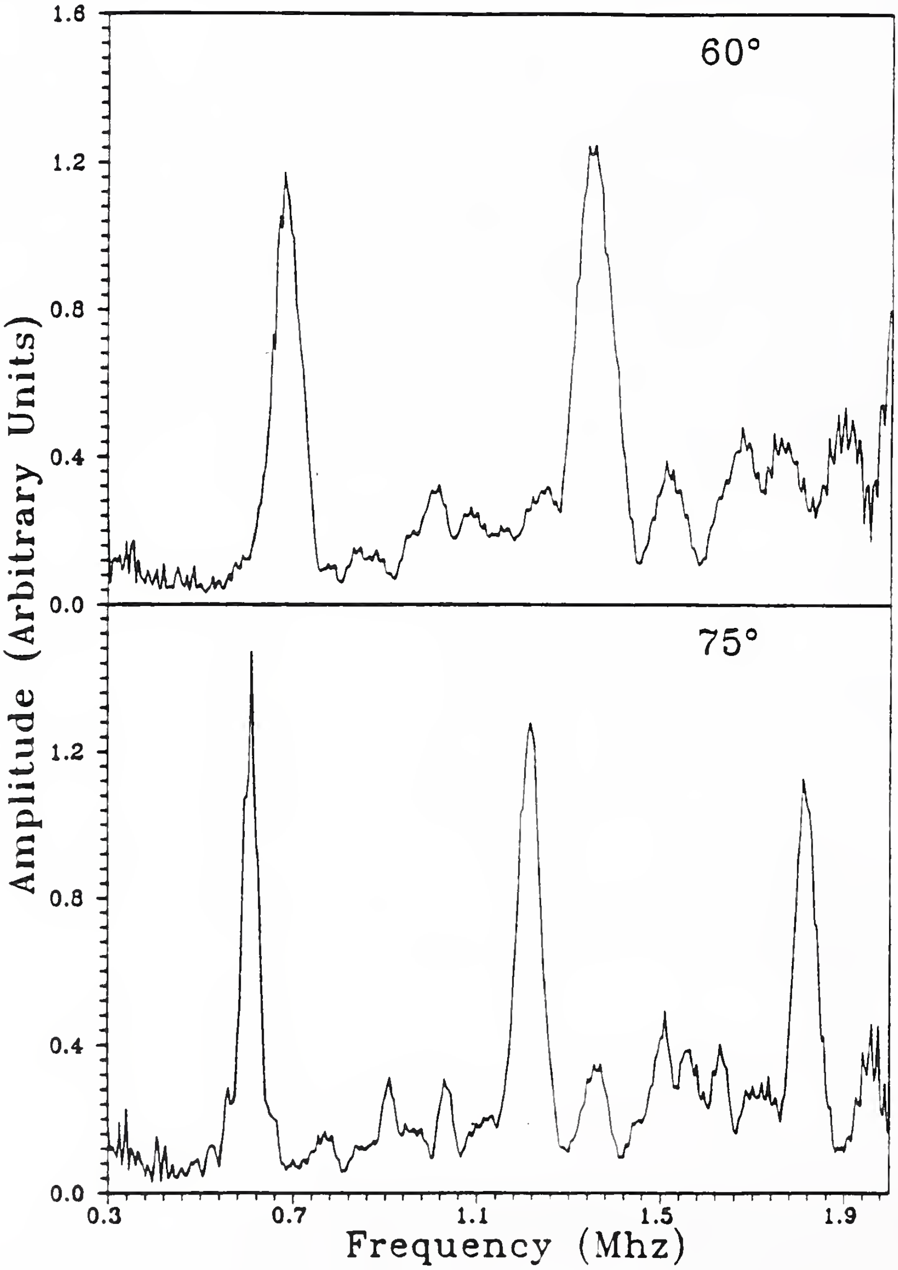

Figure 2. Amplitude versus frequency of ultrasonic waves reflected off a flat array of parallel 0.20 mm. diameter copper wires in water, with the ultrasonic wave-vector at an angle of 60° and 75° with respect to the normal of the plane of wires.

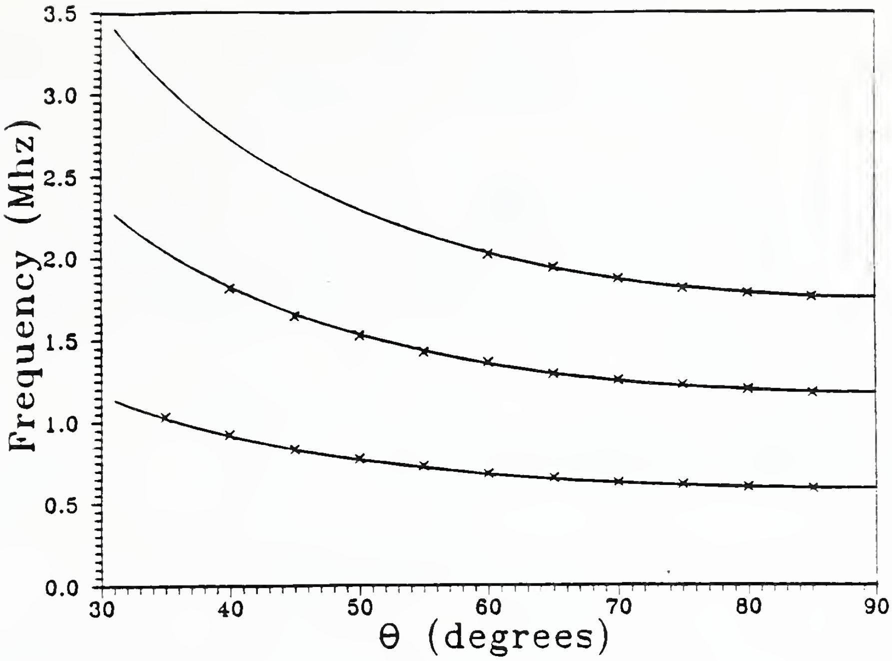

Figure 3. Measured (X) and theoretical (solid line) resonant peak positions as a function of the angle between the wave-vector and the normal of the array of wires.

D. NDE STANDARDS AND METHODS

The objective of the Standards and Methods activity is to provide the scientific understanding of NDE measurement methods and to develop, maintain, and disseminate effective standards for NDE measurements that are traceable to national standards. Four project reports are incorporated in this activity dealing with ultrasonics and acoustic emission (AE), real-time x-ray radiology, eddy currents, and thermography. It is fair to say that the NDE Standards and Methods activity is a cornerstone of the entire NDE Program, as it continues to serve as the basis for intensive standardization, calibration and consultation services provided to the nation's NDE community.

Ultrasonics and Acoustic Emission

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

The object of this project is to develop and disseminate artifact and documentary standards, develop and maintain measurement and calibration services, and develop new or improved methods for acoustic emission (AE) and ultrasonic NDE techniques. The writing of documentary standards is preceded by extensive and careful measurements and analysis to form an adequate technical base and, frequently, by the design and piloting of round robins. The standards are typically guided through ASTM and then promoted as international standards.

Secondary Calibration of AE Transducers

Currently, secondary AE calibration techniques which are imperical and not quantitative or accurate are being used by industry to calibrate transducers for tests and safety recertification of structures such as industrial gas tube transport trailers. These methods are codified, but ASTM has agreed to replace them as soon as a practical (and rational) system and technique has been developed and documented.

Two secondary (transfer) calibration apparati were tested last year. The first used a steel transfer plate 90 cm square by 3.3 cm thick. The results did not compare accurately enough to the primary calibration results for the same commercial AE test transducers even though good agreement was obtained when comparing the results for conical transducers. The problem arises because the front face aperture of commercial transducers is comparable to the thickness of the plate resulting in significant variations in the surface displacement across the aperture.

The second calibration apparatus utilizes a steel transfer block 40 cm square by 20 cm thick. The large faces had been Blanchard ground for good transducer coupling. The transducer under test (TUT) and a reference transducer (in this case a NIST standard reference conical transducer, SRM 1856) are placed symmetrically on the same surface as the input source, a breaking glass capillary. The TUT and reference are subject to the same dynamic displacement. The voltage output of both transducers are captured and the sensitivity of the TUT at each frequency is calculated using the sensitivity of the reference standard and its corrections known from its calibration on the primary calibration system. As reported last year, the agreement between calibrations performed using this secondary calibration system and the primary system were quite good.

Comparisons of the secondary and primary calibration of additional types of commercial transducers have been conducted to be certain that the proposed secondary system gives reasonable results for all transducer types. And each exercising of the secondary system is also a check of the procedure of using the standard reference conical transducer. An example is shown in Figure 1. This commercial transducer has rather complex behavior but all major features are characterized acceptably by the secondary calibration as compared with the primary. The disagreement is largest for the lowest frequencies of interest, but this is to be expected from the smaller transfer block for transducers which have a long ring down time when subject to a transient waveform. Details of the apparatus and technique will be written up in the format of a draft ASTM standard early next year.

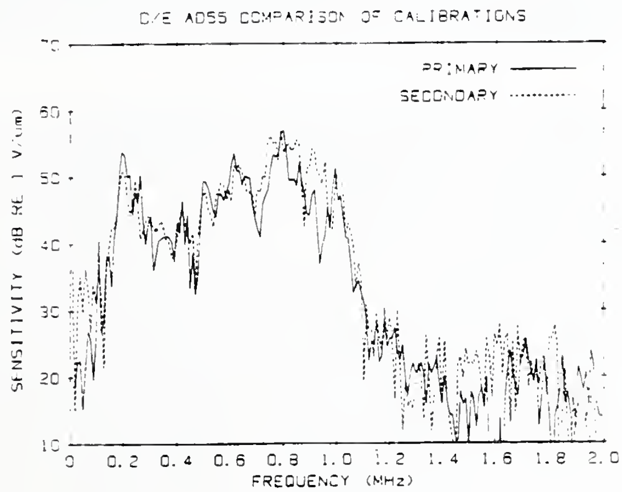

Figure 1. Comparison of a primary and a new secondary calibration performed on a commercial AE transducer with rather complex response.

While this new secondary system is workable and rational, it requires the use of the breaking glass capillary technique. This technique gives very good results but requires a bit of dexterity, skill, and practice. This observation motivates some of the work on sources described in the section on Transient Sources for AE. If a better source is developed it will be easily incorporated as a revision to the standards documents on secondary calibration of AE transducers.

Elastic Impact Experiments

The study of the impact of elastic spheres on an elastic plate has significance for a number of reasons: the validation of the use of structural transform techniques to obtain quantitative source information is essential to advanced AE methods; the development of a technique whereby the unknown force-time history due to a source can be accurately measured remotely is important for AE source development work and is a new tool for experimental mechanics; the ball impact source has applications in other NDE or monitoring of highly attenuating materials (concrete, composites) by the impact/echo method; and applications as a measurement method of particle size in particle processing. Initial results on the ball impact experiments were reported last year. In the experiments, steel balls of various sizes are dropped from 3 cm onto a glass plate. A NIST conical transducer, previously calibrated in place is located on the plate opposite the point of impact and is used to record the resultant displacement time waveform (which is much different than the input force time waveform). This signal is convolved with the transform function for this specific glass plate and with the transform for the small lack of fidelity of the transducer to yield a measurement of the "unknown" force time waveform produced by the impact source. The details are described in the manuscript[1] just completed.

The initial results of the experiments were promising but had a larger error than expected. For example, the half-amplitude contact time was in error by about 15% when compared to the approximate theoretical description of the force time function given by a half-sine function. The exact solution can only be numerically calculated, but when this is done it is seen that the half-sine approximation is in error in estimating the half-amplitude contact time by about 15%. The force of the impact is proportional to the square of the ball radius. The contact time is proportional to the radius. If the force-time histories determined by convolution are normalized, respectively, by the radius squared and by the radius they should agree with each other and with the exact numerically evaluated theory. The new and normalized force-time curves for spheres with ten different radii are shown in Figure 2. The theoretical waveform scaled down to account for air drag and less than perfect restitution is also plotted in Figure 2 but cannot be seen within the nesting. One waveform, that produced by a 3.17 mm sphere, is somewhat less than the others. Much closer inspection of this ball under high magnification showed it had an unusual amount of surface pitting. Using the waveforms to determine the maximum force results in an average error of about 3% as compared to theory and an average error in half amplitude contact time of less than 2% as compared with theory.

These results are quite encouraging and led to the development of an apparatus built specifically to study various sources (see Transient Sources for AE). Some work will be initiated to investigate the implications of this method for application to materials studies. If resources permit, the possibility of using the technique to monitor and control metal particle production by measuring sphere radius will be investigated.

Transient Sources for AE

There are a number of potential techniques for producing rapid transient disturbances in elastic media. These techniques for generating transient sources are necessary for characterizing AE systems components such as transducers and fielded systems, transfer media and other sources as well. The ideal source would create a point force, have a simple waveform such as a step or delta function, would have a relatively large repeatable amplitude which could be determined from a priori considerations, and would be easily repetitive.

An approach similar to that outlined in Elastic Impact Experiments was employed for this work. However, a carefully designed and constructed capacitance transducer was used because of its ultimate fidelity and ease of theoretical description. Also a highly polished aluminum plate was used because it serves as one plate of the capacitive transducer. Seven sources were studied with previously unavailable accuracy: Hsu pencil lead break, glass capillary break, capacitive source, conical transducer source, ball impact, spark source, and high explosive ($NI_3 \cdot 2NH_3$). Force waveforms were determined by convolution of the received transient with the system transfer function and spectra of these waveforms were determined. Details of the apparatus, data treatment, waveforms and comments on the relative merits of the sources are given elsewhere.[2]

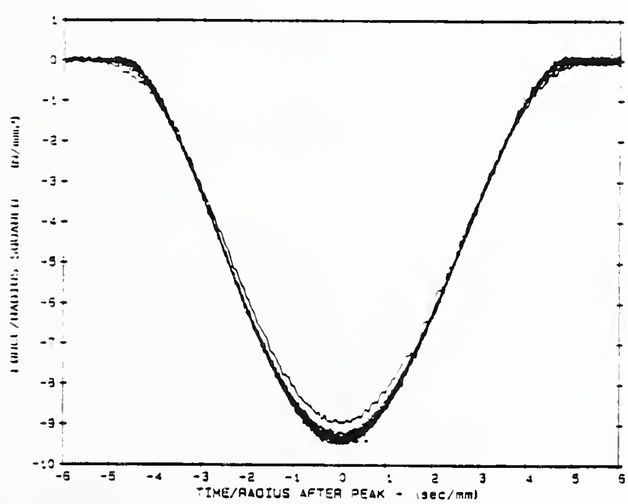

Figure 2. Force-time histories determined remotely for ball drops of 10 different sizes and theoretical result (not seen within the nested results for nine ball sizes). The one low force ball was discovered to have considerable surface pitting.

The spectra of the seven different sources are shown in Figure 3. The frequency characteristics of the sources is shown up to about 5 MHz, except for the pencil source. It is thought to have little energy above about 2 MHz; the spectra above that appeared to be noise generated. The amplitudes are all on the same log scale but this requires a range of 70 dB. The capacitive source has extremely good frequency characteristics, but it is distributed with a relatively low amplitude. The ball impact shows some content at 3-5 MHz, which is not yet understood but appears to be real because of its structure. The spark and explosive show good content except at low frequencies. Some additional source characterizations are anticipated in the future.

PVDF Characterization

We have begun development of a system to characterize PVDF transducers. This work was only possible because of recent improvements to the radiation force balance which now measures ultrasonic sources with unprecedented accuracy. We have acquired the necessary planar scanning equipment, checked it mechanically, and evaluated the digitizer. The software supplied with the system is, however, hopelessly flawed and some rewriting of software will be necessary.

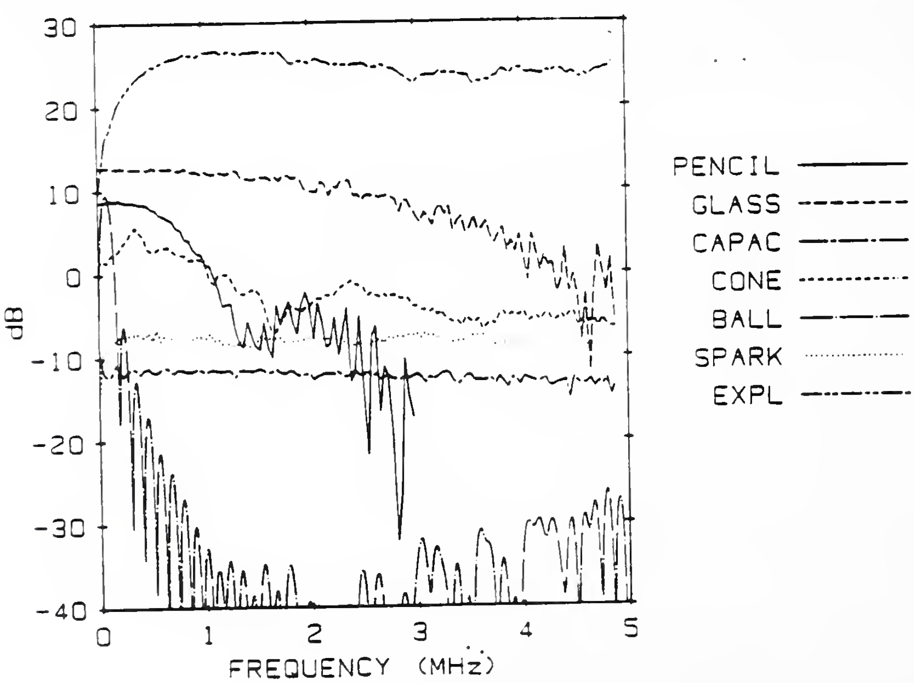

Figure 3. Spectra for seven transient sources determined by remote measurement of their force-time waveform and FFT.

Document Standards

Substantial work related to documentary standards was also accomplished. Two round robins (ultrasonic reference blocks and computer controlled evaluation of ultrasonic search units) were completed and already led to draft Annexes to two ASTM standards. The writing of one chapter for the ASNT handbook on ultrasonics is well along as is the complete rewrite of another chapter authored outside of NIST. Progress was also made on documents for measurement of bolt stress and for assessment of ultrasonic velocity measurement accuracy.

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Real-Time X-Ray Radioscopy

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Materials Science and Engineering Laboratory

Real-time radioscopy uses x-rays, like film-based radiography, but replaces the film with an imaging system. Acceptance of this efficient inspection technology has been limited by the lack of standards, both physical and procedural. This program is intended to overcome these limitations by developing new devices and test procedures to measure the special imaging capabilities of real-time x-ray radioscopy (such as evaluation of a part while in motion) and helping to include these devices and procedures in specifications and standards.

Last year, the results of a survey on the needs for standards in real-time x-ray radioscopy were reported.[1] This survey indicated that the major needs were for measuring the image quality and the performance of the entire system. This year, a device for evaluating the image produced by real-time systems was constructed. It consists of a 300 by 300 by 12 mm plate of steel, with various image quality indicators (IQIs) on its surface. The IQIs consist of the various penetrameters that would be used on this thickness material, together with line-pair gages and other research oriented devices. This plate was radiographed with our film based system for base line data, and the results will be presented to ASTM committee E07.01.02 (Real-time Radioscopy) for their review. We are also working on a task force of this ASTM committee to prepare a system qualification document. This device could be used in a round robin evaluation using the new document.

While conventional devices such as penetrameters should be suitable for measuring most critical image parameters in a static mode, they are not expected to be able to evaluate the image quality or system performance during translation or rotation, two unique capabilities of a real-time system. The line pair gages and a hollow sphere design are being evaluated for use with these capabilities. The line pair gage resolution is expected to degrade when translated normal to the line direction. The hollow sphere design produces the same image invariant to orientation, and it can detect translation in any direction by the blurring of its edges. A complete report on the evaluation of the spheres is being prepared for publication.[2] At this point, we are evaluating the use of the spheres to measure image contrast. Figure 1 summarizes the contrast measurements for a hollow sphere with a copper wall thickness of 120 micrometers (diameter of 12 mm) on a 6-mm-thick copper plate. In this figure, the gray level histograms on regions of interest (ROI's) on the radiographic image are compared. These regions are the center of the sphere image and an area outside the sphere. The separation of the means of the two distributions indicates that a 2 percent contrast resolution (measured with a densitometer) was easily attained. An attempt is now being made to automate the analysis and develop a mathematical technique to reduce this statistical distribution to a simple representation of the contrast.

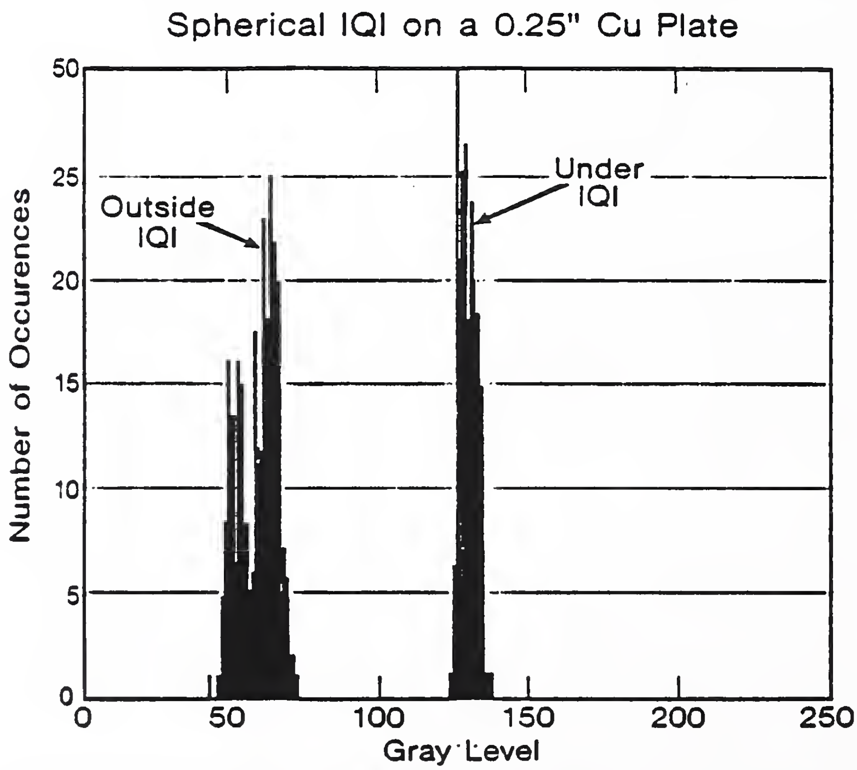

Figure 1. Pixel histogram for the regions inside and outside the image of a copper sphere (120 micrometer wall) on a 6-mm-thick copper plate.

NIST research into the application of real-time inspection to advanced materials, such as composites, has advanced to the stage where we are assembling a real-time system. The first application will be the low kV x-ray inspection of composites .

NIST has also been involved in numerous standards activities[3,4] including cooperation with the General Electric Company and the U.S. Army Materials Technology Laboratory in developing a military standard for radioscopic inspection, and leading an ASTM Task Group to modify the specifications for Radiographic Inspection of Electronic Devices to include real-time systems.

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2. T. A. Siewert, D. W. Fitting, and M. W. Austin, "A Rotationally Symmetric Image Quality Indicator for Real-Time Imaging Systems," to be submitted to Materials Evaluation, 1990.

3. T. A. Siewert, "Improved Standards for Real-Time Radioscopy," Nondestructive Evaluation: NDE Planning and Application, NDE-Volume 5, ed. R. D. Streit (contributing editors T. A. Siewert et al.), The American Society of Mechanical Engineers, New York, NY, July 1989.

4. T. A. Siewert, "Report on 1989 Actions by International Institute of Welding," Materials Evaluation, p. 408, March 1990.

Eddy Current Techniques

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

This program is 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. We are developing and testing empirical and computer based models of eddy current probe performance so that probe design and selection can be optimized in a predictable fashion[2].

Ferrite-core, Single-coil Probes

A study of how the physical construction of eddy current coils affects inspection sensitivity is being conducted. The study was approached using the statistical concept of multi-dimensional response surface modeling. The idea is to empirically determine the response of some quantity to variations in its controlling factors. This response can be expressed as a mathematical function of these factors and this function describes the response surface of the quantity under study. One of the very significant advantages of using these statistical techniques is that a study can be designed which will produce the most information with the fewest number of points before any data is taken.

The construction, data taking, and analysis of Stage 1 of the ferrite-core coil study was completed. A linear model of probe impedance change (ΔZ) as a function of a number of coil construction factors was developed with the help of statisticians from the NIST Center for Applied Mathematics. It was found that the linear model worked well for the prediction of ΔZ but not for predictions of resonant frequency or inductance[3] .

The statistical design for the Stage 2 study was also completed. The additional coils we are planning to wind will provide the ability to produce a response surface with curvature (a model with quadratic terms) necessary for the predictions of resonant frequency and inductance. When this work is completed we will be able to quantitatively predict virtually any performance related electrical parameter of interest over the range of coil construction factors studied.

Single-coil Probes

The work on this project showed that the Stanford ΔZ program was not able to account for the self-resonant effects of the coils. As a result of this work, however, a new analysis technique that incorporates frequency into the predictive model was developed We think that the relative ranking of the construction factors determined by using the ΔZ program are correct, but the program is unable to produce accurate results for use in a quantitative ΔZ prediction model. Since the computer program cannot produce the needed results, an empirical experimental design for modeling air-core, single-coil probe sensitivity was generated.

Differential Probes

We produced and ran on the computer, two different experimental designs for air-core differential probes using the Stanford ΔZ program. This produced a relative ranking of the order of importance of construction parameters. Using these results, we designed and built four probes to experimentally verify some of the predictions of the program[4] .

Flaw Catalog

In order to simulate a variety of inspection situations to optimize eddy current probe designs, an extensive inventory of well characterized flaws has been under development. This year, three rectangular electrical-discharge-machined (EDM) notches, nine semi-elliptical EDM notches, two 5 mm CDF notches (named for the inventors, Capobianco, Dube, and Fizer), and six fatigue cracks ranging in size from five to nine mm long were produced in in 7075-T6 aluminum. Production of EDM notches in titanium has been initiated.

Quantitative Flaw Sizing

An apparatus for vacuum forming room-temperature-vulcanizing (RTV) silicone rubber casts of EDM notches has been developed. These casts or replicas were removed from the notch after curing and the profile was measured on an optical comparator. The discretized profile was then entered into a computer drafting program which calculated measurements such as areas and perimeters based on the replica measurements.

The differential probes built this year as part of the probe characterization studies were used for quantitative flaw characterization. A useful feature of this type of probe is a linear response to increasing flaw area within certain constraints such as probe orientation and flaw size. Flaw areas of EDM notches were determined from the vacuum-cast RTV flaw replicas. These constraints on the linear response of our four differential probes were mapped[5].

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1. L. L. Dulcie and T. E. Capobianco, "New Standard Test Method for Eddy Current Probes," Proc. of the 36th DoD Conference on NDE, US Army Aviation Systems Command, St. Louis, Mo, 1988.

2. T. E. Capobianco and D. F. Vecchia, "Coil Parameter Influence on Eddy Current Probe Sensitivity," Proc. of the Review of Progress in Quantitative NDE, 7, 487 (Plenum, NY), 1988.

3. T. E. Capobianco, J. Splett, and H. Iyer, "Eddy Current Probe Sensitivity as a Function of Coil Construction Parameters," accepted for publication in Research in Nondestructive Evaluation.

4. T. E. Capobianco, J. Splett, and L. L. Dulcie, "Optimizing Differential Eddy Current Air-Core Probe Design," The Review of Progress in Quantitative NDE, La Jolla, CA, 1990, to be published.

5. T. E. Capobianco and S. J. Ciciora, "Characterizing Differential Air-Core Eddy Current Probes," The Review of Progress in Quantitative NDE, La Jolla, CA, 1990, to be published.

New Standard Test Methods for Characterizing Performance of Thermal Imaging Systems

J. Cohen
Consultant
Bethesda, MD

It is only a few years since infrared thermography began its rapid rise from a minor to a major method of wide utilization in the commercial sector. Then the need for new and improved standard test methods for characterizing the performance of thermal imaging systems was identified and a program of writing the standards was begun.

The salient characteristics of the standards address the determination of resolution, detectability, and thermal sensitivity. The first test method for minimum resolvable temperature difference (MRTD), was issued by ASTM in 1988 (E 1213-87). The second, minimum detectable temperature difference, received full ASTM approval and is issued as E 1311-89. The Navy and NIST, among others, are already using the MRTD standard. Also, the previously published Tech Note[1] is the basis for a new chapter on IR thermography in a forthcoming NDT textbook.[2]

The work for the past year involved writing the standard test method for noise equivalent temperature difference (NETD). First, an assessment was made of three different methods to determine the best, based on precision and accuracy, and to establish acceptable temperature limits. Next, a standardized reference noise filter, which has no illegitimate effect on the noise, was specified in detail. The specifications given for the measurement instrumentation preclude any influence on noise, or the NETD value, or subjectivity in readings. The draft was just completed and is ready to undergo the extensive ASTM review process required for approval as a standard. The future work will be to guide the document through the review and revision process.

References

1. J. Cohen, "Elements of Thermography for Nondestructive Testing," NBS TN 1177 (May 1983).

2. W. J. McGonnagle, Nondestructive Testing, 3rd ed., Gordon & Breach, New York, in preparation.

PERSONNEL

The permanent staff of the Office of Nondestructive Evaluation is listed below.

ONDE PERMANENT STAFF IN FISCAL YEAR 1990

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

*Leonard Mordfin, who until recently was Deputy Chief, is now Group Leader for Mechanical Properties and Performance, Metallurgy Division.

OUTPUTS AND INTERACTIONS

A. NDE SEMINARS AT NIST

Dr. Torben Lorentzen, Riso National Laboratory, Roskilde, Denmark
"Bulk Residual Stress Studies by Neutron Diffraction"
October 24, 1989

Dr. John Gilmore, Lawrence Livermore Laboratory
"A Method for Monitoring the Mechanical States of Materials"
May 9, 1990

JOINT NDE/METALLURGY SEMINAR

Dr. J. D. N. Cheeke, University of Sherbrooke, Quebec, Canada
"Acoustic Microscopy of Engineering Materials"
February 7, 1990

B. INVITED TALKS BY ONDE STAFF

"Spectroscopic Studies of Collision-Induced Phenomena in Non Polar Gases," George Birnbaum, Goddard Institute for Space Studies, New York, New York, October 29, 1989.

"Intelligent Processing of Materials," H. T. Yolken, ASM Materials Science Seminar on Intelligent Processing of Materials, Indianapolis, Indiana, October 2, 1989.

"Applications of NDE in Intelligent Processing of Materials," H. T. Yolken, Boston Section of the Metallurgical Society and the Boston Chapter of ASM, Waltham, Massachusetts, February 12, 1990.

"An Introduction to Intelligent Processing of Materials," H. T. Yolken, ASM International Educational Symposium, NIST, Gaithersburg, Maryland, March 1, 1990.

"Intelligent Processing of Materials," H. T. Yolken, ASME Manufacturing International 90, Atlanta, Georgia, March 26, 1990.

"Intelligent Processing of Materials," H. T. Yolken, NIST Metallurgy Division Seminar, Gaithersburg, Maryland, May 3, 1990.

"A Kinetic Approach in the Study of Pressure-Broadened Molecular Band Shapes," George Birnbaum, The University of Texas at Austin, June 27, 1990.

C. AWARDS AND APPOINTMENTS

Department of Commerce Gold Medal

The Gold Medal Award for Distinguished Service, the Department of Commerce's highest honor award, is bestowed for distinguished achievements of major significance to the Department or the nation. George Birnbaum, Senior Scientist, Office of Nondestructive Evaluation, (NDE) was honored with a gold medal for his highly distinguished authorship and editorship in molecular spectroscopy and for his unusually outstanding scientific leadership in the NDE program.

Edward Bennett Rosa Award

The Rosa Award recognizes outstanding achievements in the development of meaningful and significant standards of practice in the measurement field. In recognition for his outstanding leadership in the standardization of mechanical testing and nondestructive evaluation (NDE) methods and measurements, the Rosa Award was granted to Leonard Mordfin, Group Leader, Mechanical Properties and Performance, Metallurgy Division, who served until recently as Deputy Chief, Office of Nondestructive Evaluation.

ASTM Award of Merit

An ASTM Award of Merit and the title of Fellow of the Society were awarded to John Gudas of the Office of Nondestructive Evaluation. Dr. Gudas was cited for distinguished leadership and exceptional contributions in the promotion and development of test standards for elastic plastics and for his role as a liaison to other organizations. Of equal importance is the recognition of his efforts to promote membership and encourage participation within ASTM Committee E24.

NIST Safety Award

The NIST Safety Award for Superior Accomplishment recognizes unusually significant contributions to the NIST Occupational Safety and Health program activities. Patricia L. Salpino was presented with the NIST Safety Award for her exceptional and effective efforts in fulfilling her duties as the safety officer of the Office of Nondestructive Evaluation.

George Birnbaum, Office of Nondestructive Evaluation, was appointed research professor in the Physics Department of The Catholic University of America.

G. V. Blessing was asked to serve on NASA Langley's Peer Review Panel.

NDE Researchers Honored by JSNDT

Nelson Hsu and Frank Breckenridge of the Center for Manufacturing Engineering received formal recognition from the Japanese Society for Nondestructive Testing for their research at NIST on calibration of acoustic emission test methods.

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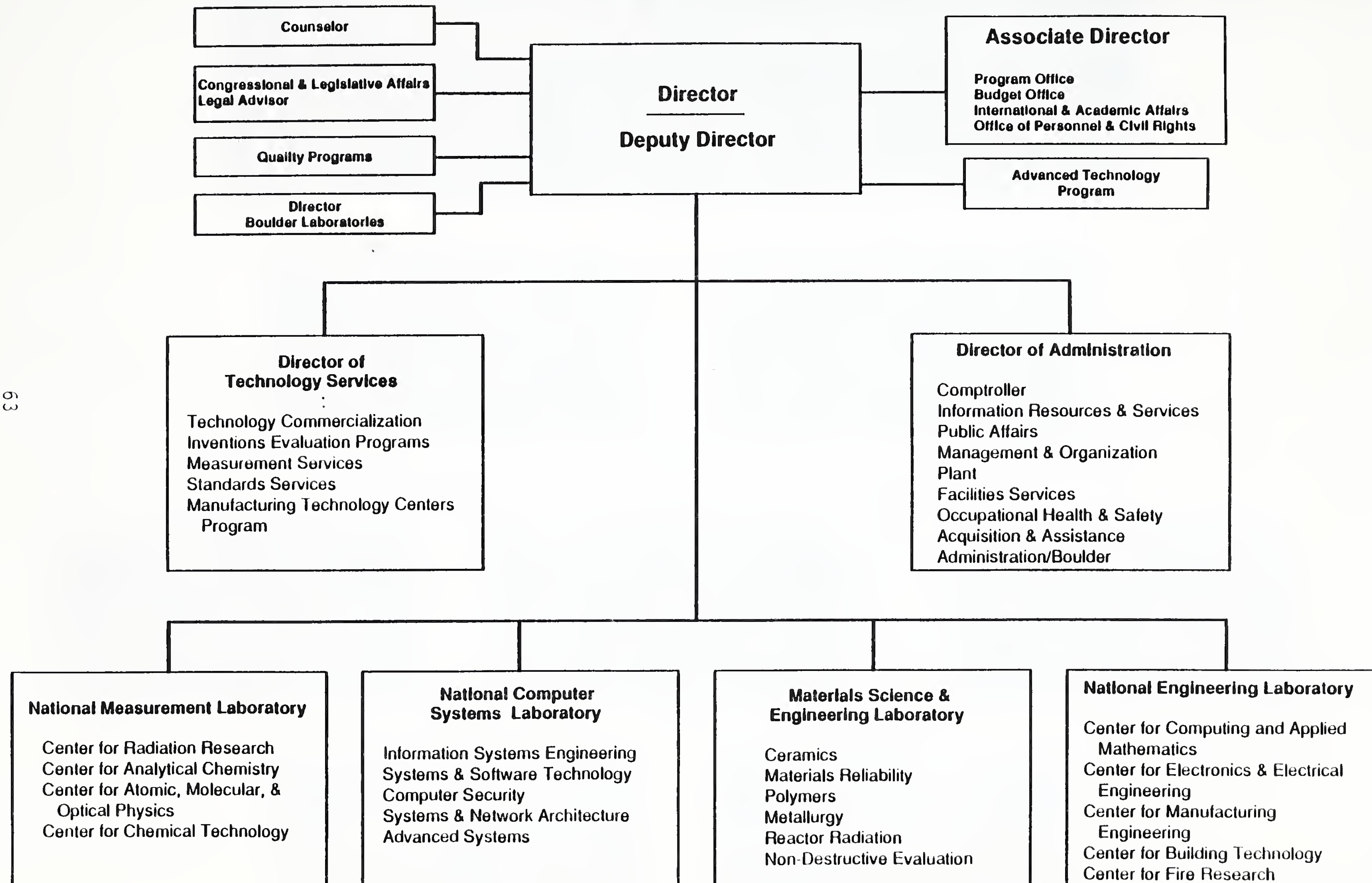

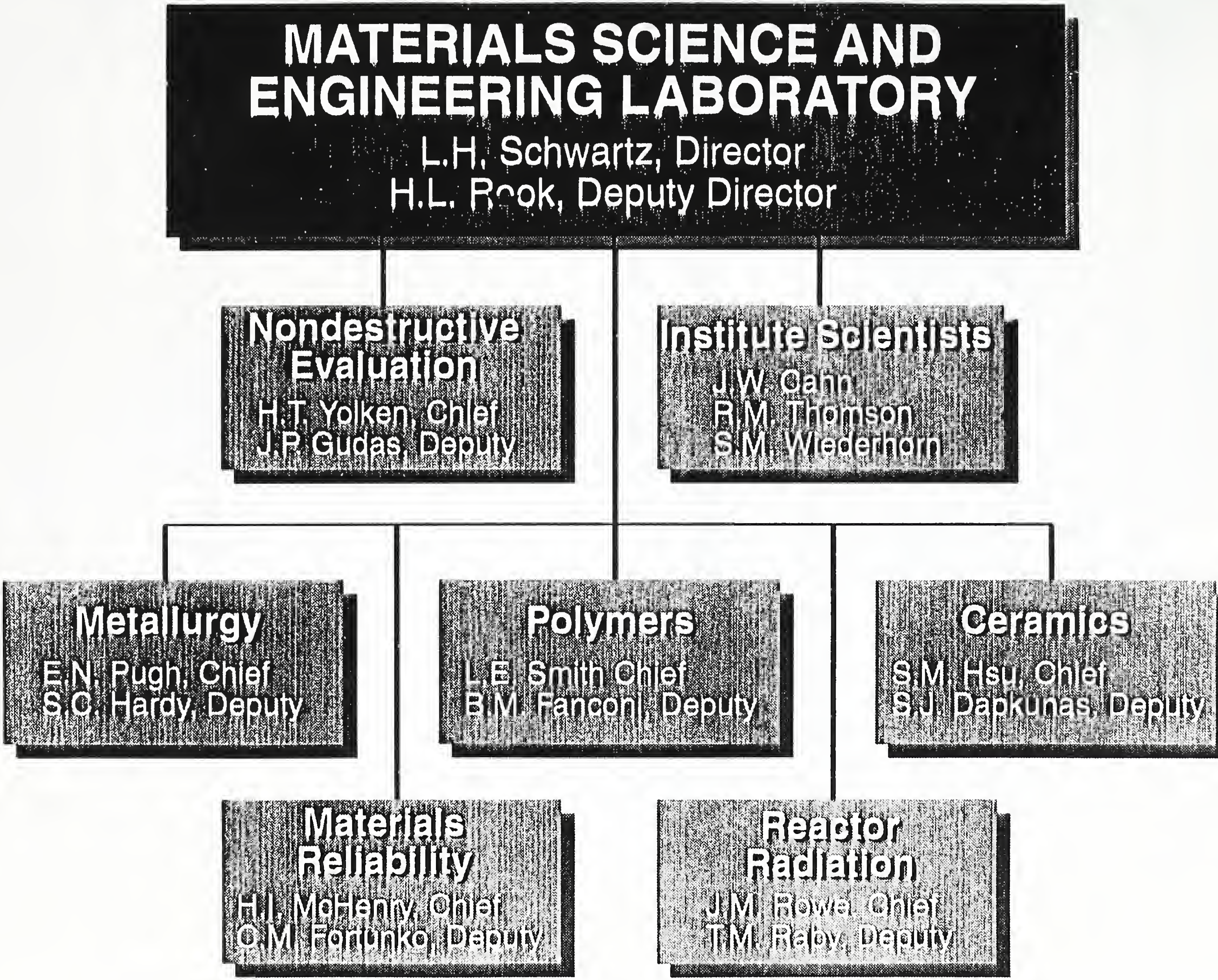
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(REV. 3-89)

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NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

BIBLIOGRAPHIC DATA SHEET

1. PUBLICATION OR REPORT NUMBER
02496

2. PERFORMING ORGANIZATION REPORT NUMBER

3. PUBLICATION DATE
December 1990

4. TITLE AND SUBTITLE
Technical Activities 1990--Nondestructive Evaluation

5. AUTHOR(S)
H. Thomas Yolken, editor

6. PERFORMING ORGANIZATION (IF JOINT OR OTHER THAN NIST, SEE INSTRUCTIONS)
U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
GAITHERSBURG, MD 20899

7. CONTRACT/GRANT NUMBER

8. TYPE OF REPORT AND PERIOD COVERED
Annual, FY90

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (STREET, CITY, STATE, ZIP)
National Institute of Standards and Technology
U.S. Department of Commerce
Gaithersburg, MD 20899

10. SUPPLEMENTARY NOTES

☐ DOCUMENT DESCRIBES A COMPUTER PROGRAM; SF-185, FIPS SOFTWARE SUMMARY, IS ATTACHED.

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

A review of the Nondestructive Evaluation Program at NIST for fiscal year 1990 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

13. AVAILABILITY
[X] UNLIMITED
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[ ] ORDER FROM SUPERINTENDENT OF DOCUMENTS, U.S. GOVERNMENT PRINTING OFFICE, WASHINGTON, DC 20402.
[X] ORDER FROM NATIONAL TECHNICAL INFORMATION SERVICE (NTIS), SPRINGFIELD, VA 22161.

14. NUMBER OF PRINTED PAGES
70

15. PRICE
A04

ELECTRONIC FORM