Institute for Materials Science and Engineering

NONDESTRUCTIVE EVALUATION

NBSIR 86-3434
U.S. Department of Commerce
National Bureau of Standards

Technical Activities
1986
One of several possible models of the liquid jet break-up process that takes place during the production of rapidly solidified metal powders by the inert gas atomization of liquid metal. Process models of this kind constitute an important part of the knowledge base which is needed in the development of an automated materials processing facility. See the report, "Sensors for Metal Powder Atomization Systems," by S.D. Ridder, F.S. Biancaniello, H.G. Semerjian and G.E. Mattingly.
Institute for Materials Science and Engineering

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

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INTRODUCTION

This report provides brief reviews of the technical activities of nondestructive evaluation (NDE) that were carried out by or for the National Bureau of Standards (NBS) NDE Program in fiscal year 1986 (October 1, 1985 through September 30, 1986). Collectively, these technical activities constitute the Bureau's NDE Program, which is managed programmatically and administered on a Bureau-wide basis by Office of Nondestructive Evaluation (ONDE).

During the last several years quantitative NDE has been applied by industry to the measurement of material properties and characteristics such as residual stress, texture, and various other microstructural features. Such applications have been receiving increased attention because of the nationwide effort to raise the quality of manufactured products. Clearly, the ability to monitor important material properties and characteristics during a manufacturing process can often be used to guide or to control the process and, thus, to help avoid the manufacture of subquality products or materials. In response to these developments, the Bureau's NDE Program has for a number of years sponsored work on the application of NDE to the measurement of materials properties and characteristics and more recently has placed major emphasis on such measurements in regard to on-line monitoring and process control in manufacturing.

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

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

Reports such as this one have been issued on an annual basis since 1978 and are commonly referred to as the "NBS NDE Annual Reports." A parallel series of reports, also issued annually, presents bibliographies and abstracts for the Bureau's technical reports and publications on NDE and its supporting technologies. The purpose of both of these report series is to serve as an introduction to the Bureau's NDE Program. Many readers will
want further details on specific aspects of the work or its outputs, and such inquiries are welcomed and encouraged, both by the principal NDE investigators (whose names and affiliations precede each of the articles in the report) and by ONDE. Either can be addressed in care of NBS, Gaithersburg, Maryland 20899, or reached by telephone via (301) 975-2000. Requests for further information and suggestions regarding the Program always receive prompt and careful attention.
NDE FOR CERAMIC AND METAL POWDER PRODUCTION AND CONSOLIDATION

This activity is broadly concerned with developing approaches, sensors, and procedures for nondestructively determining those properties of ceramic and metal powders, and of consolidated materials, that relate to the quality and performance of the materials and manufactured parts. The emphasis is on those measurements which 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. This activity includes: a study of pore size and distribution in alumina powder materials by small angle neutron scattering (SANS); near-surface characterization of ceramics by thermal waves; determining the elasticity of ceramic samples by measuring sound velocities; and investigation of appropriate sensors for metal atomization powder systems. Although all of these projects are aimed at developing sensors for the manufacturing process, the project on metal powders represents the most complete approach to this end. It involves the development of a process model, the investigation of a variety of sensors, the study of their performance in an actual inert gas/metal atomization facility, and ultimately the use of an expert system in 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.

SANS differs from other Nondestructive Evaluation methods in that it requires a highly specialized facility and certainly is not a candidate for use in the manufacturing environment. However, SANS provides the scientific understanding required to develop NDE methods, based on ultrasonic and AC impedance, which may be used in process monitoring and in-service inspection. The determination of elastic properties of ceramics by ultrasonics, and surface properties by thermal waves, has potential for process sensing and in-service inspection.

Representative Accomplishments:

- SANS measurements have shown that detailed information on particle size, size distribution, and shape can be obtained on undensified $\text{Al}_2\text{O}_3$ powder. Measurements on a compacted sample and analysis of the data yield particle volume fractions and porosities averaged over a one cubic centimeter volume. However, the current theory and analytical techniques which were derived on the assumption of small polydispersivity must be extended to deal with strongly polydisperse systems and coherent inter-particle interference effects (multiple scattering).

- A study of the porosity of alumina specimens has revealed that thermal waves propagated through a specimen are more sensitive to changes in porosity and less affected by surface roughness and optical absorption variations than thermal waves detected on the front (irradiated) surface. Other measurements have been made which suggest the effect caused by inclusions that may have been introduced in the ball-milling of the powder or in the firing of the sample.
Measurements of Young's modulus and Poisson's ratio by ultrasonic longitudinal and shear velocities in green-state (unsintered) ceramic compacts were used to study the effect of a naturally occurring flaw (calcination) on the elasticity of the sample at three stages of processing. Samples made from hard agglomerates resulted in low modulus compacts with a reduced strength compared with samples made from normal, spray-dried powder. A first step toward in-process monitoring was taken by measuring sound velocities in the powder during compaction. A patent disclosure was filed for this in situ sensor technique which is sensitive to the quantity of binder in the powder, excess moisture, and the rate of compaction.

Initial work on metal powder atomization was focused on developing a sensing system capable of measuring particle size in real time. To this end, an atomization system for preliminary fluid flow studies with surrogate fluids, such as water and low melting-point alloys, was constructed. The apparatus is being used as a model system to establish an understanding of liquid-gas interactions in the atomization process, to study optimum geometry and jet velocity, and how these variables can be used to control droplet formation. Several laser scattering techniques have been investigated for measuring particle size, number of particles per unit volume, and velocity, although final evaluation awaits tests with metal particles.

Nondestructive Evaluation of Alumina Materials with Small Angle Neutron Scattering (SANS)

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It is well known that stress failure of ceramics is strongly related to pore size and distribution, which depends on the early processing history. To investigate this correlation we have performed SANS measurements on two sequences of powder densification, both originating from the same undensified Alumina ($\text{Al}_2\text{O}_3$) powder material. In sequence (1) 500g of 99.99% pure powder was spray-dried with PVA and glycerol additives to form weakly agglomerated ("normal") material, which was then uniaxially compacted to the green state and subsequently heat treated to 1000$^\circ$C to form the "bisque." In sequence (2) the normally agglomerated powder was densified at 1000$^\circ$C to form solid ("calcinated") agglomerates, and this powder was then taken through the green and bisque states. The two sequences thus differed only in the nature of powder agglomeration.

SANS measurements were performed on the undensified powder and the two processing sequences, at several incident neutron wavelengths in the range of 0.5 to 1.0 nm. Scattering intensities were measured as a function of wavevector $Q$ (proportional to scattering angle). We have shown that particle size and surface area information can be obtained from analysis of the shape of these intensities at small and at large $Q$ [1,2]. The wavelength dependence is characteristic of strong multiple scattering ("beam broadening") which is interpreted as being produced by the powder particles in the powder sample, and from the pores of the undensified green compact and the densified
material. Analysis of these data give a particle volume fraction of 0.2 and porosities of approximately 0.47 for the compacted samples. Our analysis gives values of specific void area in the narrow range 6-7 m²/gm, somewhat smaller than gas absorption results for the normal green compact (20 m²/gm) but closer to the range of some determinations by mercury porosity (10-15 m²/gm). The analysis of size statistics from these experiments is incomplete, but we have determined that the porosity is strongly polydisperse in the densified samples, with ∆R/R > 1.0, where R is the radius of the void and ∆R = (∫R² - ∫R²)⁴. This apparently reflects a substantial polydispersivity in the original powder, which we estimate to be ∆R/R = 1.0.

Significantly, the SANS data from the two processing sequences were qualitatively and quantitatively similar. Thus, the nature of the powder agglomeration seems to have little effect on porosity, although it may affect the "framework" microstructure to which the current measurements are insensitive. We also performed SANS measurements on densified samples, heat treated near but just below the sintering temperature. We find significantly less beam broadening, indicating a substantial reduction in porosity.

The current theory and analytical techniques were derived on the assumption of relatively small polydispersivity. Accurate determination of size distribution characteristics from strongly polydisperse systems and the inclusion of coherent inter-particle interference effects into the theory are important problems that we plan to study in FY87.

References:


Near-Surface Characterization of Ceramics by Thermal Waves

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Thermal wave techniques are being evaluated as a possible NDE method to study ceramics because the types of defects of interest, e.g., pores, cracks, inclusions, etc., provide discontinuities in thermal properties to which thermal wave methods are sensitive. The work this year includes construction of a mirage detection system, study of thermal wave interactions in insulating materials, [1] continuation of the study of alumina, and investigation of polishing damage in vitreous silica.

5
The study of porosity in alumina specimens has continued with the mirage system instead of the photoacoustic cell used previously. Front and rear surface alternating temperature measurements were made while the specimens were heated from the front surface. Comparison with preliminary theoretical work indicates that measurements of the thermal wave propagated through the bulk of the material are more sensitive to changes in porosity and are less affected by surface roughness and optical absorption variations than measurements made on the front (irradiated) surface of the specimen. Thermal wave measurements of an alumina specimen have been made which suggest the presence of inclusions or specimen contamination [3]. Comparison with optical and electron microscopy and x-ray analysis has shown that the regions of the specimen in which strong thermal wave signals are present are contaminated with K, Cl, Fe, Si, and other elements. The contamination may have occurred as a result of the ball-milling of the powder or the firing of the specimen. These defects averaged about 30 μm in size and were in well-localized regions of the specimen.

Measurements have been made on optically polished vitreous silica to determine if residual damage exists which can be detected with thermal waves. Polishing scratches were detected on some specimens investigated by the photoacoustic technique. However, measurements made with the mirage technique have resulted in other signals which cannot be correlated with optical features.

Future plans include continuing the efforts to measure porosity, to detect residual polishing damage, and to examine the problem of crack detection in poorly conducting samples, e.g., alumina and glass. While cracks have been observed in highly conducting ceramics such as SiC, preliminary experiments suggest that in silica they are difficult to detect thermally even when clear optically.

References:


Characterization of Ceramics by Ultrasonics

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Since the latter stages of ceramic processing are energy intensive, the development of techniques for early process fault detection, especially those suited to on-line applications, are important. This research focuses on those material parameters which are candidates for quality control in the early processing stages.

The initial choice for a process-sensing variable is the material sound speed, a dynamic elasticity parameter. To this end, to make precise measurements of the shear and longitudinal sound velocities in fragile green-state (unsintered) ceramic compacts, dry coupling techniques using elastomers were developed. These techniques [1] were used in studying the effect of a naturally occurring flaw type--calcination or burning off of binder resulting in hard agglomerates--on the elasticity of samples at three stages of processing: in the compacted green state, after partial sintering in the bisque state, and after complete sintering in the dense state. Samples made from normal spray-dried powder were compared with calcined samples made from the hard agglomerates, which were conjectured to act as elastically uncoupled particles that would result in low modulus compacts. This was observed to be the case for the green and bisque samples, but not for the dense samples, reinforcing the need for fault detection in the early stages of a process. Subsequent fracture tests revealed a reduction in strength for the dense samples.

A first step toward in-process monitoring was then taken by measuring sound velocities in the powder during compaction [2,3]. Figure 1 illustrates the arrangement of transducers used on die shafts acting to couple the pulses of ultrasound into the powder samples. A patent disclosure has been filed for this in-situ sensor technique that is sensitive to a reduction of binder in the powder, the presence of excess moisture, and the rate of compaction. Figure 2 shows the result of monitoring Young's modulus in-situ on a normal powder sample, over a wide range of compaction pressures.

Future plans call for ultrasonically delineating porosity from calcination (especially in the green state), correlating the ultrasonic results with other nondestructive and destructive tests, and determining whether the sample surface structure as measured ultrasonically is a valid parameter for process control.
References:


Figure 1. Illustration of die shafts with ultrasonic transducers for sound speed measurements in sample powders during compaction.

Figure 2. Young's modulus as a function of the applied pressure for normal and calcined sample powders during compaction.
Sensors for Metal Powder Atomization Systems

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

Our work on metal powder atomization has focused on developing a sensing system capable of measuring particle size in real time and using this information in a process feedback loop that would continuously control the particle size being produced. The approach taken to realize this device centers on a joint effort between the Institute for Materials Science and Engineering and the Center for Chemical Engineering, with specific goals in three separate disciplines. (1) Droplet formation will be studied using high speed photography with an emphasis on optimizing the geometry of the atomization hardware. Process variables will be monitored to determine how particle size varies with gas jet geometry and velocity, metal flow rate, and type of atomizing as used. (2) Particle size sensing techniques will be evaluated and an appropriate device for metal powder measurement will be procured and installed in the Metallurgy Division's inert gas atomization system. (3) The atomization system will be modified for process feedback control, for example, by updating the high pressure gas system to allow computer control of gas pressure. The feedback loop used will depend on results of 1 and 2 above.

This year's accomplishments include the construction of an atomization system to be used in preliminary fluid flow studies, with surrogate fluids substituting for high temperature, molten metal, and high pressure inert gas systems. The surrogate fluids chosen are water and low melting point alloys; air and other gases can be used for the inert gas. These surrogate systems will allow initial parameterization and will establish an understanding of the liquid-gas interactions. The apparatus is currently being used to determine aspiration pressures developed in the gas jet impingement zone as a function of gas pressure and the metal stream delivery nozzle geometry. When complete, this information will determine not only the optimum geometry and jet velocity, but how these variables can be used to control droplet formation. This apparatus is also being used to photograph the aspiration and atomization of water. These investigations will determine what photographic equipment and techniques will best show the droplet formation sequence in the model system.
and later in the actual inert gas atomization system. Several point techniques based on laser scattering have been evaluated for measuring particle size distribution and velocity [2]. The third technique provides a direct measurement of the particle size distribution, since it is a single particle measurement technique. It has the added advantage that it also provides simultaneous measurement of particle velocity.

The study to understand the fundamental phenomena that control the powder characteristics will continue next year in the small transparent tank system. Conditions will be modified so as to approach the actual parameters in the real system. It is expected that these studies will be transferred to the inert gas atomization system.

References:


NDE FOR BULK METAL PROCESSING

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

Current efforts in this activity include: the development of NDE temperature sensors based on ultrasonic and eddy current techniques for determining the internal temperature distribution in hot metal objects; developing an in-process ultrasonic monitor for metal grain texture in manufacturing of aluminum products; research on the use of magnetic NDE methods to determine material properties; and determining the feasibility of utilizing ultrasonic and optical techniques for on-line monitoring of metal surface roughness during machining. There have been several noteworthy accomplishments in this activity during the past year.

- Today, many of the complex processes that make up the processing of steel rely upon intermittent and often inaccurate measurement of surface temperature rather than upon the quantity of fundamental import - the internal temperature distribution. In a joint research effort at NBS involving NBS researchers and research associates from The American Iron and Steel Institute, advances have been made on utilizing ultrasonic tomography to determine internal temperature distribution and liquid-solid interface position in solidifying metal bodies.

- During manufacture, the determination of internal temperature distribution in thin extruded aluminum sections for process control has been a troublesome problem for the aluminum industry. In a joint effort between NBS and the Aluminum Association, both theoretical and experimental results have shown that a multifrequency eddy current measurement approach in the 100 kHz to 1 MHz range appears likely to provide an NDE temperature sensor for process control.

- A critical part of the manufacturing process for about two billion pounds of aluminum beverage cans produced annually is control of texture or preferred orientation of the grains. In order to develop an on-line NDE sensor and save an estimated $40 million annually in the United States, researchers at NBS, Iowa State University, and Alcoa have demonstrated that an ultrasonic technique can monitor texture quickly. Ultrasonic results obtained with noncontacting electromagnetic transducers were compared to a reference technique utilizing neutron diffraction and qualitative agreement was obtained. Work is now progressing to seek quantitative agreement.

- The determination of surface roughness during machining of bulk metal parts is desirable for both quality control of the finished product and for control of the cutting or grinding process. An ultrasonic sensor has been developed for on-line monitoring of surface finish during machining of metal parts. The sensor is noncontacting and utilizes the liquid stream of cutting-cooling solution as a means to couple the ultrasonic signal to the metal.
Sensors for Steel Process Control

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Today, many of the complex processes that make up the processing of steel rely upon intermittent, often inaccurate measurements of surface temperature rather than the quantity of fundamental import the internal temperature distribution. By utilizing the internal temperature distribution, modern continuous casters can be controlled to produce optimal material quality commensurate with maximum casting speed and elimination of breakouts. The benefits of an internal temperature sensor have been estimated as equivalent to over $200M annually for the domestic industry. Such sensors also have important applications in reheat furnaces and continuous annealing operations.

The research reported here is aimed at establishing the feasibility of an ultrasonic tomographic approach to the measurement of internal temperature. The approach is based upon the strong effect of temperature upon the velocity of ultrasound. For example, a 1°C temperature change at 1000°C causes a 1-part-in-10⁴ velocity change for AISI 304 stainless steel, well within the scope of today’s laboratory instrumentation.

This year’s research has progressed on three fronts: application of the tomographic approach to solidifying bodies, evaluation of a subsurface temperature gradient sensor, and feasibility studies of an eddy current approach for thin strip. Progress with each is more fully discussed below.

Internal Temperature Distribution of Solidifying Bodies--The presence of a liquid domain within a cooling body introduces complications to the tomographic algorithms used for solidified bodies because of a discontinuous change in velocity upon melting. It is, therefore, necessary to map the interface between solid and liquid regions, as well as to determine the temperature distribution. Several measurement strategies have been identified for this including: shear wave transmission tomography (molten metal does not transmit shear waves), longitudinal wave transmission tomography using an iterative method to locate the solid-liquid interface, and reflection tomography combined with one of the above. The utilization of simplifying assumptions, such as uniform liquid temperature and symmetric heat flow coupled with these modified measurement methods, shows promise of adequately measuring internal temperature in solidifying bodies.

To experimentally evaluate these various approaches a fully automated 10 channel sensor has been designed, constructed, and is presently undergoing evaluation. The sensor is based upon measuring the times of flight (TOFs) of two orthogonal sets of five collinear rays propagating over a cross-sectional slice of a nominally 6-in. square metal bar. The capability also exists of simultaneously recording fan-beam TOF values at all ten receivers from each source. Thus, up to 100 measurements can be made in the time taken to scan the ultrasonic source over two faces of the bar (about 25 seconds presently). In the present arrangement an 800-mJ laser pulse is used as a noncontact ultrasonic source. It is scanned across the bar faces using a system of
moving mirrors. Ultrasonic signals at each receiver point are detected with piezoelectric transducers attached to conical AISI 304 stainless steel buffer rods (the design of noncontacting EMAT arrays to replace this contact approach has been completed).

Initial experiments are underway with solidifying aluminum. This system shows some of the physical features expected for solidifying steel, but at lower temperatures. Aluminum also offers the opportunity of systematically studying "mushy zone" effects by using Al-Si alloys of varying silicon contents. We expect to proceed to steel solidification studies by the end of 1986, and to evaluate, at the high temperatures of solidifying steel, the performance of high temperature EMAT arrays destined to replace the contact receivers in the current sensor design.

Subsurface Temperature Gradient Sensor--The steel industry today makes heavy use of thermal modeling to predict internal temperature distributions at various stages of processing. These models have grown in sophistication to the point where, if accurate heat transfer coefficients are known, the internal temperature can be predicted to within a few degrees everywhere. Unfortunately, the rate of heat flow through a steel surface is a highly variable quantity and only poor estimates of heat transfer coefficients exist for most processes of interest. However, if the temperature of the hot surface and the subsurface gradient were measured, then the heat transfer coefficient is determined and it becomes possible to calculate the temperature everywhere within simple shapes.

A proposed method of measuring the near surface temperature gradient is to observe surface-wave velocity dispersion. Surface (Rayleigh) waves are inhomogeneous waves whose displacement amplitude decays exponentially with depth. Thus, high frequency surface waves sample the elastic constants of the surface, while low frequency waves sample the constants of both the surface and interior. If a gradient in modulus/density exists (due to a temperature gradient), dispersion is observed. If the dispersion data (velocity values at specific wavelengths) can be inverted to determine the depth dependence of the modulus/density, then the subsurface temperature gradient can be deduced and the heat transfer coefficient effectively defined.

To evaluate this potentially important approach, we have modeled the propagation of surface waves in inhomogeneous media and are in the process of exploring the sensitivity of Rayleigh wave dispersion to temperature gradient.

Eddy Current Temperature Sensor--Noncontact ultrasonic approaches to internal temperature measurement currently utilize EMATs with magnetic fields of between 0.2-0.8 T. These fields are up to ten times stronger than the fields currently envisaged for the containment of solidifying steel in prototype electromagnetic thin strip continuous casters. These sensors are thus likely to significantly perturb the process of interest and contravene the first rule for successful sensing. Temperature and solidification sensors for this application might more promisingly be developed based upon electrical conductivity distribution measurement; a measurement which can be tackled with less intrusive methodologies.
This approach also has use in other metal processing areas and is being explored at NBS for temperature profiling purposes during the extrusion of aluminum. The latter application is being addressed in a cooperative program initiated in June with the Aluminum Association, and many of the conclusions of this work are expected to be directly relevant to the steel problem.

The eddy current approach to measuring temperature profiles in metallic materials is based on prior knowledge, for a given alloy, of the monotonic increase of electrical resistivity with temperature. The depth of penetration of an AC magnetic field (and the accompanying eddy currents) decreases with increasing frequency. Since impedance measurements utilize varying frequencies, they sense average resistivities over varying depths. By analyzing multi-frequency measurements, it is possible to obtain a reconstruction of the resistivity profile and thereby the temperature profile. To implement this approach, we have set up a computer-controlled impedance analyzer capable of performing measurements at hundreds of frequencies in a minute. Figure 1 shows examples of measured impedance plane data from (a) a solid brass rod and (b) a brass rod with a copper core demonstrating the changes associated with a nonuniform conductivity profile. The measured impedances will be used as input data for constructing the resistivity profiles.

![Figure 1](image-url)

Figure 1. Impedance plane plots of measured data taken on (a) a solid brass rod of 1/4 in. diameter, shown on the left; and (b) a brass rod with a copper core shown on the right. The horizontal axes give the real parts of the impedances; the vertical axes give the imaginary parts.
Another approach to eddy current determination of temperature profiles depends upon measurement of the resistivity and resistivity gradient at the surface of the test material. This, in turn, determines the temperature and the near-surface temperature gradient for a given alloy. With this information it is possible to obtain the solution to the heat flow equations and to calculate the entire temperature profile. This method depends on high frequency (100 kHz to 1 MHz) impedance measurements, a range readily accessible with the newly acquired instrumentation. A theoretical approach based on the JWKB (Jeffrey, Wentzel, Kramers, Brillouin) approximation has been developed for obtaining the resistivity and resistivity gradient from the high frequency data, and is presently being used to evaluate the approach.

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

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It has been estimated that the U.S. aluminum industry produces about two billion pounds of material for can manufacture annually. A crucial part of the manufacturing process is control of texture or preferred orientation of the aggregate of single crystals making up the rolled sheets from which cans are made. Proper texture is essential in providing the mechanical properties needed to mechanically form the cans. If better process controls than current ones could be implemented, an estimated savings of about two cents per pound or $40 million annually could be effected.

Currently, texture is characterized after an intermediate hot-rolling step by a temperature measurement. A high degree of cube texture is desirable at this point in the manufacturing process, i.e., the cubic single crystals should have one of their axes aligned in the rolling direction. In some cases, temperature measurement is inadequate, and rejection of the material occurs when it is later discovered that the rolled sheet cannot be successfully deep-drawn to can shape.

The need for a better process control monitor to evaluate texture has led to a consideration of ultrasonic velocity techniques because ultrasonics has the potential for real-time texture measurement. In contrast, other techniques such as x-ray or neutron diffraction pole figures cannot be made in real time and could require samples to be taken off line to a radiation source.
Recent theoretical advances at NBS and elsewhere have shown that velocities of bulk and surface acoustic waves in rolled aluminum sheet are related to three of the orientation distribution coefficients (ODC) which occur in the expression for the orientation distribution function [1,2,3]. This function gives the probability that a single crystal has a certain orientation relative to the rolling direction.

In the current program, tests on two aluminum alloys were performed. First, a proof of concepts test was performed on 5052, a common alloy having a cube texture. The purposes of this study were to determine which orientation distribution coefficient can be used to characterize cube texture and to determine precision and ease of measurement of various ultrasonic systems for texture characterization. The ODC were measured using shear-horizontal (SH) waves at normal incidence, guided wave modes which were SH and Lamb waves, and Rayleigh surface waves. Contacting piezoelectric and noncontacting electromagnetic acoustic transducers (EMATs) were used.

As an example we show the angular variation of the guided SH waves in Figure 1. Here EMATs were used to transmit and receive SH waves propagating in the plane of the plate at different angles $\psi$ to the rolling direction. The difference in velocity $\Delta V(\psi)$ is given by [2]

$$\Delta V_{SH}(\psi) = AW_{440}\cos^22\psi$$

(1)

where $A$ is a constant and $W_{440}$ is an ODC. The maximum in the velocity along the rolling and transverse directions shown in Figure 1 indicates cube texture; all other common aluminum textures give a minimum in these directions [4].

A neutron scattering pole figure made using the same specimen is shown in Figure 2. It shows a high degree of cube texture, in qualitative agreement with ultrasonic results. A computer program is being written to obtain the ODC from pole figures to compare with ultrasonic results. From our ultrasonic studies, we concluded: the ODC, $W_{440}$, is easily measured with both SH and Lamb waves and appears to characterize cube texture well. Measurements were easier to make and somewhat more repeatable with EMATs.

A similar series of measurements were next performed on specimens of 3004 aluminum alloy which are used in can manufacture. Specimens had different hot-rolling temperatures and hence different amounts of cube texture. The ultrasonic techniques described above were used to characterize texture in these specimens after cooling to room temperature.

We found that two of the ODC were relatively insensitive to rolling temperature. However, the values of $W_{440}$ measured with both SH and Lamb waves increased with decreasing exit temperature, in agreement with metallurgical considerations. The correlation of $W_{440}$ with exit temperature is promising for the use of ultrasonics. Probably only $W_{440}$ is necessary to discriminate between acceptable and unacceptable texture. To ascertain this, a larger database from additional specimens needs to be examined and quantitative comparison made between ultrasonics and neutron diffraction pole figures which can be considered as an accurate reference technique. In addition, comparisons should be made with deep-drawing tests. We hope to address these problems in the near future.
Figure 1. Angular variation of guided SH wave in rolled aluminum alloy plate. Open circles are data; solid line is theoretical value based on fitting equation (1) to data at $\psi = 0^\circ$, $45^\circ$. Minimum at $45^\circ$ indicates cube texture.

Figure 2. Neutron diffraction pole figure for rolled aluminum alloy plate. Rolling direction is at top of figure. Solid lines are contours of intensity greater than that for random crystallite orientation; dashed lines are contours with intensities less than random. Pole figure shows specimen to have cube texture.
In summary, we have identified a key step in aluminum can production where ultrasonics can be used as a process control monitor, namely, texture measurement after hot-rolling. We have examined two alloys having texture with various ultrasonic techniques and identified two techniques which appear the most promising: measurement of angular variation of SH\textsubscript{0} and S\textsubscript{0}-modes. An SH\textsubscript{0}-wave is a plate wave moving parallel to the surface of the plate; the atoms are excited and moving perpendicular to the propagation direction and in the plane of the plate. An S\textsubscript{0}-wave is a plate wave moving parallel to the surface of the plate; the atoms are excited and moving perpendicular to the propagation direction and perpendicular to the plane of the plate. S\textsubscript{0}-waves are the lowest order Lamb waves and have long wavelengths. Both the SH\textsubscript{0}- and the S\textsubscript{0}-modes are measures of the ODC, W\textsubscript{440}, which characterized cube texture well. Furthermore, for 3004 alloy sheet, values of W\textsubscript{440} increased as exit temperatures decreased. This result, in qualitative agreement with metallurgical considerations, is a necessary condition for the use of in-process ultrasonic texture monitoring.

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Magnetic Method for Nondestructive Evaluation

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A variety of magnetic methods are available for nondestructive evaluation of metals, both of defects and material properties. A number of these, such as magnetic flux leakage, magnetic particle inspection, and eddy-current testing, are widely used in industry, primarily for the detection of defects. Some other methods have been used on a limited scale but still require development to make them more reliable and more widely applicable. These include: Barkhausen noise, magnetoacoustic emission, magnetomechanical damping, and AC magnetometry. Finally, magnetic property measurements, including saturation magnetization, coercivity, and initial permeability, contain a wealth of information concerning material properties that have barely begun to be exploited.

To date, this project has been primarily focused on magnetic leakage field methods, especially the magnetic particle method, and on Barkhausen noise measurements. However, further progress in the use of magnetic methods, especially as applied to the measurement of material properties, will depend on improving our understanding of how the measurement of magnetic properties
correlates with material parameters such as alloy composition, heat treatment, stress states, etc. It is clear that a multivariate approach will be required. That is, two or more magnetic properties can be expected to yield useful information, such as grain size, where a single property measurement would give such information only under very restricted conditions. To this end, we have been expanding our ability to perform magnetic measurements, both for nondestructive evaluation and for other purposes. During the present year, a vibrating sample magnetometer has been put into operation and is in the process of being computerized. Also, a unique automated ac susceptibility apparatus has been developed. The latter instrument allows the measurement of initial magnetic susceptibility over a frequency range of 20 Hz to 100 kHz. A unique feature is the ability to measure both the real and the imaginary parts of the susceptibility over a wide temperature range. Initial studies are expected to center on the correlation of ac susceptibility, coercive force, and Barkhausen noise with the microstructure of iron-carbon alloys.

Work has continued in cooperation with the American Society for Testing and Materials (ASTM) Committee E7 and the Standards Subgroup of the Joint Services Technical Coordinating Group on Nondestructive Inspection (JTCG-NDI) on improvement of magnetic particle inspection standards. A major problem with both the ASTM standard (ASTM E709) and with the new military standard (MIL-STD-1949) is the tool steel test ring currently used to verify magnetic particle performance. A recent test held by members of ASTM Subcommittee E7.03, Society of Automotive Engineers (SAE) Committee K, and the American Society for Nondestructive Testing (ASNT) Aerospace Committee revealed a wide variation in the performance of such rings. An attempt is being made to select a steel and a heat treatment with will reduce the variability in performance of the test rings. Further, even when such variability is reduced to acceptable levels, the currently specified test ring does not do an adequate job of testing the ability of particles to detect narrow cracks. The design of a new test device is being explored at NBS.

Ultrasonic and Optical NDE of Surface Roughness

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This report covers research into ultrasonic and optical NDE techniques for the measurement of surface roughness. The wavelengths of the probing radiation for the two techniques differ by over two orders of magnitude. For the optical research we are using the HeNe laser wavelength of 0.6328 μm; for the ultrasonic research, the wavelength is 40 to 1500 μm. Since the ratios of roughness height and spacing to incident wavelength are important parameters affecting the scattering phenomena, the two techniques are complementary; that is, the resolution limit of the ultrasonic technique and the range of the optical technique are about the same, approximately 0.5 μm. The present emphasis of the ultrasonic work is the development and demonstration of a real-time in-situ tool-condition monitor for a computerized numerical control
(CNC) machining center work station. The present emphasis of the optical work is the development and demonstration of an instrument to measure surface finish in a final inspection work station. Both work stations are in the NBS Automated Manufacturing Research Facility (AMRF).

Ultrasonic Characterization of Surface Roughness--The goal of the ultrasonic portion of the project is to develop and demonstrate new ultrasonic techniques and tools for monitoring and characterizing surfaces, primarily for in-process control of materials/part processing. Coupling of the ultrasonic pulse to the surface can be by immersion, by air, or by a laminar stream of liquid (squirter). The principle used is that the roughness of a surface affects the scattering of ultrasonic waves incident on the surface. The problem is to evaluate the surface based on the scattered energy. The surface is intrinsically important as a feature of the part or material and also as a parameter directly related to the process. For example, in unattended machining the surface finish reflects tool condition (worn or broken tool) and can be used as an automatic tool management strategy [1].

The emphasis in FY85 was on measurements on flat stationary surfaces and on the basic technique. The current work emphasizes the development of a demonstration system for in-process control of tool condition and improvements in the robustness of the techniques.

A new nozzle for squirter coupling of ultrasound patented by Martin-Marietta was evaluated. Its use significantly decreases the signal variations and signal drop out which sometimes occur with other systems. A flexible extension was fitted to such a nozzle to permit the transducer/nozzle to be located remotely from the work piece surface. By proper choice of the tubing material, the extension acts as a waveguide and remarkably good ultrasonic back-scattered signals can be obtained from a surface even with a severely bent extension. The system has been used to obtain ultrasonic back-scattered signals from round parts rotating with a surface speed of over 300 m/s, a surface speed range common in turning parts. The system worked well with the three coolant/cutting fluids so far tested as the couplant stream. The resolution obtained using the peak amplitude of back-scattered ultrasound to monitor the surface of rapidly rotating parts was better than 3 μm. Signal processing techniques are expected to significantly improve this resolution. Peak-amplitude signals which were collected in real time from a rotating part with two roughnesses differing by 9 μm are shown in Figure 1 [2].

Some preliminary results show very interesting capabilities using higher frequencies (15 to 40 MHz) and focused transducers. On static parts a resolution of better than 0.5 μm in the roughness average Ra has been achieved. Focusing appears to lead to a profilometry capability. The surface topography of a 1-μm sinusoidal surface has been mapped in a preliminary measurement indicating the possibility of developing real-time in-situ capability for profilometry rather than only a spatial area average. Preliminary work has also made use of non-specular scattering by aligning the transducer (or stream) for non-normal incidence. In this case, a perfectly smooth surface gives zero signal and the signal amplitude increases with increasing roughness as opposed to the back-scattered signal which decreases with increasing roughness. This relationship suggests a ratio method for enhanced resolution.
Figure 1. Peak-to-peak back-scattered ultrasonic amplitude from a rotating part with two surfaces differing by 9 μm R\text{a}.

Optical Measurement of Surface Roughness--During FY86 we also made progress on both the fundamental and applied aspects of our research into optical measurements of surface roughness. On the fundamental side we developed a computer code to describe the fully three-dimensional optical scattering distribution from rough surfaces. The code assumes a simple phase screen approximation to characterize the outgoing scattered wave. It uses as input information a 512 x 512 square array of surface data points to describe the topography z(x,y) of a surface in three dimensions, and it yields an angular distribution of 87 x 87 points spaced over both the polar and azimuthal angles of the hemisphere. Preliminary results for the scattering from a rough surface are shown in Figure 2. The incident light is assumed to be incident from the right hand side descending in the sin\(\theta\), z plane at a polar angle of 54°. The resulting scattering pattern shows a strong specular peak with relative intensity equal to one and a weaker diffuse scattering distribution. Because the original surface is significantly rougher along the sin\(\theta\) direction, most of the scattering takes place along that direction and very little scattering is observed in the \(\phi\) direction out of the plane of incidence. Comparisons with scattering data obtained with the NBS developed DALLAS optical scattering instrument [3] will test the accuracy of the calculation.
In the applied part of the optical research, we have been developing two surface roughness inspection instruments, one for automated inspection of surfaces for the AMRF and one for monitoring the roughness of fairly smooth components with airfoil shapes that are difficult to inspect. For the latter project, data have been taken as a function of proximity to the leading edge of the airfoil and, hence, as a function of surface curvature. We are currently analyzing the correlation between three variables: rms roughness average as measured by a stylus, diffuseness of optical scattering as measured by the optical instrument, and surface curvature.

Similar work was done using experimental results from the DALLAS instrument. Figure 3 shows the empirical relationship between the roughness average $R_a$ of a set of hand lapped parts, as measured by a stylus, and the width of the optical scattering distribution in terms of the variance $\sigma^2$ expressed in (degrees)$^2$. 

Figure 2. Calculated light intensity scattered from a rough surface, shown as an angular distribution (in arbitrary units) over a hemisphere. $\theta_s$ and $\phi_s$ are the polar and azimuthal scattering angles, respectively.
Figure 3. Roughness average $R_a$ measured by a stylus instrument plotted versus the variance $\sigma^2$ of the optical scattering intensity pattern along the polar scattering angle $\theta$. The variance is expressed in units of (degrees)$^2$. These data were taken for a set of hand lapped, stainless steel surfaces. The parameters of the best fit straight line are also shown.

References:


NDE FOR COMPOSITES 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: determining the feasibility of utilizing fluorescent spectroscopy, dielectric measurements, and microwave techniques to monitor the processing of polymer matrix composites; applying ultrasonic and acoustic emission techniques to improve the understanding of, and the ability to monitor, interfaces; and utilizing x-ray diffraction techniques to increase the understanding of residual stress in composites. Noteworthy accomplishments in this activity include:

- Research demonstrated the applicability of two complementary nondestructive evaluation techniques for cure monitoring and process control of epoxy resin composites. One technique combines the sensitivity of the fluorescence intensity of some organic dyes to local viscosity with embedded optical fibers. During the past year refractive index effects were quantified so that appropriate optical systems could be selected. The second technique is based on dielectric measurements utilizing embedded electrodes. Research during the past year demonstrated the use of conductivity and dielectric loss mechanisms to follow the cure of several different resins.

- In order to monitor the processing of composites with nondestructive evaluation sensors, the effects of residual stress, which greatly affect the mechanical behavior of composites, must be understood. To this end, aluminum reinforced with silicon carbide particles was studied both experimentally and theoretically. Quantitative stress levels were obtained and the results show that the matrix is in hydrostatic tension and the particles are in compression.

- Interfaces in composites have a major effect on material properties. In order to gain more knowledge about interfaces and to develop nondestructive evaluation techniques for characterizing them, a theoretical study was completed for interface effects on ultrasonic phase velocity and attenuation in composite materials. A wave scattering approach was used to calculate attenuation from the scattering cross section of a single particle. The results suggest possible use of ultrasonic waves to study and characterize interfaces in composite materials.

- A theoretical and experimental study using guided interface ultrasonic waves (Stoneley waves) to study metal matrix composite interfaces was initiated. Near agreement between theory and experiment was obtained for a model system and the nondestructive evaluation technique might prove useful for detecting poor quality interfaces.
Cure Monitoring in Epoxy Resin Composites

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The need for process control in the production of epoxy resin-based composites has stimulated investigations into the use of fluorescence and electrical measurements for monitoring cure reactions. This need has become more pressing as the use of composites offers important advantages in strength and weight as well as potential savings in fabrication costs.

With the development of suitable techniques, reported last year, the current work on electrical measurements has explored the use of conductivity and dielectric loss mechanisms to follow cure of several systems. The fluorescence technique combines the sensitivity of the fluorescence intensity of some organic dyes to local viscosity and optical fibers, as a means of probing the interior of a thick specimen.

During the past year, fluorescence experiments were conducted to investigate factors that may affect or limit the usefulness of optic fibers in cure monitoring systems. One factor investigated was the effect of the difference in the refractive indices of the optic fiber and surrounding medium. In order for a glass fiber to operate as a waveguide, the refractive index of the glass must be higher than that of the surrounding material. The penetration depth of the evanescent wave and the efficiency of the waveguide to collect the fluorescent light depend on the refractive index difference. In addition, the refractive index of the resin system is expected to change during cure as a result of changes in density and chemistry. To explore the degree to which the observed fluorescence is affected, experiments were conducted using dyes dissolved in mixtures of two solvents having different refractive indices.

All experiments were carried out using a setup that simulated the arrangement envisaged for real-time cure monitoring of composites. Laser light of 501.7 nm was focused onto one end of a glass fiber of approximately 100 μm diameter. The glass fiber, 1.47 refractive index, was immersed in mixtures of propanol-1 and o-dichlorobenzene containing the dye rhodamine-B. The light exiting the other end of the fiber was focussed onto the entrance slit of a double 0.25-m monochromator and the intensity monitored as a function of frequency using photon counting electronics.

The fluorescence intensity as a function of the refractive index of the solvent mixture is shown in Figure 1. The intensity of the fluorescence from a solution whose refractive index is lower than that of the fiber is about 1000 times greater than the intensity from a solution whose refractive index is higher than that of the fiber. The refractive index range of the transition is rather narrow, about 0.05. This suggests that as long as the refractive index of the waveguide is higher than that of the curing resin by at least this amount there should be little effect on the measured intensity caused by small changes in the refractive index during cure. The residual intensity observed where the medium refractive indices substantially exceeds that of the fiber is attributed to emission excited by light which escapes the fiber at the air/fiber/solution interface.
In future work on the fluorescence technique, optical fibers will be used to probe consolidation and fiber wetting in polymer matrix composites.

The electrical measurements were carried out on three different resins cured with imidazole, and one of them was also cured with a diamine curing agent. All three resins showed similar behavior with the imidazole curing agent and one of them was intensively studied at a cure temperature of 55°C for comparison with other techniques. The frequency used for conductance measurements was 50 Hz.

A normal cure curve for conductance is shown by the upper curve of Figure 2. The leveling off of the conductance, at long times after the main cure, is associated with a loss mechanism, as shown in the last year's report, and slowly evolves with the resin. The data obtained by isothermal differential scanning calorimetry (DSC) and ultrasonic measurements on the same systems showed very similar times to cure. In contrast, the viscosity data show a rapid rise earlier than the conductance data and become impossible to obtain while the cure is still progressing.

![Figure 1. Fluorescence intensity transmitted in optic fiber immersed in solvent mixtures.](image)

In addition to studying a full cure cycle on freshly prepared resin, mixed resin was allowed to age at room temperature prior to cure for increasing periods of time. The aged material was then cured at 55°C with the results shown as the lower curves in Figure 2. The time shifts, needed to superimpose the curves, defined a new cure curve at room temperature whose time scale extrapolated well from higher temperature data. These results agree with a previous conjecture that a simple conductivity measurement can be used to monitor the state of a mixed resin system prior to cure; this would be useful in defining the processing conditions (time, temperature) required to achieve a predetermined degree of cure.
Figure 2. Conductance at 55°C for epoxy - imidazole resin.

The measurements of the system using a diamine curing agent show some new features not found in the same resin cured by imidazole. The conductance as a function of time, Figure 3, shows a rapid decrease with time as the resin gels, but at about the time the resin has hardened the conductance increases and goes through a maximum. The loss spectrum is typical of a dipolar loss mechanism that extends to frequencies below the lowest frequency used. The appearance of this loss is clearly due to the lower conductance level of the resin system using the amine curing agent. Other measurements, including ultrasonics and DSC, show that this loss process occurs after the resin has set.

Figure 3. Conductance at 45°C for epoxy - diamine resin.
Interface Effects on Phase Velocities and Attenuation in Composite Materials

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Strength and mechanical behavior of composites depend strongly on the properties of interfaces between the reinforcing phase and the matrix. Interface characteristics are affected by both thermomechanical processing and by mechanical-load history. Although strong interface bonds promote high strength, for some specialized applications we may seek weak interfaces to enhance damping.

The present study examines the dependence of overall elastic moduli and attenuation on the presence of an interface transition layer. We do not assume a sharp discontinuity of elastic properties between the reinforcing phase and the matrix (as would exist for perfect bonding). Instead, we consider the effect of a thin transition layer through which properties change continuously. We consider wave propagation through a composite of a random distribution of spherical particles with interface transition layers in a homogeneous matrix. Using a wave-scattering approach, we derive effective phase velocities of plane longitudinal and plane shear waves. We have presented long-wavelength approximate results, and have derived expressions for both phase velocities and attenuation [1-4]. The latter is calculated from the scattering cross section of a single particle. We also derived the frequency dependence of the scattering cross section [3,4]. The results suggest possible uses of ultrasonic waves to study and characterize interfaces in composite materials.

References:


Internal Strain (Residual Stress) in Composite Materials

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Internal strain, or residual stress, interferes with many materials uses. Its detection, explanation, measurement, and control present many problems to materials scientists and engineers. This is especially true for multiphase materials which are prone to internal stress. This is the case because the phases possess different thermal expansivities and temperature change induces stress through incompatible strains. Even small temperature changes may cause large stresses. As an example, consider SiC particles embedded in an Al-alloy matrix. The temperature decrease, ΔT, required to cause a hydrostatic tensile stress equal to the Al-alloy yield strength, σ_y, 274 MPa, is

\[ ΔT = \sigma_y / 3B^m(\alpha^m - \alpha^P) = 64 \text{ K} \]  

(1)

where B denotes bulk modulus, \( \alpha \) is the coefficient of linear thermal expansion, superscript m matrix, and superscript P particle. Here, we use the ambient-temperature physical properties:

\[ B^m = 74.9 \text{ GPa}, \ \alpha^P = 3.3 \cdot 10^{-6} \text{K}^{-1}, \text{ and } \alpha^m = 22.5 \cdot 10^{-6} \text{K}^{-1} \]

Many methods exist for studying internal stress including x-ray diffraction, ultrasonics, Barkhausen noise, and photoelasticity. By far, the most widely used of these methods is x-ray diffraction. Our experimental studies use a standard commercial x-ray diffractometer, without modification. We use a \( \sin^2 \psi \) method, in modern version [1], to account correctly for \( \epsilon_{13} \) strains. We are currently studying reinforced metal-matrix composites, especially SiC_p/Al and SiC_x/Al.

Our approach has been to try to combine measurements with modeling. So far, our modeling consists mainly of elastic sphere-in-hole calculations. The calculations provide an essential background for guiding the measurements and their interpretation. We hope to extend the modeling to an Eshelby-inclusion approach and include plasticity.

Here, we summarize results on SiC_p/Al with details published elsewhere [2,3]. Equation (1) shows that cooling only 64 K below the material's consolidation temperature (where good interfaces form) causes a matrix tensile stress equaling the yield strength, 274 MPa. Further cooling might workharden the material and increase the stress. Table 1 summarizes the measurements with a and c denoting unit-cell dimensions, V volume, \( \epsilon \) strain, and \( \sigma \) stress. These results represent average macroscopic stresses. Transformation to local polar co-ordinates suggests stresses up to 419 MPa.
Table 1. For both matrix and particles: unit-cell dimensions, strains, and calculated stresses.

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Reference Value (from virgin powder)

References:


Measurement of Flow Birefringence at Microwave Frequencies

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The object of the project is to develop microwave sensors to monitor polymer properties of flowing, molten thermoplastic polymers. The sensors would be mounted at right angles in the wall of a flow channel and detect the small differences in dielectric constant induced by flow at the boundary. The difference would reflect molecular size distribution and extent of crossbinding.

The thrust of the work this year has been to design and fabricate a microwave sensor with sufficient sensitivity to detect the small dielectric differences. A hairpin line was selected because theoretical consideration showed that its propagation characteristics are very sensitive to the properties of the material on top of the line. The hairpin line consists of a repetitive sequence of conductor strips each in the shape of a hairpin. The conductors are laid on top of a thin dielectric substrate. The adjacent conductors couple with each other via field lines which extend into the material above the hairpin line. The detailed description of the propagation of energy along the hairpin line requires specific calculation of the coupling of adjacent lines. The present model assumes that all the lines are equally spaced and that the thin conductor model can be used to calculate the coupling of adjacent lines. Based on this model we designed a hairpin line and had it fabricated commercially with dimensions shown in Figure 1. Examination of the fabricated line under a microscope showed that the lines and line spacings were very uniform and that the actual line looked very much like the pattern in Figure 1. The hairpin line was connected to a 50 Ω strip line via a tapered strip line. The impedance of the hairpin line was estimated by using the model and care was taken to match the impedance to a 50 Ω line. The tests of this set-up proved to be negative, since no signal was transmitted through the line.

Work was undertaken to explain this negative result. Three transmission lines were fabricated on 0.032-in. material. They were a meander, hairpin and comb lines. The results of the transmission measurements are shown in Figure 2. As expected, the meander line shows no band pass properties. The comb line is supposed to have a wide band pass and this is reflected in the transmission data (the reason for the existence of the two peaks is not clear yet). The hairpin line was designed to operate at 2000 MHz with a bandwidth of a few hundred megahertz. The data show that the model does not predict the bandwidth properly. The transmission peak of the hairpin line is near 2000 MHz but the amplitude is too low by an order of magnitude.
HAIRPIN LINE
(625 lines/inch)

Figure 1. Cross-sectional view of the hairpin line which was fabricated for the preliminary tests.

Figure 2. Results of transmission measurements on various lines. \( V(t) \) is proportionate to the power transmitted through the line and \( V(r) \) is proportionate to the power transmitted through a reference branch of the circuit.
Further work is needed to upgrade the model for predicting hairpin line behavior. It was noted that hairpin lines used in commercial integrated microwave circuits do not have equally-spaced conductors. The conductors of the same hairpin are widely spaced while the conductors of adjacent hairpins are much closer. An upgraded model of the hairpin line will provide for different conductor spacing and a more realistic calculation of the coupling of adjacent lines. The model will be checked with a small number of lines (approximately 40 per conductor) etched on 0.032 material.

Guided Interface Waves

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The performance of advanced metal matrix composites is dependent upon the properties at the metal-reinforcement matrix interface. Of particular import are the interface adhesion and local elastic properties. It seems reasonable to hope that characterization of interfaces might be possible through the use of interface elastic waves. The existence of interface waves at the planar interface between two media of differing elastic constants/densities was first predicted by Stoneley [1] and later observed in seismograms following major earthquakes. It has since been shown that very restrictive conditions must be met by the two media in contact if the wavevector (or velocity) of the interface wave is to be a purely real number so that non-attenuating propagation occurs [2].

For most material couples, the restrictive rules are not exactly met, but modes still exist wherein the wavevector of the interface wave is found to be complex [3]. Thus, the interface wave suffers attenuation (at a rate determined by the magnitude of the imaginary component) as it propagates. The physical mechanism of attenuation is the radiation of elastic energy away from the interface. If the attenuation is not too great, these interface waves may still be used to characterize the interface region provided the relationships between velocity and local elastic properties can be established.

Our first year's effort in this area has focused upon a combined theoretical/experimental study for testing these concepts using guided interface wave propagation in a model system consisting of a stainless steel rod shrink-fitted into an aluminum matrix. The normal modes of a cylindrical rod in an infinite matrix, including interface modes, is obtained from matching displacement and traction conditions at the interface. The condition for such modes is the vanishing of a determinant which depends on the phase velocity of the modes. Complex values of the velocity can also cause the determinant to vanish, and those velocities lying in the correct complex half plane, correspond to leaky normal modes whose amplitude decays exponentially with distance from the driven end of the rod.

The form of the determinant for radial modes (those whose displacement depends only on radius and longitudinal distance along the cylinder) is well-known (c.f. [3]). However, accurate computer codes for determining such modes may not exist.
We have developed new, accurate algorithms for calculating modified Bessel functions over the large complex parameter range needed for normal mode determination in the cylindrical geometry, and modified the asymptotic form of the determinant so that it remains analytic and of limited dynamic range throughout this region. Rouches' theorem for analytic functions then permits calculating the number of zeros inside a box-shaped region by use of an adaptive integration scheme. Combined with Newton's method for obtaining roots and several search routines, the phase velocity for all useful ordinary and leaky radial modes has been mapped out over a range of frequency and cylinder geometry for a sample consisting of a stainless steel rod encased by an aluminum matrix. The phase velocity depends on a curvature frequency parameter (f.R) which is valid for all fiber radii and applied frequencies down to the atomic scale until the elastic hypothesis no longer applies. Two leaky radial interface modes have been theoretically predicted for a steel rod embedded in an aluminum matrix. In one case the mode appears to be an attenuated shear wave concentrated in the aluminum. Its damping increases rapidly with increasing curvature so that, at curvatures where the velocity deviates significantly from the shear velocity, the mode is undetectable by ultrasonic means (assuming that the measurable attenuation range is \(-80\)db).

This wave was calculated and measured [3] for a cylindrical aluminum steel interface at 10 MHz for \(R = 8.75\) mm.

The model sample was composed of a 3.2-mm radius 316 stainless steel rod embedded in a 2024 aluminum alloy cylinder. The rod was attached to the aluminum by shrink fitting. This process created a good cylindrical interface between the two materials. Interface waves were generated by the conversion of surface waves propagated along the rod, although many other methods and techniques were investigated. The interface wave velocities measured as a function of frequency for the model sample are compared with the theoretical solution in Figure 1 for frequencies of 1, 3, 6, 11 and 15 MHz. The accuracy of measurements was \(30\) m/s. Excellent agreement with theory was observed. The other results (triangles) shown in Figure 2 were obtained on a cast-metal matrix composite model which contained a number of rods of radius 1.195 mm spaced 4 mm apart in an aluminum matrix. In this cast sample there are small flaws at the interface (Figure 2). These separations at the interfaces are thought to be the origin of the slight deviation of the experimental results from the theoretical predictions at higher frequencies. A much larger deviation from the predicted behavior occurs at low frequency when the wavelength becomes comparable with the spacing between the fiber-model rods and interaction occurs between neighboring interfaces.
Figure 1. There is good agreement between theory curves and experiment for a good interface obtained by a shrink fit (circles). As expected, a poor interface (triangles) results in deviation between theory and experiment.

Figure 2. Cast metal matrix composite model. Visible interfaces between aluminum and steel.

In conclusion, very good agreement was obtained between the measured and theoretically predicted interface velocities in the aluminum steel model material. Deviations from theory may serve to characterize the quality of the interface. We are now examining the displacement wavefields of these waves as a prelude to addressing the inverse (elastic constants determination) problem.
This work has been performed with partial support from the Office of Naval Research.

References:


**NDE STANDARDS AND METHODS**

When the Nondestructive Evaluation Program was established at the National Bureau of Standards in 1975, its goal was to upgrade the reliability and the reproducibility of those NDE methods that are widely used in American industry. The approach was research that would provide a scientific understanding of the NDE methods, and permit the development of measurement standards and a system of traceability for NDE measurements. The principal use of NDE at that time (and even today) was for in-service inspection.

Considerable progress toward this goal was achieved over the past decade and, accordingly, the Program's objectives have been broadened. Major programmatic activities are now directed toward developments in NDE which will facilitate its use in metal processing, manufacturing, and other production processes. Nevertheless, the original goal of the Program is still very valid. Based on continued need and importance, the development of measurement standards and a system of traceability for NDE measurements is still one of the Program’s four basic activities and, indeed, it is the central activity through which the others are related.

The objective of the NDE 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 which are traceable to national standards. In FY 1986, projects to develop new and improved measurement standards were carried out for eddy currents, x-ray radiography, leak testing, thermography, and fluorescent liquid penetrants. Work to develop new and improved measurement techniques was carried out for eddy currents, thermal waves, and acoustic methods, and a new effort on real-time radiology was initiated in collaboration with Iowa State University. Many significant accomplishments were recorded. A few examples are listed here:

- New prototype standards for evaluating eddy current probes were developed. One of these is an unflawed metal artifact which contains regions of different, but well characterized, conductivity. This standard will probably lead to a simple, fieldworthy technique for qualifying probes. Another artifact forms the basis for a laboratory procedure for calibrating probes. This artifact, which incorporates a radially symmetric, artificial defect in a plate, produces an impedance change in a probe. The magnetic field strength of the probe per unit current—which must be known in order to assess flaw sizes--may be calculated from the impedance change.

- New standards have been developed for measuring the quality of radiographic images produced at very low and very high x-ray energies. These standards are being evaluated in the public sector and are expected to supplement a standard developed previously for moderate energy levels. These standards permit the various parameters involved in the radiographic process to be assessed in terms of their individual influences on image quality.

- The framework for an NBS calibration service for leak measurements was established. Construction and verification of a primary leak standard were completed, and robust procedures for characterizing transfer standards (so-called "calibrated leaks") were developed. A special test service was initiated for helium diffusion leaks in the range of $10^{-8}$ to
10^{-11}\ \text{mol/s} (2 x 10^{-4} \text{ to } 2 x 10^{-7}\ \text{atm} \cdot \text{cm}^3/s).\ \text{Announcement of a formal calibration service awaits completion of detailed quality control documentation.}

In the brief, project reports that follow, two reports are included on projects that were supported by other agencies. These, it will be noted, are very closely related to the objectives of the NDE Standards and Methods activity.

**Eddy Current Coil Characterization**

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Center for Electronics and Electrical Engineering

The major goal of this program is the investigation of methods for characterization of eddy current test coils. Three approaches are under investigation. The first is the determination of electrical parameters, including a study of known defective coils; the second is the application of magnetic field mapping techniques to the various coils and probes; and the third is the investigation of the use of artifact standards coupled with precision electronic measurements for coil evaluation. A secondary goal is an investigation of the application of high sensitivity flux detection using superconducting quantum interference devices (SQUIDs) to flaw detection problems.

Last year we developed the basic systems for the various measurements now being used for probe evaluation. Foremost among these were the electrical parameter characterization and field mapping instrumentation. Last year's results led us in the relatively new direction of some unique artifact standards. Last year's work also convinced us that, for probes of importance to commercial NDE, the SQUID field mapping system was not superior to one based on a sensitive lock-in amplifier which is much easier to operate.

Our studies on electrical parameter characterization have encompassed a number of different coil systems and made use of a large number of commercial coils, both good and defective, acquired as a result of our interactions with various military organizations. The major study was on the variation of electrical parameters among ostensibly identical probes [1]. This work is directly tied to our effort to develop a military standard for eddy current probes [2]. The main outcome of this work is a field test method for probe characterization that uses unflawed metal artifact standards of varying resistivity. Work has also been completed on our studies of differential-coil eddy current probes [3].

Further development of field mapping instrumentation has been completed including a software system that gives an informative display of the field configuration of the coil. A sample plot is shown in Figure 1. Field plots have been made on a large number of commercial coils and on several specialty coils developed in the laboratory, and these results were related to electrical and mechanical probe parameters [4].

A SQUID-based system for flaw detection-at-a-distance studies is under construction. The concept is based on our earlier work using the SQUID for field mapping [5] and in conventional flaw detection configurations [6].
In a cooperative venture with the Fracture and Deformation Division, we have jointly developed and produced a number of uniform field probes for use in their flaw detection studies. We have characterized these probes both by measuring electrical parameters and by making three-dimensional field maps [7]. These probes were then successfully used in flaw detection experiments [8].

![Field map of commercial bolt-hole probe showing magnitude and direction of field across probe face.](image)

Figure 1. Field map of commercial bolt-hole probe showing magnitude and direction of field across probe face.

References:


Measurement Methods, Calibration Procedures, and Standards for Quantitative Eddy Current NDE

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Institute for Materials Science and Engineering

B. A. Auld and S. Jefferies
Edward L. Ginzton Laboratory
Stanford University

The major objective of this program is the development of measurement methods, calibration procedures, and standards for quantitative eddy current NDE. Experimental measurements at NBS Boulder are carried out in close collaboration with theoretical studies at Stanford University.

In the past we compared experimental eddy current measurements with the predictions of theoretical models of the interaction of nonuniform probe fields with surface-connected cracks [1,2]. This year we extended these studies to reflection probes. Eddy current reflection probes are two-port devices with an excitation coil surrounding a pair of oppositely phased pickup coils. A general $\Delta Z$ theory for this type of probe was developed at Stanford last year for application to new types of electromagnetic robotic proximity sensors [3]. In our study this year, this theory was applied to the characterization of surface flaws. A special air-core eddy-current reflection probe was constructed at NBS Boulder and used to obtain flaw signals for a number of rectangular-shaped EDM notches in aluminum. An automatic network analyzer was used to measure flaw signals as the probe was scanned over the length of the flaw. Experimental results showed excellent agreement with theoretical predictions [4].

Last year, we devised a new calibration method for uniform-field eddy current (UFEC) probes that consists of measuring the impedance change $\Delta Z$ induced in the probe by a cylindrical recess in the surface of a specimen of known
conductivity. From this measurement, the magnetic field strength per unit current H/I can be deduced, a quantity that enters into any calculation of flaw signals for the probe. This year, we tested this method more thoroughly and have identified some difficulties in applying the method to low-conductivity materials like titanium. The equation relating $\Delta Z$ to H/I is

$$\Delta Z = \frac{i(H/I)^2}{(2/\sigma)(Ad/\delta^2)}$$ (1)

where $\sigma$ is conductivity, $\delta$ is skin depth, $A$ is surface area, and $d$ is depth of the cylinder. Equation (1) was derived in the limit of small skin depth ($d/\delta > 2$) using Slater's microwave perturbation theory applied to a smooth depression in a perfect conductor. When using low conductivity materials, it is necessary to make the cylinder relatively deep in order to exceed the skin depth, but this in turn invalidates the assumption of a smooth depression in the surface. Measurements on a series of recesses in titanium have shown that Eq.(1) underestimates the field strength by a factor of two. Further studies are underway using air core probes, for which H/I can be accurately calculated, in order to verify this finding.

A new project was begun this year with funding from the Air Force to evaluate the use of uniform field eddy current probes to characterize surface-connected flaws. Our colleagues in the Electromagnetic Technology Division have collaborated with us in designing and characterizing the probes for this work [5], and an extensive series of flaw measurements were made in order to evaluate the reliability of the method in determining flaw sizes [6].

References:


Development of Leak Standards and Calibration Facilities at NBS

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Center for Basic Standards

A leak standards project was initiated at NBS in response to industry requirements for both improved leak measurement accuracy and, in some cases, legal traceability to a recognized standards authority. The project objectives were to develop a primary leak standard, characterize transfer leaks, and provide a publicly accessible leak calibration service. We have met the first two of these objectives on a limited basis, and are establishing a special test service* for helium diffusion leaks in the range 10^{-8} to 10^{-11} mol/s (2 \times 10^{-4} to 2 \times 10^{-7} atm. cm^3/s @ 0°C).

The capabilities of the NBS primary leak standard were explored extensively this year, particularly in the low flow rate regime of 10^{-9} to 10^{-12} mol/s for helium. A schematic diagram of this standard is presented in Figure 1. The pressure indication "P" in the vacuum chamber, caused by gas flow from the leak, is matched by flow from the flowmeter. The flow rate is then measured using the flowmeter. A new bellows flowmeter has been used extensively and compared with the original piston flowmeter whenever possible. Performance has been evaluated for the two flowmeters; this information is being prepared for publication in an archival journal [1]; a summary error budget is given in Table 1. A new vacuum chamber was added to the system this year, as were several residual gas analyzers (RGAs). The RGAs are used to measure not only the total pressure "P" in the vacuum chamber, but also the partial pressure of the gas of interest, avoiding background gas complications. The stability and repeatability of these RGAs are also under investigation.

The results of initial transfer leak characterizations and comparisons with Sandia National Laboratories were published [2]. Considerable effort was devoted to developing and measuring fixed-reservoir helium permeation leaks, which have been found to have superior stability over other types of artifacts. A leak comparator system, which will be used to perform the majority of the customer special tests, has been used extensively to measure both the temperature and pressure response characteristics of permeation leaks.

The first two special test reports were issued for NBS-owned fixed-reservoir helium permeation leaks, based on measurements performed on the primary leak standard over a six-month interval. These artifacts were subsequently used in the evaluation of nuclear fuel shipment casks. The comparison program with the Primary Standards Laboratory at Sandia is continuing with a 10^{-12} mol/s range helium permeation leak. A leak measurement workshop at Sandia provided a forum for presenting some of the earlier comparison data, as well as for discussing the topic of the most appropriate leak rate units for high accuracy work [3].

Future plans for the leak standards project include completing the documentation required for calibration service status, extending the capabilities to lower (and possibly higher) values of leak rate and including other

* A special test service involves a measurement or calibration method which is still being perfected and for which all the quality control documentation has yet to be completed.
gases in the transfer leak characterization program. Long-range plans include the development of a second primary standard of a different type, as well as investigations into novel measurement techniques.

The support of Richard Hyland, Fred Long, and Donald Martin in this work is gratefully acknowledged.

References:


Figure 1. Schematic diagram of the NBS primary leak standard.
Table 1. Measurement uncertainties of the piston flowmeter (in percent). The uncertainties of the bellows flowmeter are the same except at the lowest tabulated flowrate, where they are marginally smaller. Random errors are evaluated at three times the standard deviations or maximum observed deviations.

<table>
<thead>
<tr>
<th>Flow Rate (mol/s) and Piston Size</th>
<th>10^{-6} to 10^{-9}</th>
<th>10^{-10}</th>
<th>10^{-11}</th>
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<td></td>
<td>1 inch</td>
<td>1 cm</td>
<td>1 cm</td>
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<td>0.50</td>
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<td>0.01</td>
<td>0.10</td>
</tr>
<tr>
<td>( \delta (AV)/AV )</td>
<td>Area Systematic</td>
<td>0.001</td>
<td>0.001</td>
</tr>
<tr>
<td>Length Systematic</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>( \delta T_1 / T_1 )</td>
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<td>0.003</td>
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<tr>
<td>( \delta (At)/At )</td>
<td>Random</td>
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<tr>
<td>( V_2 \delta P )</td>
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<td>( \Delta V P_1 )</td>
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<tr>
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<td>( \Delta V T_1 )</td>
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<tr>
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<td>2.10</td>
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Radiographic Image Quality Evaluation At Very Low And Very High Energies

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The measurement of image quality is a basic concern of industrial users of radiographic or radiologic imaging equipment because image quality relates directly to detection sensitivity for flaws or other discontinuities. A recently developed national standard (ASTM E 746 and NBS SRM 1844) permits each aspect of a total radiography system to be evaluated in terms of its effect on image quality. However, this standard was developed for use at 200 kV and cannot be applied at very low kilovoltage, such as is commonly used in composite material inspections, or in the megavoltage region, which is required for the inspection of thick-walled materials such as pressure vessels or armor plate. NBS is remedying this deficiency by developing suitable testing devices and formulating techniques for their evaluation.

At 30 kV, image quality is strongly dependent on the spectrum (beam quality) of the x-ray source. For field use, the desired spectrum is achieved by filtering the x-ray beam and then adjusting the voltage until the half-value layer (HVL) in a given material is a specified thickness. The harder portion of the spectrum (the high-energy end) is least affected by the filtration and is similar for all users. For our purposes, 1100 aluminum alloy was specified for both filtration and HVL. Outside laboratories (DuPont and Fuji) confirmed this prescription for beam quality. The proposed image quality device was modeled after SRM 1844 except that, because of the low energies involved, lucite is used for the image quality indicators (IQI) and the absorber. Note that while the voltage adjustment is made with aluminum the image quality indicator is fabricated from lucite. The principal laboratories participating in the round-robin evaluation of this device according to our protocol are Agfa Gevaert, DuPont, Eastman Kodak, Fuji, McDonnell-Douglas, and Monsanto.

Over the past two years a test device and an associated standard for measuring image quality at very high energies were developed and tested. On the basis of over 500 tests it is clear that the proposed device and standard does indeed detect small changes in image quality and can be used to evaluate the effect of film type and other parameters on image quality. The draft document, written by Dan Polansky and R. C. Placious, was reviewed by a task force of outside specialists in high-energy radiography and, following several suggested changes, will be submitted for ASTM peer review and acceptance. The outside laboratories cooperating in this work on a high-energy image quality standard include Combustion Engineering, Babcock & Wilcox, X-Ray Industries, and Lynchburg Foundry.

In a related activity, we have supplied several other laboratories with beam attenuation data in copper at 50 kV intervals from 50 to 300 kV. The objective is a means for voltage calibration for users of x-ray equipment. Copper is a logical material to use for this because of ready availability in high-purity form and convenient thicknesses. Furthermore, the attenuation cross
sections for copper in this energy range vary over a factor of 20 or so, which causes the normalized attenuation curves to have different slopes for each of these energies. We are now awaiting attenuation measurements in copper by our collaborating laboratories, which include Lawrence-Livermore, Monsanto, Agfa, Eastman Kodak, and X-Ray Industries.

**Standard Test Methods for Characterizing Performance of Thermal Imaging Systems**

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Infrared thermography is the process whereby the thermal radiation from an object, invisible to the unaided eye, is remotely detected and rendered into a visible image of varying contrast. All motion, mechanical and electronic, generates heat; excessive or imbalanced heating and hot spots in an equipment, device, or facility generally forebodes failure. Thus, for decades, industry has routinely used thermography for nondestructive evaluation in a wide variety of applications; e.g., monitoring electrical transformers, motors, bearings, and tube furnaces, and for testing printed circuit boards. Further, subsurface defects, such as corrosion pits and delamination, may be investigated with the aid of an external heat source. Notwithstanding, the past few years have been witness to an explosive growth in new and sophisticated applications of thermography, brought about, on the one hand, by newly available digitized imagers with data storage, computational, and temperature measurement capabilities, and new emphases on automation, quality assurance, and reliability, on the other. Thus, thermography is now being successfully used for design verification, quality assurance testing, in-process monitoring and control, and accurate temperature determinations.

These new, important roles of thermography underscore the vital importance of standards for characterizing the performance of thermal imagers. Performance standards make possible instrumental calibration, monitoring, and intercomparisons at the marketplace and in the laboratory; further, the data may serve as a guide to experimental design and feasibility studies.

The objective of this project is to develop new standard test methods for characterizing performance of thermal imaging systems. Three performance measures, which are those of greatest value to the user, are to be addressed sequentially. These are (1) minimum resolvable temperature difference (MRTD), (2) minimum detectable temperature difference (MDTD), and (3) noise equivalent temperature difference (NEAT). MRTD and MTD relate to imagery, while NEAT is a measure of the temperature sensitivity of the instrument.

Thus far, standards work on this project has dealt solely with MRTD, which is a measure of the compound system-observer ability to spatially resolve temperature differences in a standard test pattern (target) by observing its display on a monochrome video monitor; hence, it relates to the capacity to distinguish details in imagery. Figure 1 depicts examples of standard blackbody bar targets of specified geometry and temperature. With fixed $T_b < T$, MRTD is the threshold $\Delta T = T - T_b$ at which the observer can just distinguish the four bars.
Plainly, MRTD is a function of the linear period of the bars and the observation distance. Details of the test method for measuring MRTD of thermal imaging systems used for NDT work are given in a new draft standard which is currently in the ASTM approval process and early adoption is expected.

\[ T \] is the temperature of a bar, \( T_B \) is the background temperature, and \( T > T_B \).

Figure 1. Targets used for MRTD determinations. The target is a periodic 4-bar chart of aspect ratio (width:length) 1:7. \( T \) is the temperature of a bar, \( T_B \) is the background temperature, and \( T > T_B \).

**Thermal Waves Visualized by Holographic Interferometry**

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A technique was developed for using optical holographic interferometry to investigate the propagation of long thermal waves through metal objects. These waves were created when the surfaces of thin aluminum plates were subjected to a slowly modulated heat source. In this manner surface deformations large enough to be observed by holographic interferometry were induced. A stroboscopic technique was used to visualize the radial propagation of the thermal wave. Predicted variations of fringe properties, such as visibility and density, were verified experimentally. The measured out-of-plane deflections agree quite well with the analytical thermoelastic solution.

A scheme for detection of large flaws, based on this combined technique, was also developed. Preliminary results showed that flaw detection can be improved when thermal waves, rather than unmodulated heating, are used in
holographic nondestructive testing. Increasing the modulation frequency is desirable. However, as frequency is increased, either more intense heating or increased interferometric sensitivity is required. From the perspective of thermal waves, the technique is interesting in that it produces an analog whole-field output which is conceptually equivalent to parallel processing of the entire image rather than to forming it by point-by-point scanning.

Theoretical Investigation of Acoustic and Thermal Wave Scattering

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A spatial moment representation of acoustic wave scattering was previously developed for both transient and steady state conditions. This formulation, which employs the Born approximation, provides a clear way of understanding how information on the size and shape of isolated scatterers is contained in scattered acoustic signals. During the past year, this formulation was used to help understand the reasons for the apparent success of "engineering" approaches, which use the Born approximation for acoustic wavelengths and scatterer dimensions for which it is known to be inapplicable.

The utility of the moment formulation for the acoustic case encouraged an attempt to make a similar formulation for the thermal wave case. Two physical situations were examined for which analytical solutions exist. These were the scattering of a thermal wave by a horizontal, penny-shaped crack, and the reflection of a thermal wave by a layer under the surface to which the thermal input is applied. While an expansion in powers of the frequency of the source can be made, the desired information on the position, shape, etc., of the scatterer is not easily extracted in this way. This is due in large part to the critical damping of the input signal. It appears that the moment approach to thermal waves is not likely to be a useful one.

Determination of Green's Functions and Their Application to Acoustic Emission and Ultrasonics

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Common to both acoustic emission and ultrasonics, the central theory in need is an accurate quantitative description of the generation, propagation, interaction with boundaries, and detection of the transient stress waves in a solid. Our approach has been based upon the concept of a generalized Green's function representation. Our objective is to determine the Green's functions of a solid of arbitrary shape and material properties, either theoretically or experimentally, or both if possible. Once the Green's function is known for a particular specimen, general test results can be predicted by simple integration. Numerical simulation of acoustic emission and ultrasonic tests then can be carried out to provide insight or to help interpret the test results.
Last year we reported the formulation and development of procedures for the experimental determination of Green's function. The concept involves two novel ideas: (1) the theoretical formulation of a probing waveform which facilitates the deconvolution process; (2) the design and construction of a system, a transducer pair together with high speed digital to analog conversion and power amplifier, which introduces a mechanical pulse of specified waveform and detects the propagated transient wave without loss of fidelity. Feasibility test results were encouraging.

This year we made progress in improving the design and computational procedure for the experimental determination of the Green's functions. The computational algorithm has been successfully transferred from the main NBS computer to local laboratory minicomputers. Consequently, experimentally acquired data can be processed in the laboratory without tedious digital data transfer. Recently we annotated and made portable our computer program to compute a large number of the Green's functions for an infinite plate [1]. The program now runs on IBM PC or compatibles rather efficiently. It is made available to the public mainly for its possible use for acoustic emission transducer calibrations. However, we have found many practical applications of the theoretically computed Green's functions in the development of various techniques related to ultrasonic testing of materials. We summarize these applications.

(1) Ultrasonic Receiver Calibration--Development of Hi-Fi Transducers--The ability to properly characterize and calibrate transducers provides the only means to compare, select, and develop new transducers. The computed Green's function serves as an ideal reference standard.

(2) Impact-Echo Technique for Flaw Detection--A point-source point-receiver pulse-echo technique to locate flaws within hardened concrete was developed at NBS [2]. A large concrete slab was cast with a known internal flaw. Steel balls of selected sizes were dropped onto the slab as a point impact source and an NBS conical displacement transducer was used as a point-receiver. The numerical convolution of impact force-time history and the Green's function (G33) were used to help interpret experimental signals. This impact-echo technique with the help of the plate Green's function should be useful for testing massive or high loss composite materials.

(3) Novel UT Measurement Technique--Self Calibration of Thickness Measurement--Shear Wave Speed Without Using a Shear Transducer--The normal displacement at one point due to point impact at another point has successive sharp arrivals which correspond to reflected and mode-converted rays. The time intervals between various arrivals provide the time-of-flight information from which either path length or wave speeds can be derived. Shown in Figure 1 is a simulated time record. It is generated by using the Green's function (G33) assuming the source and the detector are separated one plate thickness apart. As shown in the figure, the arrivals of reflected waves P\textsuperscript{2}, PS, P\textsuperscript{s}, etc., all can be detected. The corresponding wave speeds of interest can then be computed by solving simple geometric ray path problems. Note that the arrivals of various rays have different peak amplitudes. The numerical simulation based upon Green's function computation thus provides an easy method to optimize the test configuration for specific applications.
Modeling for Optical/Thermal Sources of Ultrasound and Distributed Receivers.--The Green's functions provide a basic tool for evaluating point and distributed thermo-mechanical sources and point and distributed receivers. For distributed sources and receivers the input or output must be integrated over the area of distribution to obtain a prediction of test results. This extension of the application of Green's functions has been formulated and will be compared with measurements using a distributed receiver in the form of a circular aperture capacitance receiver (Breckenridge, NBS) and with measurements of displacement at a point due to a laser source distributed over an annular ring on the surface of a plate [3]. For point laser generation of ultrasound, the Green's functions for a normal source or dipole source are directly applicable. Buried normal force and dipole sources were simulated by a laser pulse incident on the bottom of a small slot in a plate with and without an oil drop. Normal displacement off epicenter was detected using an interferometer. The theoretical results obtained by convolving the appropriate Green's function with the assumed source-time compare well with the measured displacement-time history shown in Figure 2 [4].

![Graph](image_url)

**Figure 1.** Simulated displacement versus time record for point impact generated transient. Detector and source are on the same side of the plate at one thickness apart.
Figure 2. Comparison of experimental displacement waveforms at $x = 0.5h$ to theoretical predictions of wave propagation theory. (a) Horizontal dipole with step-like time dependence; (b) Normal force with pulse-like time dependence.

References:


Performance Assessment of Fluorescence Measurements

K. L. Eckerle, J. J. Hsia, C. L. Chang, and K. D. Mielenz
Radiometric Physics Division
Center for Radiation Research

Surface discontinuities in solid materials can be detected by nondestructive evaluation using fluorescent liquid penetrants. The objective of this project is the performance assessment of fluorescence measurements made on these penetrants. This is possible since quantitative measurements may be made of fluorescence brightness by using instruments simulating human eye responsivity. Our approach has been to develop hardware and software for a reference spectrofluorimeter. This spectrofluorimeter is a two-monochromator instrument which allows mathematical simulation of various spectral distributions for sources, detection systems, and human eye response.

Work this year included the development and study of novel material samples which could serve as transfer standards for fluorescence measurements. The preliminary method of production is described elsewhere [1]. The samples were sintered mixtures of inorganic phosphors and polytetrafluoroethylene resin. Four samples, in colors blue, green, yellow, and orange, were manufactured. Preliminary studies on these samples were performed for stability of emission spectra to ultraviolet light exposure, uniformity, temperature, and concentration. All measured spectra were corrected for the relative spectral responsivity of the detection system. Only two, the yellow and the blue, that can be excited with wavelengths near 365 nm, are suitable for possible use for performance assessment of fluorescence measurements.

We have suggested standardizing ultraviolet irradiance meters for testing irradiance levels and fading. A definition of relative response of these ultraviolet meters would be a step toward standardization. To this end, Jack Hsia has initiated coordination efforts of ASTM Committee E-7, UV meter manufacturers, international subcommittee on UV meters (with its chairman at BAM in Germany), and the NBS.

References:


Military Standards for Nondestructive Evaluation

L. Mordfin
Office of Nondestructive Evaluation
Institute for Materials Science and Engineering

Under a long-term contract with the Army Materials Technology Laboratory, engineers and scientists in the NDE Program have been developing new and improved military standards for NDE. Among the project's accomplishments since its inception were completion of two documents which were approved following full tri-service coordination in the Department of Defense and by relevant industrial contractors.
Glossary of Terms and Definitions for Acoustic Emission Testing Procedures, MIL-STD-1945 (developed by D. G. Eitzen, Automated Production Technology Division, Center for Manufacturing Engineering).

Inspection, Magnetic Particle, MIL-STD-1949 (developed by L. J. Swartzendruber, Metallurgy Division, Institute for Materials Science and Engineering).

During the past year E. H. Eisenhower (Office of Radiation Measurement, Center for Radiation Research) completed a third draft glossary of terms for radiography. This draft is now undergoing the third and final DOD coordination.

Twelve master radiographs—which will be used to produce test patterns for evaluating the visual acuity of radiographic interpreters—were developed by R. C. Placious (Ionizing Radiation Division, Center for Radiation Research). The total set, which will comprise 76 patterns, is scheduled to be completed during the coming year.

A procedural document for the primary calibration of acoustic emission (AE) transducers, which was developed by D. G. Eitzen and F. R. Breckenridge (Automated Production Technology Division), was approved by ASTM as Standard Method E 1106-86. This is now being used by Eitzen and Breckenridge to formulate a military standard for the secondary calibration of AE transducers. A field calibration document, which will include an exposition of N. N. Hsu’s award-winning pencil source, is being developed jointly by NBS and the Physical Acoustics Corporation.

T. E. Capobianco and F. R. Fickett (Electromagnetic Technology Division, Center for Electronics and Electrical Engineering) are developing a standard practice, suitable for use in the field, for qualifying eddy current coils. A preliminary version of the standard was presented at the 35th Defense Conference on Nondestructive Testing in October 1986.

The first draft of a proposed standard on NDE of corrosion of ammunition was prepared by A. C. Fraker and A. W. Ruff (Metallurgy Division, Institute for Materials Science and Engineering). The draft was coordinated through DOD and revisions are now in progress.
NDE of Shipping Casks for Spent Nuclear Fuel

D. G. Eitzen, Automated Production Technology Division
Center for Manufacturing Engineering

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

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

L. Mordfin, Office of Nondestructive Evaluation
Institute for Materials Science and Engineering

When spent fuel from nuclear reactors must be transported by rail or truck, Federal regulations require that it be enclosed in shipping casks that satisfy a number of stringent requirements. A new design configuration that is presently under consideration for such casks consists of metal cylinders approximately 17 ft. (5 m) long, 8 ft. (2.5 m) in diameter, with 14-in. (35-cm) thick walls. The casks are to be fabricated by casting with one integrally closed end. The materials being considered for this application are austenitic steel, ferritic steel, and nodular cast iron.

The thick walls are needed in order to absorb most of the radiation emitted by the contents. A much more demanding requirement is that the casks be capable of withstanding severe transportation accidents without a breach of the cask walls that would permit the escape of any radiation or radioactive fluid. In order to satisfy this requirement, it is necessary that the casks be inspectable to the extent that no potentially critical flaw in the cask walls may go undetected.

The National Bureau of Standards conducted a study for the Sandia National Laboratories to evaluate the inspectability of the casks. The study showed that current NDE technology is adequate for inspecting the casks provided that the inspection personnel are well trained in their respective methods and that they are experienced with the equipment and their specific techniques and have been properly qualified in this application.

The capabilities of many NDE techniques were evaluated in the study. These techniques were based upon all of the principal NDE methods in use today. For most of these methods the precise characteristics of the cask material are largely irrelevant, although certain general properties (e.g., magnetic versus non-magnetic) are important. For the ultrasonic methods, however, detailed specifications of the cask material are critical, mainly because they determine whether the ultrasonic beam can satisfactorily penetrate the thick sections of the cask.

Generally speaking, wrought ferritic steels do not pose significant obstacles to ultrasonic inspection. Austenitic steels, on the other hand, and particularly cast austenitic steels, are usually difficult, if not impossible, for ultrasonic NDE, particularly in large thicknesses, because of the large, anisotropic grain structure in the material. This problem can be alleviated, to some extent, by certain thermo-mechanical treatments. The ultrasonic
inspectability of nodular cast iron can range from impossible to very good depending upon the size, distribution and shape of the nodules, the grain structure, and the general cleanliness of the material. The key points are that the cask materials should be selected on the basis of a careful test program, and that the processing of the material and the manufacture of the casks should be rigorously controlled in order that it be satisfactorily inspectable by ultrasonic techniques.

Two general approaches are available for ultrasonic inspections of casks, one based upon conventional ultrasonic techniques and one upon advanced techniques. Conventional techniques (e.g., amplitude-based, manual, pulse-echo contact scanning in several directions) can reliably detect all discontinuities having reflectivities equivalent to that of a 10-mm (0.4-in.) flat-bottomed hole. While this does not mean that such techniques can detect every 10-mm flaw, they do serve as a general measure of the quality of the material. If a given quality level can be shown, say by mechanical tests on sample casks, to be acceptable, then such ultrasonic inspections can evaluate the acceptability of the casks relative to the given quality level.

Advanced ultrasonic inspection techniques (e.g., Amplituden und Laufzeit-Orts-Kurven, amplitude and transit-time dynamic curves (ALOK), synthetic aperture focusing technique (SAFT)) have been developed in recent years which employ transducer arrays or synthetic arrays and provide images of flaws. It is believed that a system based on one of these techniques can be developed that could reliably detect and evaluate critical cask flaws provided that inspectability is designed into the casks.

Radiology, like ultrasonics, is capable of detecting flaws in the interior of cask walls. Both film techniques and electronic imaging techniques are available today which can provide a high level of flaw detectability in this application. With properly selected equipment, very small cracks can be located if they are oriented parallel to the x-ray beam (perpendicular to the cask wall). The sensitivity is not as good for internal planar flaws oriented perpendicular to the x-ray beam (parallel to the cask wall). Substantially greater sensitivity in the thickness direction is obtainable by radiation gauging.

Surface methods of NDE are widely used in the casting industry because the most severe types of common casting imperfections are usually visible at the surface. Visual inspection is recommended for all castings and, when carried out by trained inspectors with knowledge of casting procedures and defects, is highly reliable except for very fine (hairline) cracks.

Magnetic particle inspection and liquid penetrant inspection are both capable of detecting extremely fine surface flaws under the proper conditions and with qualified personnel and inspection materials. Magnetic particle inspection is only useful for ferromagnetic materials, such as ferritic steel and nodular cast iron, but not austenitic steel. Excellent sensitivity could be obtained on cast ferritic steel, or on nodular iron having well-controlled nodule size at the surface and good surface finish.

Liquid penetrant inspection may be used on magnetic or non-magnetic materials and is at least as sensitive as magnetic particle inspection on clean surfaces. Cracks filled with oxide, oil, grease, or even water make the use of
penetrants difficult, whereas these contaminants do not usually affect magnetic particle inspection.

If a higher level of detectability is needed for surface flaws the eddy current method merits consideration. Used properly, eddy current techniques can detect minute surface flaws in austenitic steel. The high permeability of ferrite would be expected to cause problems with the other cask materials but, even so, the eddy current method would undoubtedly provide a higher level of reliability in the detection of surface cracks than either magnetic particle or liquid penetrant inspection. The principal deterrent to the use of the eddy current method here is that it has not been widely used in the casting industry. Therefore, its application to cast casks would require some preparatory development work and specialized training.
APPENDICES

A. NDE SEMINARS AT NBS

Dr. Richard L. Weaver, University of Illinois, Urbana, IL
"Acoustic Emission Source Characterization by Means of Diffuse Waves: Theory and Experiment"
October 10, 1985

Professor Paul Hoeller, Fraunhofer-Institute für zerstörungsfreie Prufverfahren, Saarbrucken, F.R. Germany
"Acoustic Emission Source Indentification by Signal Processing: Recent Progress in Nondestructive Structure and Stress Analysis"
October 23, 1985

Dr. Fereydoun Faridian, University College, London, England
"Acoustic Microscopy"
October 24, 1985

NDE Poster Session
December 4, 1985

Dr. Emanuel Segal, Technion-Israel Institute of Technology, Haifa, Israel
"NDE of Composites Using Computerized Tomography"
March 25, 1986

Dr. Spencer Bush, Review & Synthesis Associates, Richland, WA
"Ultrasonic Reliability: Some Recent Results"
September 16, 1986

B. INVITED TALKS BY ONDE STAFF


"The Role of NDE Sensors in Automated Materials Processing," H.T. Yolken, Texas A&M University, College Station, Texas, February 5, 1986.

"The Role of NDE Sensors in Automated Materials Processing," H.T. Yolken, University of Houston, Department of Mechanical Engineering, Houston, Texas, February 6, 1986.


"Optical Methods in NDE," G. Birnbaum, NBS Analytical Chemistry/Materials Science Workshop, Gaithersburg, Maryland, April 18, 1986.


C. PUBLICATIONS

Following is a partial listing of NBS reports and publications on NDE and related topics that have been issued since last year's Technical Activities report was prepared. A more complete compilation will be presented in ONDE's annual bibliography with abstracts.


D. AWARDS AND APPOINTMENTS

Department of Commerce Bronze Medals

The Bronze Medal Award is the highest honorary recognition available for Bureau presentation. Recipients during FY 1986 included Dr. Norman F. Berk of the Reactor Radiation Division and Dr. Kay A. Hardman-Rhyne of the Ceramics Division, who collaborated in the theoretical and experimental development of small-angle neutron scattering (SANS) as a reference measurement technique for nondestructive evaluation. Another recipient was Dr. Richard J. Fields of the Fracture and Deformation Division, who used the SANS technique to characterize low levels of distributed creep damage in metal specimens that had been exposed to elevated temperatures for extended periods while under load. Dr. Ronald F. Fleming of the Center for Analytical Chemistry was similarly honored for his significant contributions to the establishment of neutron depth profiling, another new materials characterization technique.

William Blum Award

This prestigious award of The Electrochemical Society was presented to Dr. Ugo Bertocci of the Metallurgy Division for his research into the relationship between electrochemical noise and electrode processes, notably the initiation of pitting corrosion in metals.
Dr. Nicholas J. Carino of the Center for Building Technology was awarded the Wason Medal for Materials Research by the American Concrete Institute in recognition of his significant contributions to the nondestructive testing of concrete and, particularly, the development of a pulse-echo technique for detecting flaws within concrete structures.

Dr. Jeffrey T. Fong of the Center for Applied Mathematics was elected a Fellow of the American Society of Mechanical Engineers. Dr. Fong, who recently edited a volume on NDE Reliability through Round-Robin Testing, was also named the new chairman of the 14,000-member ASME Pressure Vessel and Piping Division.

Dr. John R. Manning, Metallurgy Division, was named a Fellow of the American Society for Metals. Manning heads the Metallurgical Processing Group at NBS and is responsible for administering a major segment of the NDE Program's activity on rapidly solidified metal powder processing.

Dr. Lorretta J. Inglehart, a contract scientist in the Ceramics Division, was awarded a Fulbright Scholarship for study and lecturing in France.

Dr. Gerald V. Blessing of the Center for Manufacturing Engineering was named international program chairman for the November 1986 meeting of the Institute for Electrical and Electronics Engineers.

Dr. Donald G. Eitzen of the Center for Manufacturing Engineering was elected the new chairman of the subcommittee on acoustic NDT methods (TC 135/SC 3) in the International Organization for Standardization.

Dr. Theodore V. Vorburger of the Center for Manufacturing Engineering was elected chairman of Committee B46 on Classification and Designation of Surface Qualities in the American Society of Mechanical Engineers. During March 1986, Dr. Vorburger served as a specialist for the World Bank Chinese University Development Project, lecturing and consulting at Chongquing University on optical inspection methods.

Dr. Klaus D. Mielenz of the Center for Radiation Research was appointed director of Division 2 on Physical Measurement of Light and Radiation in the International Commission on Illumination.

Dr. Jack J. Hsia of the Center for Radiation Research was elected to the board of directors of the Inter-Society Color Council.
### E. **Principal Investigators**

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| Berlinsky, Yoram      | Acoustic emission  
                        | Wave propagation and instrumentation                                        |
| Biancaniello, Francis S. | Metals processing                                                        |
| Blessing, Gerald V.   | Ultrasonics  
                        | Experimental physical acoustics                                             |
| Breckenridge, Franklin R. | Acoustic emission and transducers  
                        | Transducer calibration                                                      |
| Capobianco, Thomas E. | Eddy current NDE  
                        | Electromagnetic technology                                                  |
| Clark, Alfred V.      | Ultrasonics and eddy currents  
                        | Mechanical properties                                                       |
| Clough, Roger B.      | Acoustic emission  
                        | Mechanical Properties                                                       |
| Cohen, Julius         | Thermography  
                        | Fourier optics                                                              |
| Droney, Bernard       | Steel processing sensors  
                        | Metallurgy                                                                   |
| Eckerle, Kenneth L.   | Fluorescence  
                        | Spectrophotometry                                                           |
| Ehrlich, Charles D.   | Calibrated leaks and leak detection  
                        | Surface physics                                                             |
| Eitzen, Donald G.     | Acoustic emission  
                        | Engineering mechanics                                                       |
| Fanconi, Bruno M.     | Optical wave guide techniques  
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| Fick, Steven E.       | Specialized measurement  
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| Fickett, Frederick R. | Electromagnetic methods  
                        | Solid state physics, superconductivity, and magnetism                       |
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**Office of Nondestructive Evaluation, Technical Activities, 1986**

**H. Thomas Yolken**

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DEPARTMENT OF COMMERCE  
WASHINGTON, D.C. 20234

National Bureau of Standards  
Rt. 270 & Quince Orchard Road  
Gaithersburg, Maryland 20899

**A review of the Nondestructive Evaluation Program at NBS, for fiscal year 1986, is presented in this annual report.**

**acoustic emission; eddy currents; leak testing; magnetics; neutrons; nondestructive evaluation; optics; penetrants; radiography; standards; thermal testing; ultrasonics**

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