WADD TECHNICAL REPORT 61-91
PART I

ULTRASONIC METHODS FOR NONDESTRUCTIVE EVALUATION OF CERAMIC COATINGS

W. E. LAWRIE

ARMOUR RESEARCH FOUNDATION
OF
ILLINOIS INSTITUTE OF TECHNOLOGY

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FOREWORD

This report was prepared by the Armour Research Foundation of the Illinois Institute of Technology under USAF Contract No. AF 33(616)-6396. The contract was initiated under Project No. 7360 "Materials Analysis and Evaluation Techniques", Task No. 73606 "Non-Destructive Methods". The work was administered under the direction of Materials Laboratory, Directorate of Advanced Systems Technology, Wright Air Development Division, with Mr. Richard R. Rowand acting as project engineer.

This report describes investigations conducted during the period March 1, 1960 to February 29, 1961.

The ultrasonic investigations were conducted by W. E. Lawrie and K. E. Feith under the supervision of R. R. Whymark. Zirconium coated specimens were prepared under the direction of S. A. Bortz.

ABSTRACT

This report describes investigations into the use of ultrasonics to detect defects in ceramic-metal bonds and to measure the strengths of the bonds. In the techniques investigated ultrasonic frequencies from 30 cps to 35 mc/s have been used and in one method two frequencies are used simultaneously. Low frequency energy (14 kc/s) has been successfully used to detect defects by decrement measurements. Low frequencies have also been used in further studies of the intermodulation method used to locate regions of bonds in which defects are present. High frequencies, up to 35 mc/s, have been used with a transmission method and visual images of defects are displayed using a simple charge scanning technique. High frequency energy has also been used in the form of surface waves. (This work is a continuation of investigations reported in WADD TR 60-157.)

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

W. J. Trapp

W. J. TRAPP
Chief, Strength and Dynamics Branch
Metals and Ceramics Laboratory
Materials Central

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Section I

INTRODUCTION

Ceramic materials are frequently used to protect refractory metals from environments that might cause rapid failure of the substrate through corrosion and erosion effects. For a ceramic coating to provide adequate protection, the coating must be bonded to the metallic substrate with a strength sufficient to withstand the stresses developed by the environmental conditions. If defects exist anywhere in the coating-substrate system, the strength of the coating may be adversely affected. Premature failure of the coating would result in eventual destruction of the protected structure.

Successful use of ceramic coatings demands that the bond strength be known and that all defects be located and evaluated prior to the exposure of the coated surface to a destructive environment. At present no completely satisfactory method is available to test the coating. The objective of this program is to develop ultrasonic techniques to detect small defects in the bond and to measure the bond strength.

The investigations reported herein were conducted during the second year of the project. During the first year, many different techniques were investigated and some were discarded where the technique was obviously inapplicable. Of the methods investigated, the intermodulation, direct transmission and charge scanning, and surface wave techniques provided greatest promise of ultimate success. These techniques found most useful during the first year were the main basis for the second year's investigations.

During the first year, the majority of the specimens were composed of a zirconium oxide coating on mild steel. During the second year, greater emphasis was placed on molybdenum specimens coated with Chromalloy W2.

This report is divided into five main sections. In Section II, we enlarge upon the basic concepts of flaw detection in systems of the present type. The results of the first year's work are discussed in outline[1] and the utilization of these results and the underlying concepts

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[1] The results of the first year's work are discussed in detail in WADD TR 60-157.

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are discussed in connection with the present phase of activity. Section III is directed to detailed discussions of techniques and results for the charge scanning method. In Section IV, the use of surface waves is discussed. Further studies of the intermodulation method are reported in Section V. In Section VI, low frequency methods are described for detecting the presence of defects without indicating the location of the defects. The second year's work is summarized in Section VII and conclusions are listed, based on the experimental results.

Section II

BASIC CONCEPTS AND BACKGROUND INFORMATION

The use of ultrasonic energy provides many possible methods of detecting flaws in solids. All methods are based on the same concept of introducing ultrasonic energy into the specimen and measuring the modification produced in the sound field by the defect. Typical modifications of the sound field are the reflection of sound energy by a defect, the reduction of the transmitted energy, the change in phase with accompanying interference effects caused by transmission through a portion of the medium with a different sound velocity, the scattering of sound, or the generation of new vibrational modes by conversion at a surface of the defect. Shear waves, compressional waves or surface waves may be used under appropriate conditions. The criterion for the selection of the wave type and experimental technique is determined by the material, the size and shape of the test object, the size, shape, orientation and type of defect.

Normally frequencies between 1 and 10 mc/s are employed for flaw detection. Defects are usually detectable if the defect dimensions constitute an appreciable portion of a sound wavelength. The use of frequencies much below 1 mc/s produces acoustic wavelengths too long to provide adequate resolution. Frequencies higher than 10 mc/s produce wavelengths sufficiently short that parallelism of the transducers and the surfaces of the specimen becomes ciritical. In addition, sound attenuation increases rapidly with increasing frequency.

In the investigations conducted during the first year of the project, a wide variety of techniques were employed. Many of the techniques were discarded because of the properties of the ceramic coatings and the substrate. As an example, reflection methods were used in which the sound beam is directed at the defect and the reflected energy measured. The reflection methods were not considered suitable for use with a zirconia-mild steel bond because the reflection coefficient is very high and the presence of a defect makes only a small change in the total reflection coefficient. In general, transmission measurements appeared more promising. The transmission techniques consist basically of passing sonic energy through the defect region and measuring the amount of sound transmitted. However, the transmission measurements proved inadequate during the first year's work because changes in the transmission coefficient at the transducer-specimen interfaces masked changed caused by the defects. During the present work period many of these difficulties have been obviated using liquid immersed specimens.

One important aspect of the transmission technique is the measurement of the relative amplitudes of the waves transmitted through the specimen. Detailed research was carried out in this connection during the first year. In particular, the use of the entire area of a quartz crystal for detection was rejected because of a lack of sensitivity and resolution. Preliminary investigations showed that a charge scanning system offered a satisfactory method of obtaining better overall resolution. In this method, the potential at each point on the crystal is monitored, rather than just the integrated potential of the entire crystal. In this way, the effective detecting area of the crystal is greatly reduced and in fact is usually of the order of a few millimeters dimensions. The conclusions derived from results obtained using the charge scanning system were supplemented by Schlieren optical measurements of the transmitted sound field. In these latter measurements the resolution is limited purely by acoustical effects so that the Schlieren system gives the inherent acoustical resolution and allows discrimination against other effects.

Low frequency energy was used during the first year to determine the bond strength. Low frequency energy was also used simultaneously with high frequency energy in the intermodulation method. In this method, a defective bond is cyclically stressed and a high frequency sound beam, transmitted through the bond, is amplitude modulated at the stress frequency. The phenomenon of intermodulation was observed but insufficient data was available to provide an explanation of the phenomenon.

The investigations conducted during the second year were in a large part dictated by the results of the previous investigations. No further investigations of reflection techniques were made since the sensitivity was previously shown to be low. Transmission methods have been found promising but the need for increased resolution and for freedom from changing transmission coefficients at the specimen surfaces indicated that further development was required. This has received attention during the second year.

Similarly, the intermodulation phenomenon was not understood and further investigations of the mechanism were required. Low frequency methods were extended so that the presence of defects can be indicated by decrement measurements. The small attention paid to the use of surface waves during the first year produced results which merited further investigations during the second year.

Section III

DIRECT TRANSMISSION AND CHARGE SCANNING

In many conventional ultrasonic flaw detection systems, energy is introduced into the object under test and the defects are indicated by local modifications of the sound field. Such modifications may be spatial variations in the intensity of the transmitted wave, or the presence of a reflected wave. In a previous report,[2] it was shown that a high reflection coefficient occurs at the interface between mild steel and a zirconia coating and that the total reflection coefficient was largely independent of the presence of bond defects. The measurement of reflected energy then becomes an insensitive method of detecting defects as a zirconia-steel bond.

It was also found that transmission methods were capable of indicating unbonded regions of the interface. However, if transmitted ultrasonic energy is to be used to examine large specimens, methods must be found to keep the transmission coefficients constant at the transducer-specimen interfaces while the crystals are moved about during the inspection. If the intensity of the energy transmitted through the specimen is not constant, false indications of defects will be obtained. In addition, the use of the entire area of a large quartz crystal to detect the transmitted energy results in low sensitivity. The relative decrease in transmitted signal is approximately proportional to the ratio of the defect area to the crystal area. For small defects, the relative decrease in transmitted energy is small.

To obtain adequate sensitivity and resolution with an ultrasonic transmission technique, methods must be available to permit movement of the transducers over the specimen surface without spurious effects, and to detect small spatial variations in the intensity of the wave transmitted through the specimen. The method described below provides solutions to both problems.

EXPERIMENTAL METHOD AND RESULTS

The use of transmitted energy has been investigated using the

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[2] WADD Technical Report 60-157

jig shown in Fig. 1. This jig was constructed to hold the quartz crystal transducers and the specimen in precise parallelism. The slots in the block for mounting the plates are accurately ground so that one side surface of each slot is accurately parallel to similar surfaces in adjacent slots. Each of the plates used to hold transducers or specimens is also ground so that opposite surfaces, normal to the sound beam, are parallel. Measurements with a dial indicator show that the spacing between parallel faces or adjacent plates is constant within 0.0003 inches. The flat surface of the quartz crystals and the specimens are placed in contact with the ground surfaces of the plates, assuring that the crystals and specimen are mutually parallel. The high degree of parallelism between the receiving and transmitting crystals permits the use of frequencies up to 50 mc/s without phase variations in the incident sound beam causing cancellation effects.

The difficulty of obtaining repeatable coupling has been overcome by either immersing the jig in a liquid bath, or attaching side plates to form a container for the liquid as in Fig. 4. Either water or isopropyl alcohol is used as a couplant. The precision of the jig combined with the liquid couplant largely eliminates spurious effects in the transmission of sonic energy.

Two methods were used to improve the sensitivity of the direct transmission technique. Since the sensitivity depends upon the ratio of defect area to sound beam area, a reduction in the beam area should increase sensitivity. Rather than reducing the crystal diameter, a rotatable aperture was introduced between the transmitting crystal and the specimen. The aperture consisted of a thin air layer sealed between two layers of copper foil. A single circular hole through both layers of foil, with the edges of the hole again sealed, provided a passage for a small sound beam. The aperture adequately restricted the transmission of sound, but the flexible foil, distorted during the fabrication of the aperture, was no longer plane. Consequently multiple reflection of sound between the transmitting crystal and the aperture allowed sound to pass through the hole at angles to the crystal other than normal and thus excite a relatively broad region of the specimen. In addition, the distances between the aperture, specimen and receiving crystals were so large that resolution was lost through diffractive effects.

The second method of increasing sensitivity incorporated the charge scanning concept. The apparatus was modified to give the arrangement shown in Fig. 2. The space between the specimen and the receiving crystal has been reduced to minimize diffractive effects and provision has been made to scan the back surface of the receiving crystal with an electrical charge probe. The space between the specimen and the crystal is fixed by the thickness of the mounting plate, and provision is made to permit entry of the couplant into this space.

The first results were obtained using a manually moved

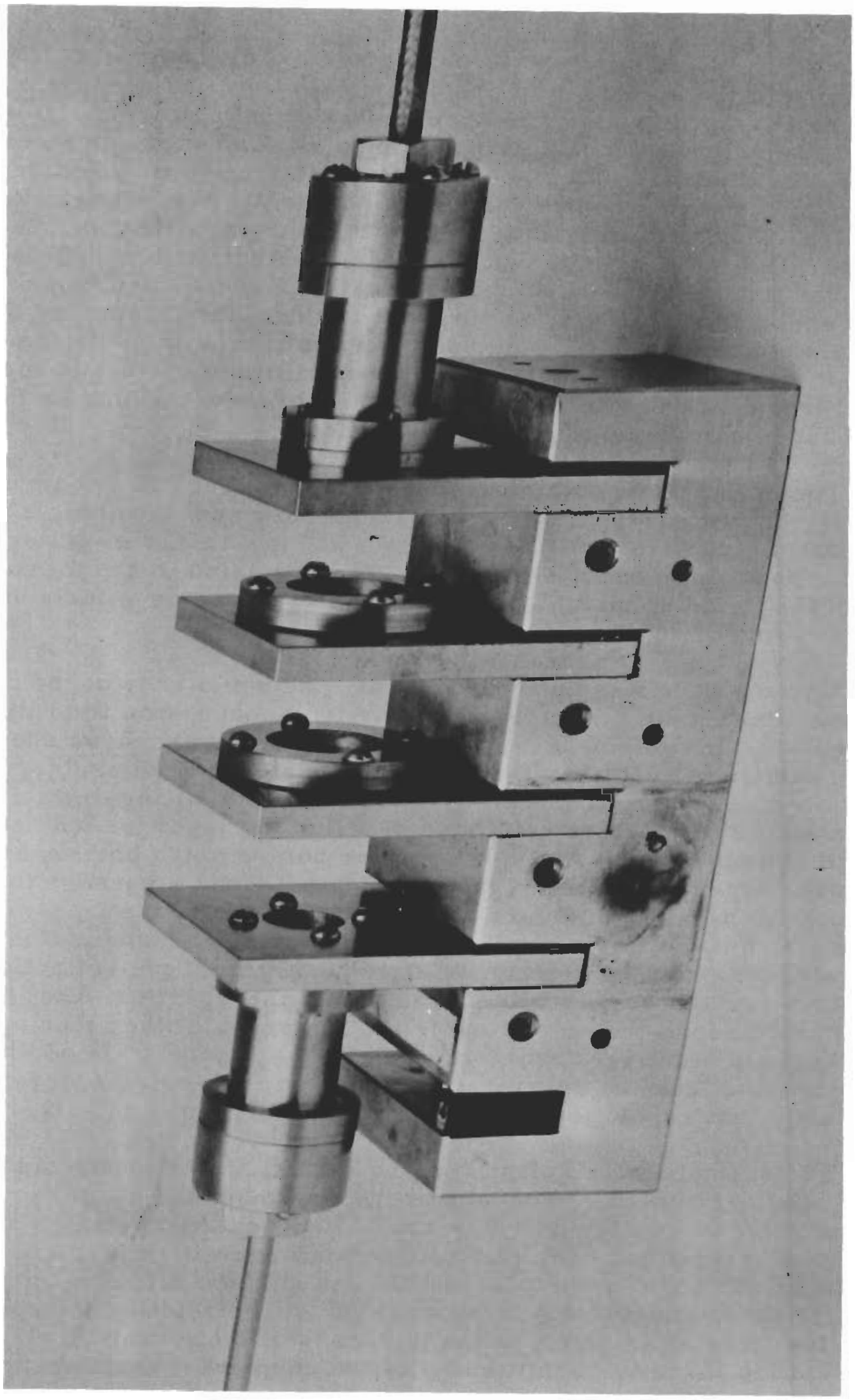

Fig. 1 - PRECISION JIG FOR HOLDING CRYSTAL TRANSDUCERS AND SPECIMENS PARALLEL FOR DIRECT TRANSMISSION MEASUREMENTS

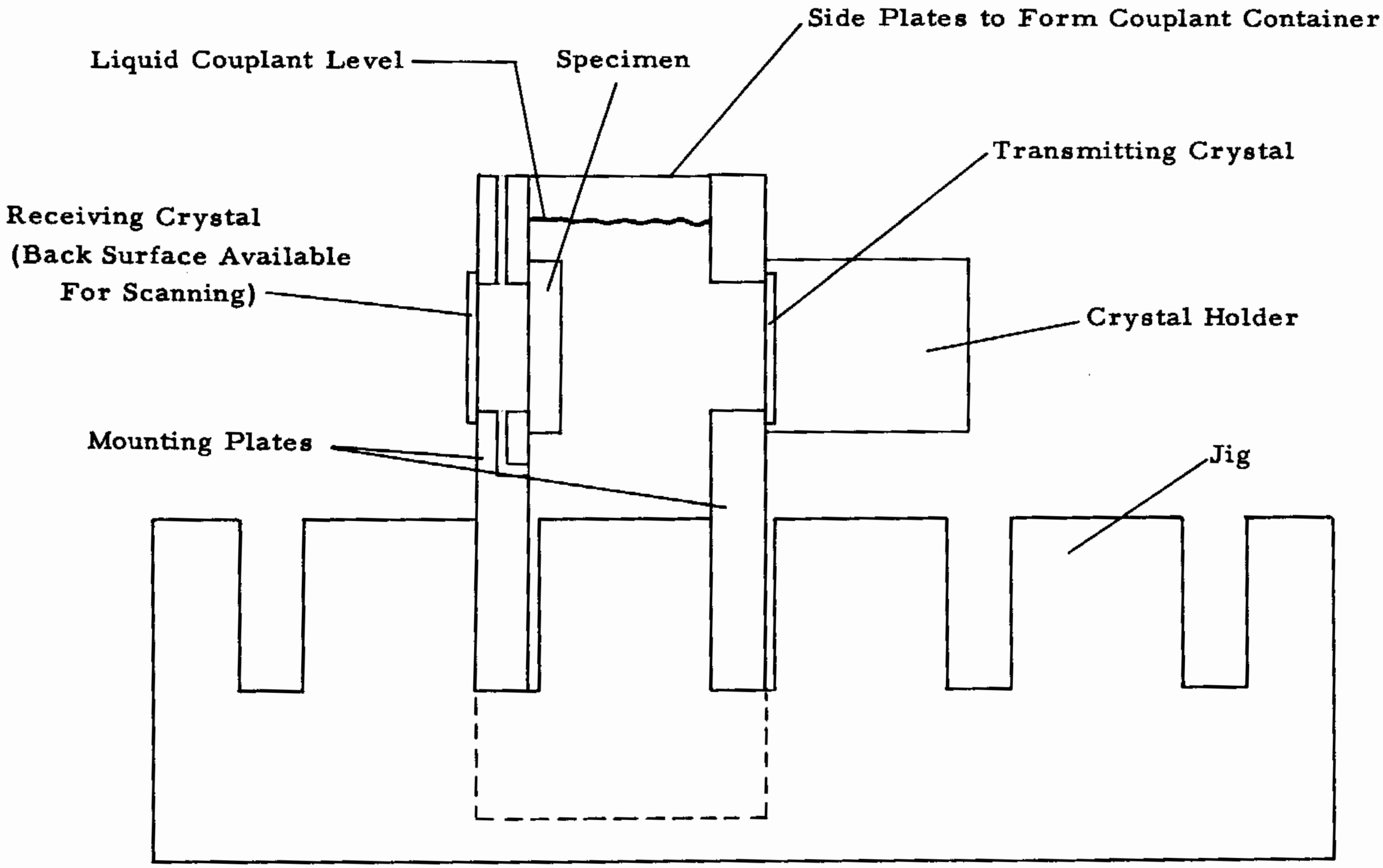

Fig. 2 - REVISED EXPERIMENTAL ARRANGEMENT FOR DIRECT TRANSMISSION MEASUREMENTS AND CHARGE SCANNING ON BACK SURFACE OF RECEIVING CRYSTAL

spring-loaded probe to detect a simulated defect 1/16-inch diameter by 0.003-inch thick. This defect was produced by drilling a 1/16-inch diameter hole through a steel disc. The hole was plugged with salt, and one surface of the disc flame sprayed with zirconia. The salt was then dissolved and the hole filled by a precision plug of length 0.003 inch less than the thickness of the disc. Scanning across a diameter of the receiving crystal provided a plot of probe voltage versus position similar to the lower curve of Fig. 3. Figure 3 shows the probe voltage-position plot for a defect 1/16-inch diameter by 0.016-inch thick. This latter defect was constructed by drilling a 1/16-inch hole through a steel plate, plugging the hole with a steel pin about 0.016 inch less in length than the thickness of the plate, and then cementing a small square of zirconium oxide over the small hole remaining in one surface. The upper curve shows the voltage measured at different positions on a diameter of the crystal with the specimen removed. The lower curve shows the voltages with the specimen in position. The decreased output at the center of the crystal indicates the presence of the defect. The results show that the distribution of charge on one surface of the crystal closely approximates the pressure distribution incident on the opposite surface After verifying that the charge scanning method was capable of resolving 1/16-inch diameter defects, a more sophisticated apparatus was developed to exploit the method. The apparatus consisted of a more elaborate mechanical scanning system, and simple electronic circuits to display the experimental data two-dimensionally on an oscilloscope.

The scanning system employs a modified microscope stage to position a spring loaded probe at any point on the crystal. A typical application of the scanning system is shown in Fig. 4. The apparatus is being used to scan the back surface of a crystal clamped to one wall of a liquid-tight enclosure formed in the test jig as discussed previously. The probe, crystal, and clamp are clearly visible in the photograph. A potentiometer is coupled to one of the positioning knobs on the microphone stage assembly. This potentiometer is used to develop a dc voltage proportional to vertical position of the probe, which is used to provide vertical deflection of the beam of a cathode ray tube. This arrangement provides semi-automatic scanning along a vertical line on the crystal. By scanning many vertical lines, each time manually readjusting the horizontal position of both the probe and the oscilloscope beam, the entire area of the crystal may be scanned. Although more elegant systems could be devised to provide two-dimensional charge scanning, the simple system described is adequate to demonstrate the merits of scanning when combined with the direct transmission of sonic energy.

Two display systems have been used, both of which depend in part upon the scaning system. In each case, the electron beam of a cathode ray oscilloscope is moved in sympathy with the probe and in

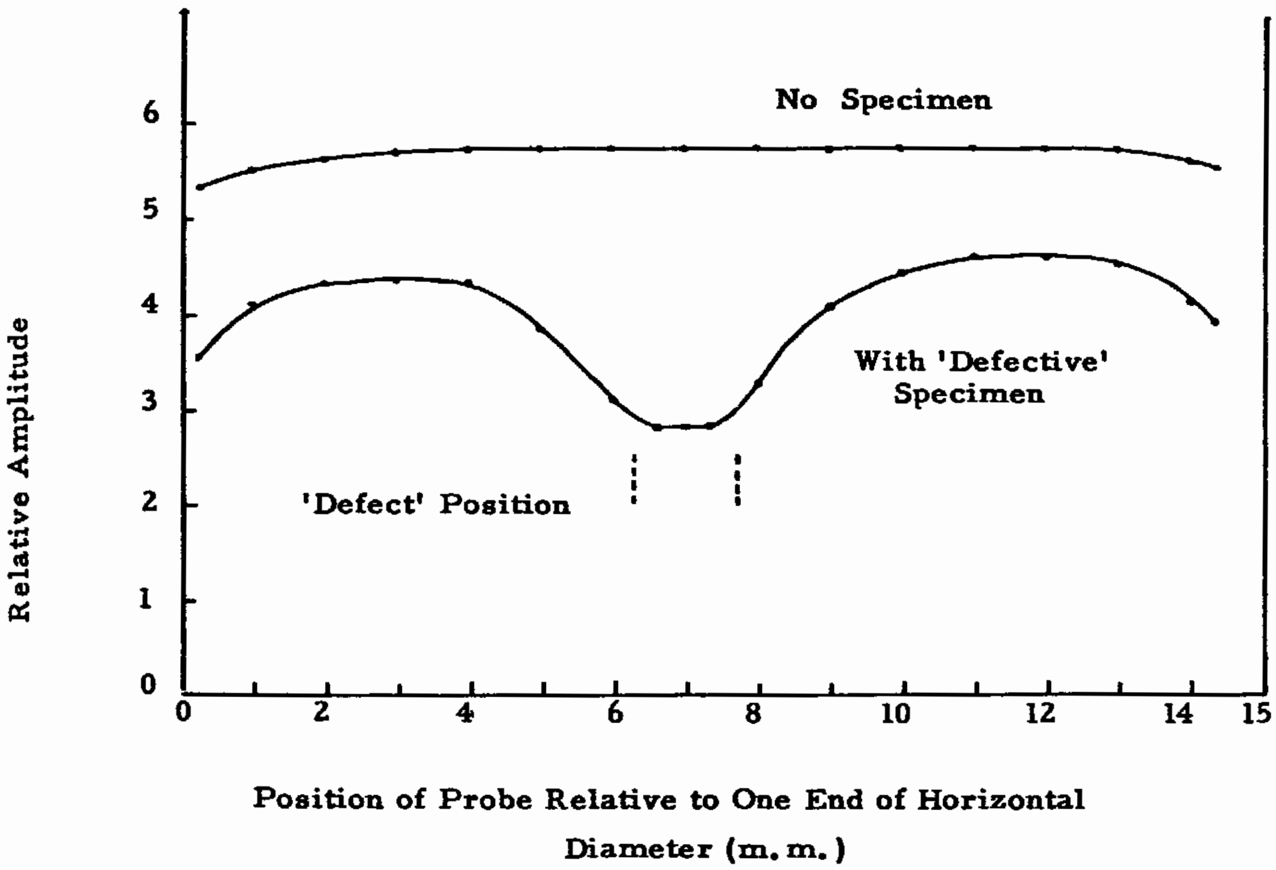

Fig. 3 - SPATIAL VARIATION OF OUTPUT VOLTAGE ON BACK SURFACE OF RECEIVING CRYSTAL WITH AND WITHOUT SPECIMEN. The specimen contained a simulated defect 1/16-inch diameter by 0.016 inch. (The amplification was increased before taking data for lower curve.)

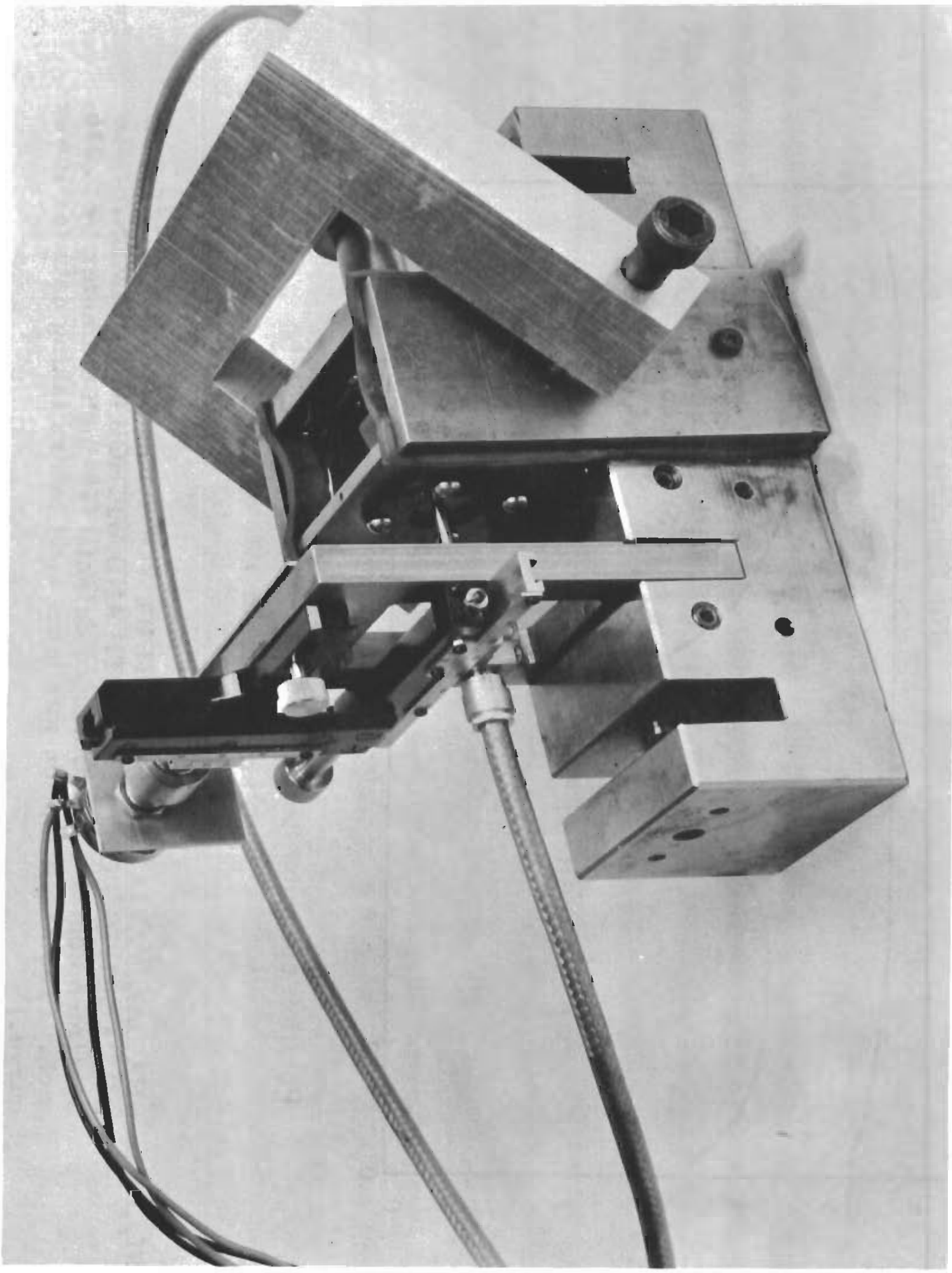

Fig. 4 - MECHANICAL SCANNING APPARATUS

both cases, the intensity of the electron beam is dependent upon the sound intensity incident on the detecting crystal in the region of the probe. In one system the intensity of the electron beam is continuously dependent on the sound intensity; in the second system, the electron beam produces a visible image only for sound intensities greater than a preset value.

In each case a conventional pulsed oscillator is used to generate the excitation for the transmitting crystal. The output from the probe is amplified and detected in a wide band amplifier to give a positive video pulse.

The two scanning systems each require an electronic circuit to convert the variations in the video pulse height into variable light intensity on an oscilloscope screen. The circuit for the continuously variable system is shown in Fig. 5 and for the step control system in Fig. 6.

The first two stages of the circuit of Fig. 5 are conventional resistance-coupled amplifiers. The last stage is a resistance-coupled amplifier with the grid biased beyond cutoff. The tube conducts only when the positive video pulses applied to the grid are of sufficient amplitude to overcome the bias. The output of the stage then consists of negative pulses which are applied to the C.R.T. cathode to increase the oscilloscope brightness. The particular bias applied to the grid determines an equivalent sound pressure at the receiving crystal which must be exceeded for the oscilloscope trace to appear. This tends to shift the crystal output voltage reference and results in greater relative variations in oscilloscope brightness.

The step control circuit of Fig. 6 uses a thyratron to discriminate between sound intensities above and below a preset level. The output of the detecting crystal, after amplification, is applied to the thyratron grid. The anode of the gas tube is supplied with 60 cps ac voltage derived from an isolating transformer. The pulsed oscillator, used to excite the transmitting crystals, is set for 60 pps by using line voltage triggering in the pulse generator. The variable delay of the pulsed oscillator is adjusted so that the pulses generated by the receiving crystal, after amplification, are applied to the thyratron grid just after the anode voltage has passed through a positive maximum. Fixed bias is applied to the tube to prevent conduction during any part of the anode voltage cycle unless a pulse with greater than a certain amplitude, determined by the bias and by the anode voltage, is applied to the grid. If a pulse of insufficient amplitude is applied to the grid, that is, if the sound intensity at the crystal is too low, the thyratron does not conduct and no output voltage is developed. For pulses greater than the critical amplitude, the thyratron fires and a positive voltage pulse is developed across the cathode resistor. This pulse is inverted in the following stage and the negative output pulse is applied to the cathode ray tube cathode to brighten the trace.

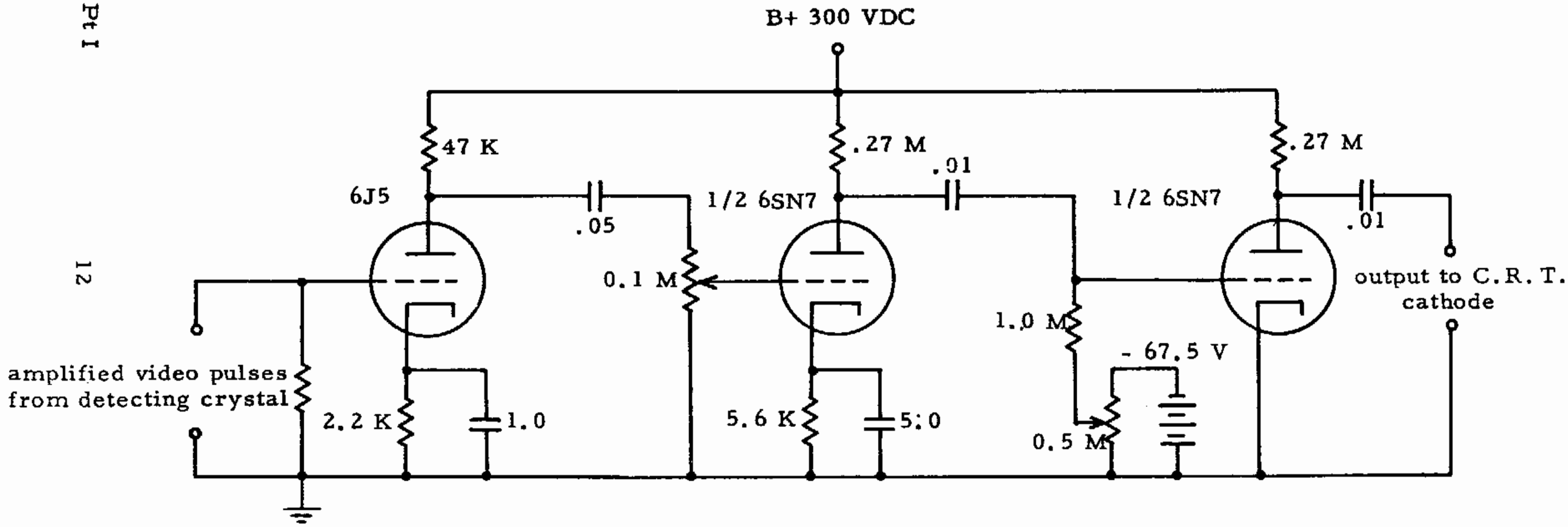

FIG. 5 - CIRCUIT FOR PROPORTIONAL CONTROL OF C.R. TUBE BEAM INTENSITY BY ACOUSTIC INTENSITY

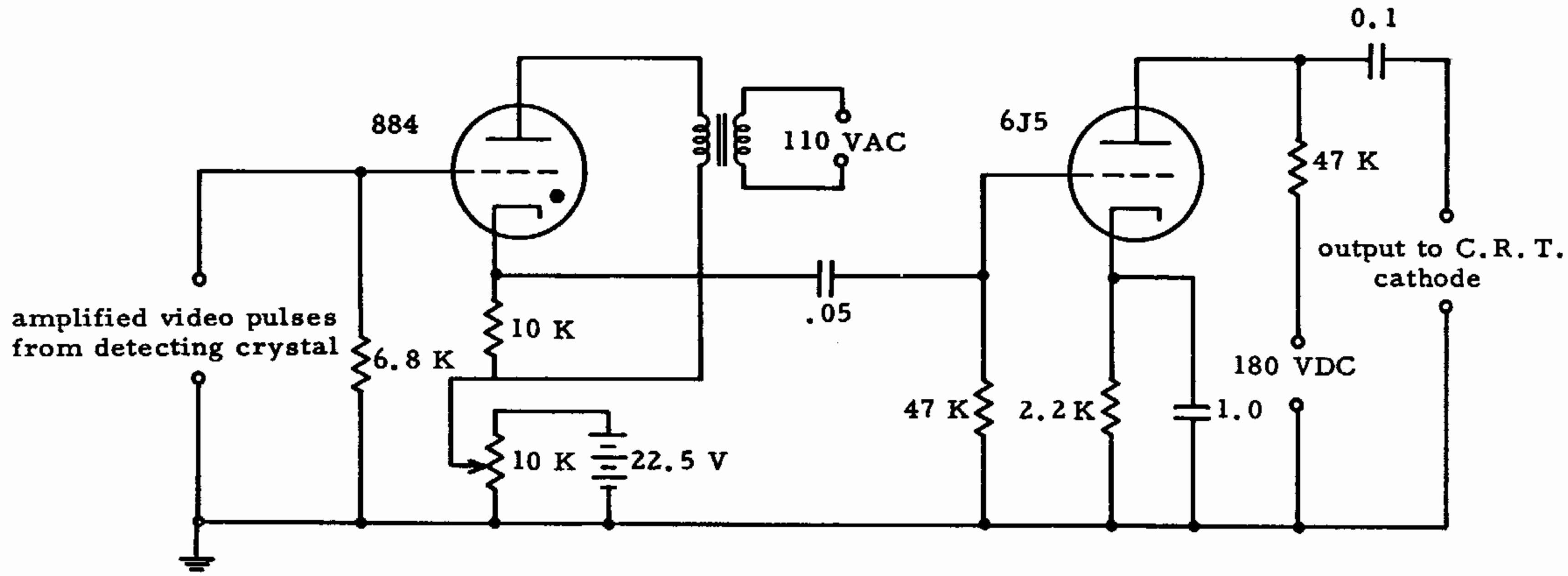

FIG. 6 - CIRCUIT FOR STEP CONTROL OF C.R. TUBE BEAM INTENSITY BY TRANSMITTED ACOUSTIC INTENSITY

The critical sound intensity for which the trace is visible is determined by the bias voltage, the level of the amplified detecting crystal output pulse, and the delay setting. When the thyratron ignites, the grid loses control and consequently any sound intensity sufficiently large to cause the thyratron to fire produces the same light intensity on the cathode ray tube.

For either of the above display systems to provide useful data, it is imperative that the voltage distribution on the back of the detecting crystal in the absence of defect structures be at worst a slowly varying function of position. If the output varies rapidly with probe position, variations caused by the crystals might be interpreted as indications of defects. For this reason it is necessary to check the response of the complete acoustic system and select only those crystals with the required smoothness of response. The spatial variation in output of the detecting crystals caused by anomalous behavior anywhere in the acoustic system has been checked by applying the amplified and filtered output of the detecting crystals to the vertical amplifier of an oscilloscope; the scanning potentiometer output is applied to the horizontal circuit. This method provides a semi-automatic plot of output versus probe position along a diameter of the crystal. The response along diameters of selected crystals is as shown in the upper trace of Fig. 7. The lower line is a reference line obtained with the pulsed oscillator inoperative. The height of the upper trace relative to the reference is proportional to the electrical output of the crystal at a point on the crystal diameter proportional to the distance to the end of the trace. The lower set of curves show the reference line and the variation in output when a 1/16-inch diameter wire is placed in front of the detecting crystal.

Typical results obtained using the scanning-display systems are shown in Figs. 8, 9 and 10. Figures 8 and 9 show results using the liquid couplant system shown in Fig. 2. Figure 8A shows the displayed intensity pattern of sound transmitted through a specimen containing the simulated defect shown in Fig. 8B. The step control display circuit (Fig. 6) was used and the transducing elements were 7 mc/s X-cut quartz crystals operated at their third harmonic, approximately 21 mc/s. Figure 9 shows sound intensity at the receiving crystal with a 1/16-inch diameter wire placed 3/16 inch in front of the detecting crystal and parallel to the diameter. Crystals operated at their fundamental frequency of 15 mc/s were used as transducers. The continuously variable control circuit of Fig. 5 was used.

Figure 10A shows the display obtained for a 0.045-inch diameter hole 1/2-inch deep drilled normal to the axis of a one-inch diameter steel rod 3/16-inch from one end. Three-quarter-inch diameter 7 mc/s X-cut crystals were mounted concentrically on the end surfaces as shown in Fig. 10B. The crystal on the end nearer the hole was used for detection.

Molybdenum specimens, coated with Chromalloy W2 have also

FIG. 7A - OUTPUT VOLTAGE OF DETECTING CRYSTAL ACROSS A DIAMETER WITHOUT SPECIMEN. The upper trace is the output; the lower is the zero voltage reference.

FIG. 7B - SAME AS ABOVE BUT WITH A 1/16-INCH DIAMETER WIRE 3/16 INCH IN FRONT OF THE CRYSTAL. Note the localized decrease in voltage.

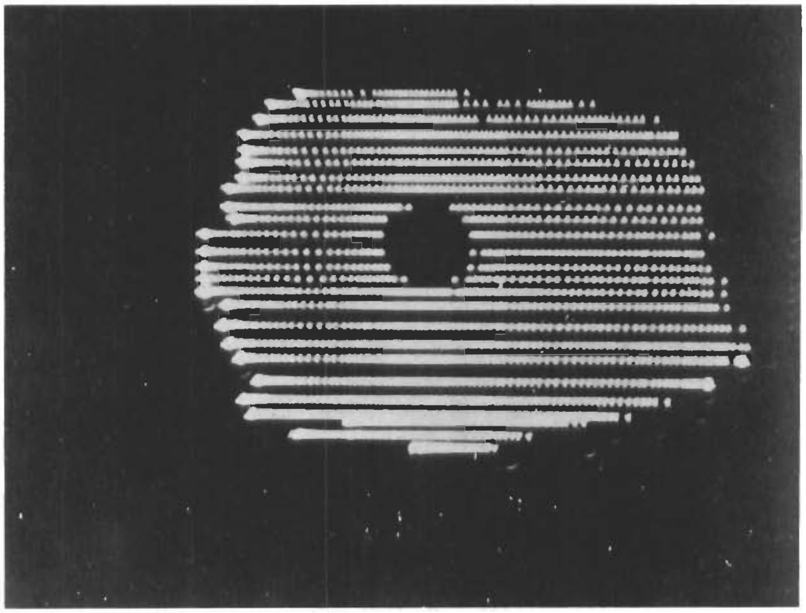

FIG. 8A - STEP CONTROL DISPLAY OF SOUND INTENSITY AFTER TRANSMISSION THROUGH SPECIMEN WITH SIMULATED DEFECT

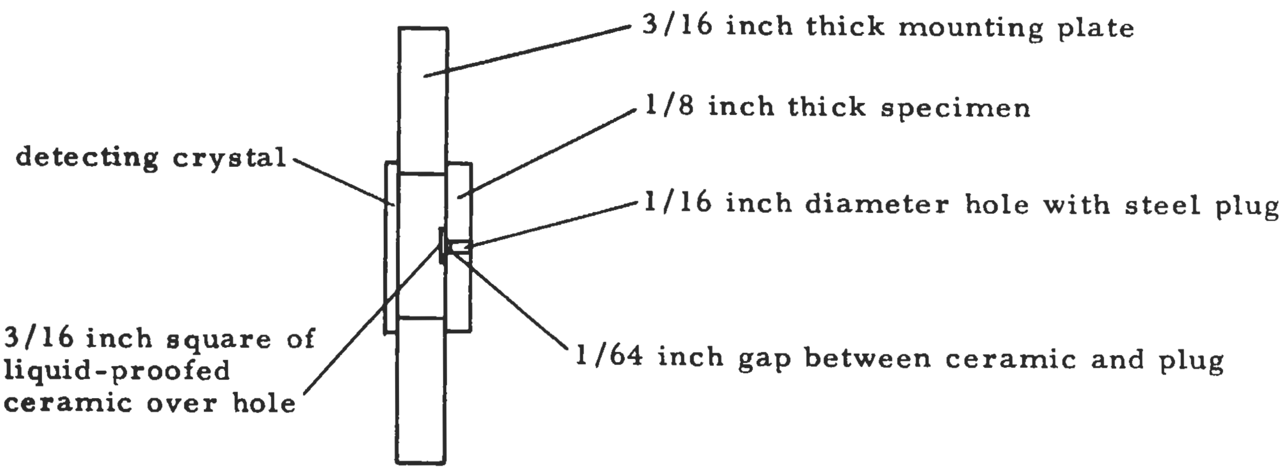

FIG. 8B - DETAILS OF SIMULATED DEFECT
The assembly is shown with the mechanical scanning apparatus in Fig. 1.

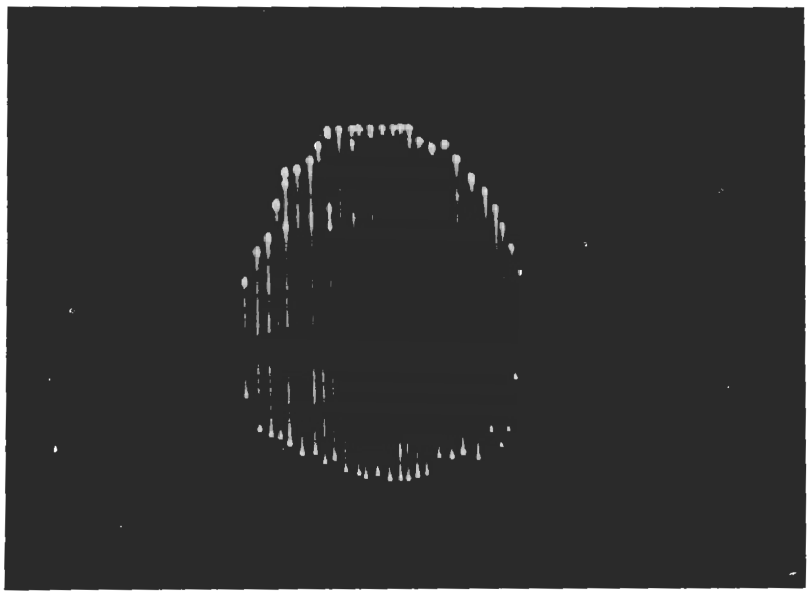

FIG. 9 - PROPORTIONAL CONTROL DISPLAY OF SOUND FIELD WITH 1/16-INCH DIAMETER WIRE IN FRONT OF DETECTING CRYSTAL

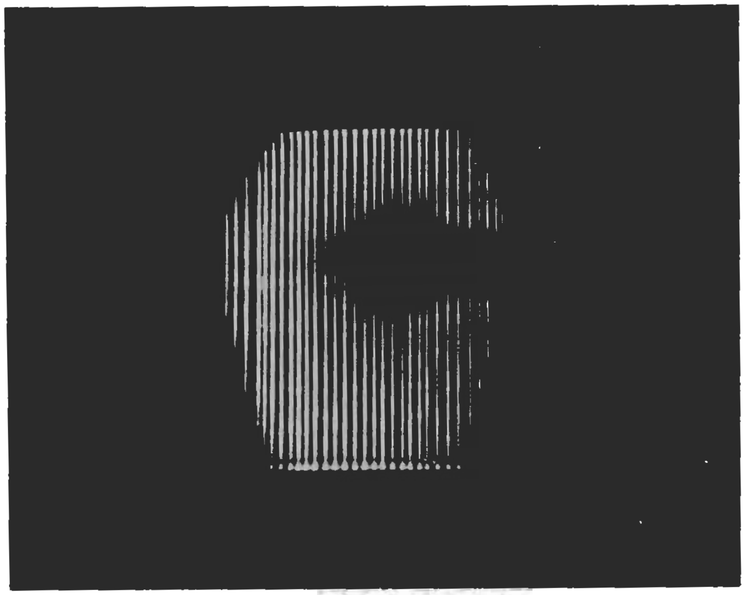

FIG. 10A - PROPORTIONAL CONTROL DISPLAY OF 0.045-INCH DIAMETER HOLE IN A STEEL CYLINDER

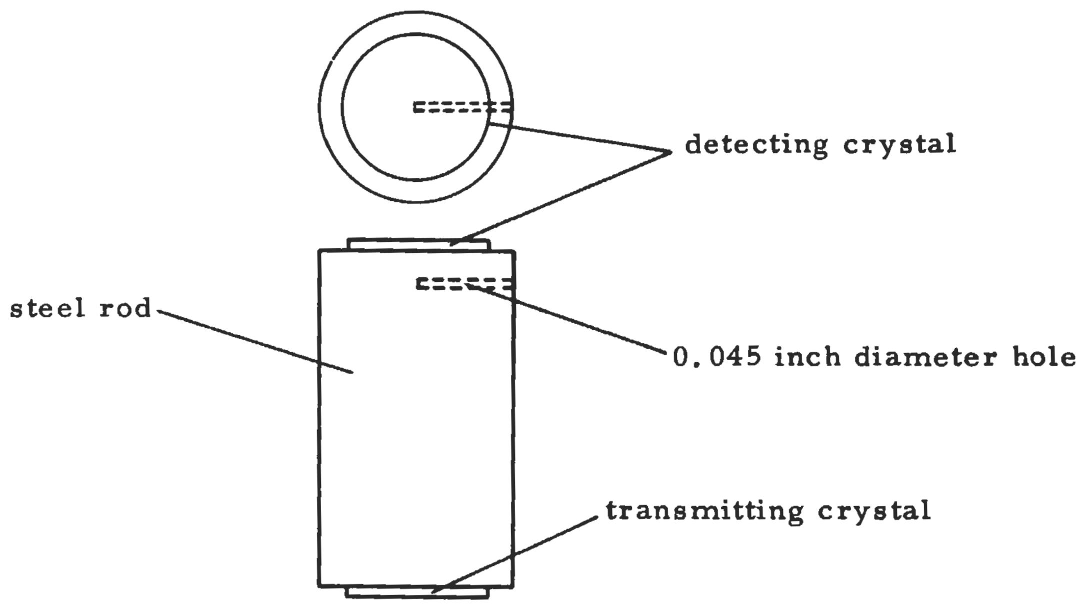

FIG. 10B - DETAILS OF SPECIMEN AND SIMULATED DEFECT been used with the scanning technique. These specimens did not contain simulated defects so a complete evaluation of the technique could not be obtained. It was noted, however, that sonic energy was transmitted more readily through the molybdenum specimens than through steel specimens coated with zirconia. Consequently, the direct transmission and charge scanning method should provide a satisfactory method of examining Chromalloy coatings on molybdenum.

As will be noted above, normally crystals with fundamental frequencies between 7 and 10 mc/s are used. However, crystals with fundamental frequencies of 15 mc/s have been used, and frequencies of 30 mc/s (third harmonic of a 10 mc/s crystal) and 35 mc/s (fifth harmonic of 7 mc/s) have been tried. Generally, poor results have been obtained using the higher frequencies. The coatings can become an appreciable part of a wavelength at higher frequencies, causing impedance changes through a transmission line transformer action. Alignment of the various elements becomes more critical and in addition, the efficiencies of the crystals operated at harmonic drops radically. Although it would be feasible to use an electrical transformer to increase the voltage applied to the transmitting crystal, the advantages gained by using higher frequencies do not merit the added circuit complexity.

RESOLUTION

The defects for which photographs of ultrasonic displays are shown have minimum dimensions of 1/16-inch. However, a 1/32-inch diameter simulated defect has been resolved and the display quality indicates that 1/32 inch is not the limit of resolution.

The inherent resolution of the system is determined by several factors:

(1) Spacing between the specimen and receiving crystal

(2) Wavelength of the ultrasonic signal

(3) Mechanical interactions within the piezoelectrical crystal

(4) Electrostatic interactions between adjacent portions of the crystal.

The first two factors together determine the acoustic resolution. The defect must be a significant fraction of a wavelength to produce a sound shadow and if the receiving crystal is too far from the defect, the shadow at the crystal will be obliterated by diffractive effects. This is illustrated in the Schlieren photographs on page 9 of WADD TR 60-157. The shadow in the lower photograph is clearly visible near the defect.

Even though no sound pressure is incident on the crystal at the projection of the sound shadow, the surrounding portion of the crystal is excited by the ultrasonic wave. The stresses developed in the crystal by the ultrasonic pressures are coupled to the region intersected by the shadow. Consequently, even with a perfect sound shadow, some charge will be produced on the surface of the crystal at the projection of the defect.

The use of a probe also tends to broaden the apparent defect size. The potential developed in the probe by the surface charge can be computed, in principle, by the equation

$$V = \int \frac{\sigma \, dA}{r}$$

where σ is the charge density in the element of crystal area dA, and r is the distance from the element of area to the probe. The integral is taken over the entire crystal surface.

Examination of this equation shows that the probe potential is not maximized when the probe is over an area of high charge density, but is largest when the probe is positioned at the point such that the integrated quotient of charge density and distance to the probe, is a maximum. Accordingly the curves of Fig. 3 and the photograph, Fig. 7B, must be carefully interpreted.

The effect of crystal surface finish was also investigated to a limited extent. Normally, crystals with a ground finish have been used. Several crystals were polished and optically checked. A one percent variation in thickness was found along with a small surface curvature. This could account for variations in probe voltage at different parts of the receiving crystal in the absence of a specimen. The polished crystals exhibit one disadvantage in that it is more difficult to retain the vapor-deposited electrode coating. The rougher ground finish provides a more tenacious bond between the coating and the crystal.

Section IV

SURFACE WAVES

When a compressional wave is obliquely incident at the interface of two different media, mode conversion may occur at the interface and shear or surface waves may be generated. A surface wave exists only in a thin surface layer a few wavelengths thick and, hence, should be an appropriate form of ultrasonic energy to investigate surface properties of a solid.

When a compressional wave is transmitted through a liquid and is incident on a solid, a surface wave is generated at the incident surface of the solid at a particular angle of incidence. This angle is determined in part by the elastic constants of the solid. If a defect structure in the surface layer locally modifies the effective elastic constants, the defect then would be expected to change to some extent, this critical angle. Accordingly, the possibility exists of utilizing surface waves for defect detection in regions close to a surface.

EXPERIMENTAL APPARATUS

Experiments employing surface waves were made using two experimental methods. In both methods, mode conversion of a compressional wave at an interface is used to generate the surface wave.

In the first method, compressional waves, generated by X-cut quartz crystals, are transmitted through a lucite wedge to the lucite-specimen interface as shown in Fig. 11. Surface waves are generated by mode conversion at the lucite-specimen interface. The angle of the incident compressional wave is predetermined so that Snell's law is satisfied, i.e.

$$V_c / \sin \theta_c = V_s / \sin \theta_s$$

where V_c is the velocity of the compressional wave in lucite,

V_s is the velocity of the surface wave in steel,

θ_c is the angle of incidence of the compressional wave,

and θ_s is the angle of refraction of the surface wave.

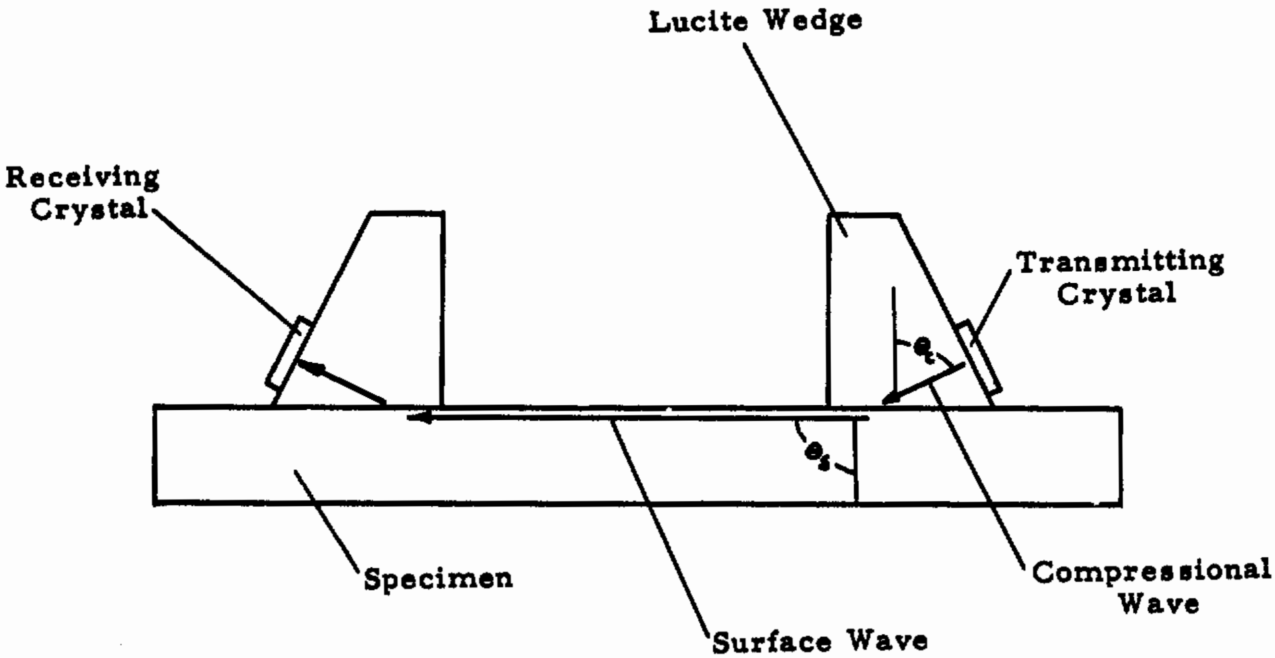

Fig. 11 - SURFACE WAVE GENERATION BY MODE CONVERSION AT A SOLID-SOLID INTERFACE

In the equation above, θ_s is 90°. Hence θ_c may be calculated from the known velocities V_c and V_s, and the lucite wedges made accordingly.

Surface waves were identified by velocity measurements and by the ease with which they are damped. A thin layer of viscous material, such as vaseline, on the specimen surface is sufficient to damp the wave almost completely. With a clean steel surface, and a frequency of 7 mc/s, energy could be propagated over a distance of about a foot with no difficulty.

The second method using surface waves makes use of the apparatus shown in Fig. 12.[3] In experiments using this equipment a compressional wave generated at one of the quartz crystal transducers is transmitted through the water in which the equipment is immersed. When the wave impinges on the sample, part of the energy is reflected and detected by the second transducer, and part is absorbed by the specimen as a compressional, shear or surface wave generated by mode conversion at the water-solid interface. Over a selected range of angles of incidence, only a surface wave can exist, since because of the higher velocities of the compressional and shear waves in the solid, these latter types of waves would suffer total reflection. The approach has been to consider angles of incidence such that only surface waves can exist in the specimen.

EMPIRICAL RESULTS

Many of the possible ultrasonic methods of detecting laminar defects can be evaluated by attempting to detect simulated defects. The theoretical and empirical basis is sufficiently well understood that supplementary experiments are not required. Although a possible method of using surface waves to detect bond defects was formulated, supplementary experiments were required before the method could be applied. The theory of surface waves has been worked out for homogeneous materials. However, in the application to ceramic coatings, the body in which the surface wave is induced is not homogeneous. The effect of inhomogeneity had to be determined and an experimental method, rather than theoretical, was selected. The study was restricted to the effects of the inhomogeneity on applications to flaw detection.

The experiments have tended to rule out the use of surface waves with zirconium oxide-coated metallic specimens. This is illustrated by experiments using the lucite wedge transducers with a

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[3] For a description of this experiment, see WADD TR 60-157, page 15.

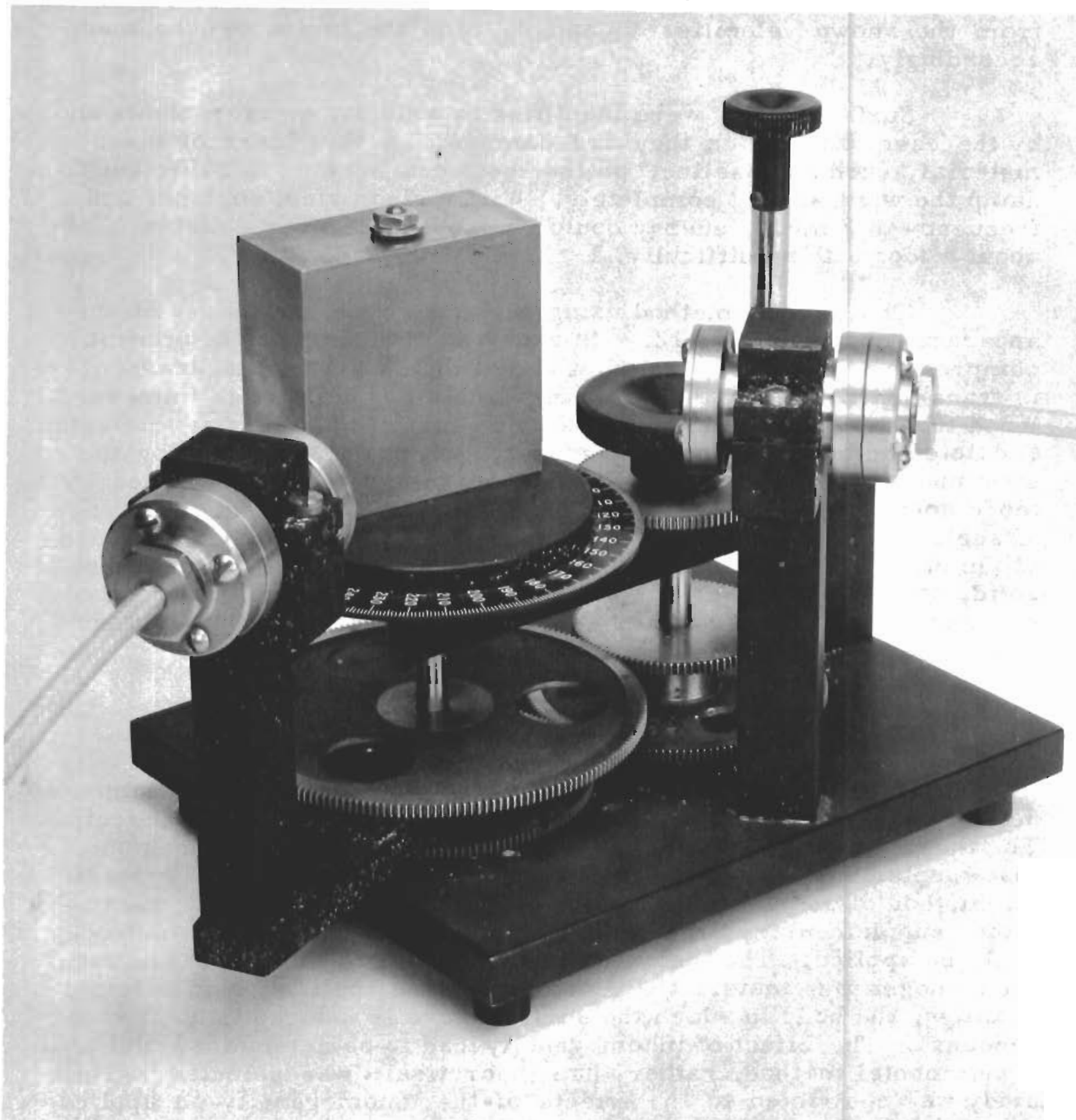


FIG. 12 - APPARATUS FOR GENERATION OF A SURFACE WAVE BY MODE CONVERSION OF A COMPRESSIONAL WAVE AT A LIQUID-SOLID INTERFACE. The apparatus is immersed in water during use.

steel specimen partially coated with zirconium oxide. When the coating is between the two wedges as shown in Fig. 13A, the amplitude of the received pulse is about 20 db less than without the coating for the same wedge spacing and the same voltage applied to the transmitting crystal. A small transmitted pulse is detected but the coating tends to damp the surface wave and makes the amplitude of the surface wave largely insensitive to damping introduced by viscous media placed on the ceramic.

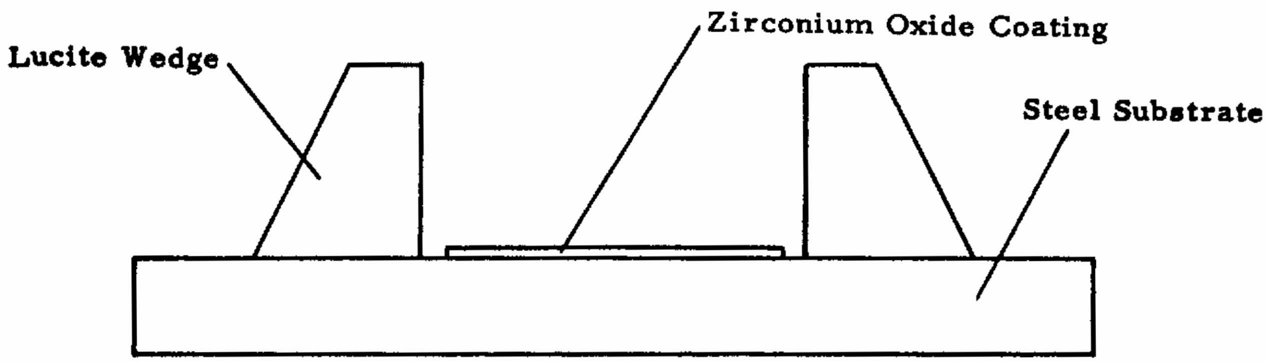
The ceramic coating also affects the generation of surface waves. When the wedges are placed on ceramic-coated portions of the surface, Fig. 13B, practically no detectable signal is received at the receiving crystal. If the crystals are moved so that they overhang the edge of the coating by about 1/16 inch, Fig. 13C, the received signal increases by an amount much greater than could be expected by the decrease in path length.

The experiment illustrated in Fig. 13 is not entirely conclusive. Using the same lucite wedges, with angles appropriate for the generation of surface waves in steel and aluminum, surface waves could not be propagated along either a brass surface or a Chromalloy-coated molybdenum has a higher surface wave velocity. The effect of a are similar while brass has a considerably lower surface wave velocity and molybdenum has a higher surface wave velocity. The effect of a thin layer of zirconia, about a quarter-wavelength thick, is not known and a theoretical investigation is necessary to determine the effects on surface wave generation. This effect of the coating becomes especially important where the elastic constants of the surface coating may be considerably different from those of the substrate.

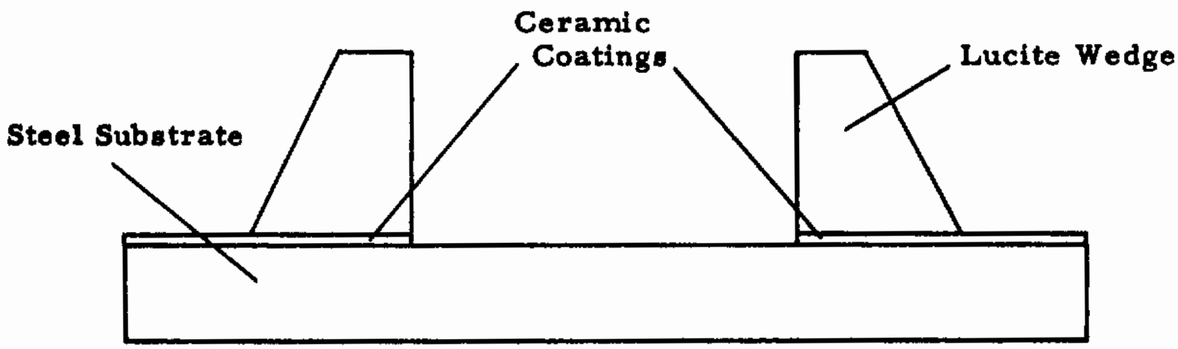
When porcelain-coated steel specimens are used with the lucite wedges, no difficulty is encountered in generating and propagating surface waves. The porcelain coating is a glass, and the ultrasonic velocities in porcelain are similar to those in steel. Consequently, the effect of the surface layer might not be as detrimental as would the effect of a zirconia coating.

Similar results are observed when the immersion apparatus of Fig. 12 is used with appropriately shaped specimens. In this apparatus, the generation and propagation of a surface wave is not measured directly. Instead, the transference of energy to a Rayleigh wave by mode conversion is inferred by the sharp reduction of reflected compressional energy at the receiving transducer. The difference in velocities between shear and surface waves is not sufficient to definitely state that the generation of either one of the two types of waves is absorbing the energy. Since a surface wave can exist only over a small range of incident angles,[4] the compressional energy must be

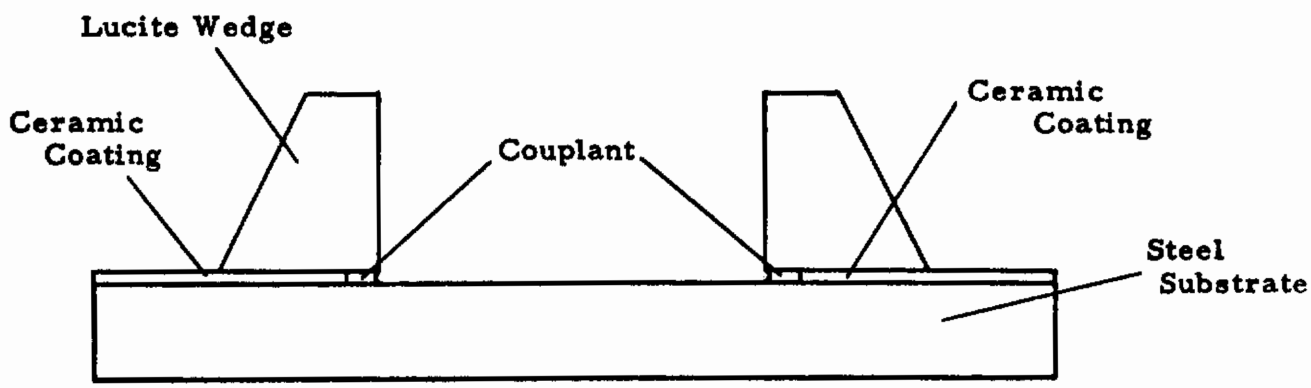
[4] Nondestructive Testing Handbook, edited by Robert C. McMaster, Vol. II, p. 43.23, Fig. 19(a) The Ronald Press Company, New York, 1959.



A. Ceramic Coating Between Transducers.
Transmitted Ultrasonic Wave is Highly Attenuated



B. Ceramic Coating Under Tranducers
Transmitted Ultrasonic Wave is Highly Attenuated



C. Transducers Extending Past Coated Area
Transmitted Wave is 20 db Greater Than in B

Fig. 13 - VARIOUS ARRANGEMENTS OF TRANSDUCERS AND COATINGS FOR STUDYING EFFECT OF CERAMIC ON SURFACE WAVE PROPAGATION

converted to a surface mode rather than to a shear mode.

Typical experimental results are shown in Fig. 14 for brass, porcelain-coated steel and Chromalloy-coated molybdenum. The sharp drop in the amplitude is characteristic of mode conversion to a surface wave. The measured angles are in excellent agreement with angles predicted using published shear velocities, which are only slightly larger than Rayleigh wave velocities, and Snell's law. Measurements of the critical angle are repeatable within six minutes of arc. In contrast to the results plotted in Fig. 14 for three different materials zirconium oxide specimens yielded no predominant critical angle. Instead of a sharp minimum, several minor minima were found, superimposed on a broad minimum. The detailed response curve is not repeatable and is not sufficiently well defined to permit differences in surface elastic properties to be determined with any accuracy.

The empirical results are not sufficient to indicate with certainty the cause of the poor results obtained with zirconia-coated specimens. The porosity of the zirconium oxide coating, together with the excellent results obtained with porcelain coatings of the same thickness (0.003 inch) indicates that ultrasonic damping may be the cause. The results are sufficient, however, to eliminate the surface wave method from further consideration as a possible method of detecting bond defects in zirconium oxide coatings. Conversely, the good results obtained with porcelain coatings on steel and Chromalloy coatings on molybdenum indicate that the method may still be useful with coatings of materials not too different from the substrate in elastic properties. The agreement between the surface wave measurements for porcelain-coated steel and Chromalloy-coated molybdenum also indicates that feasibility of a particular ultrasonic technique is being evaluated. However, since the Chromalloy and porcelain coatings fail in different ways, defects in a porcelain coating may not be similar to defects in a Chromalloy coating. Consequently, it will be necessary to simulate defects in a Chromalloy coating rather than in a porcelain coating, and verify that the technique will detect the defect.

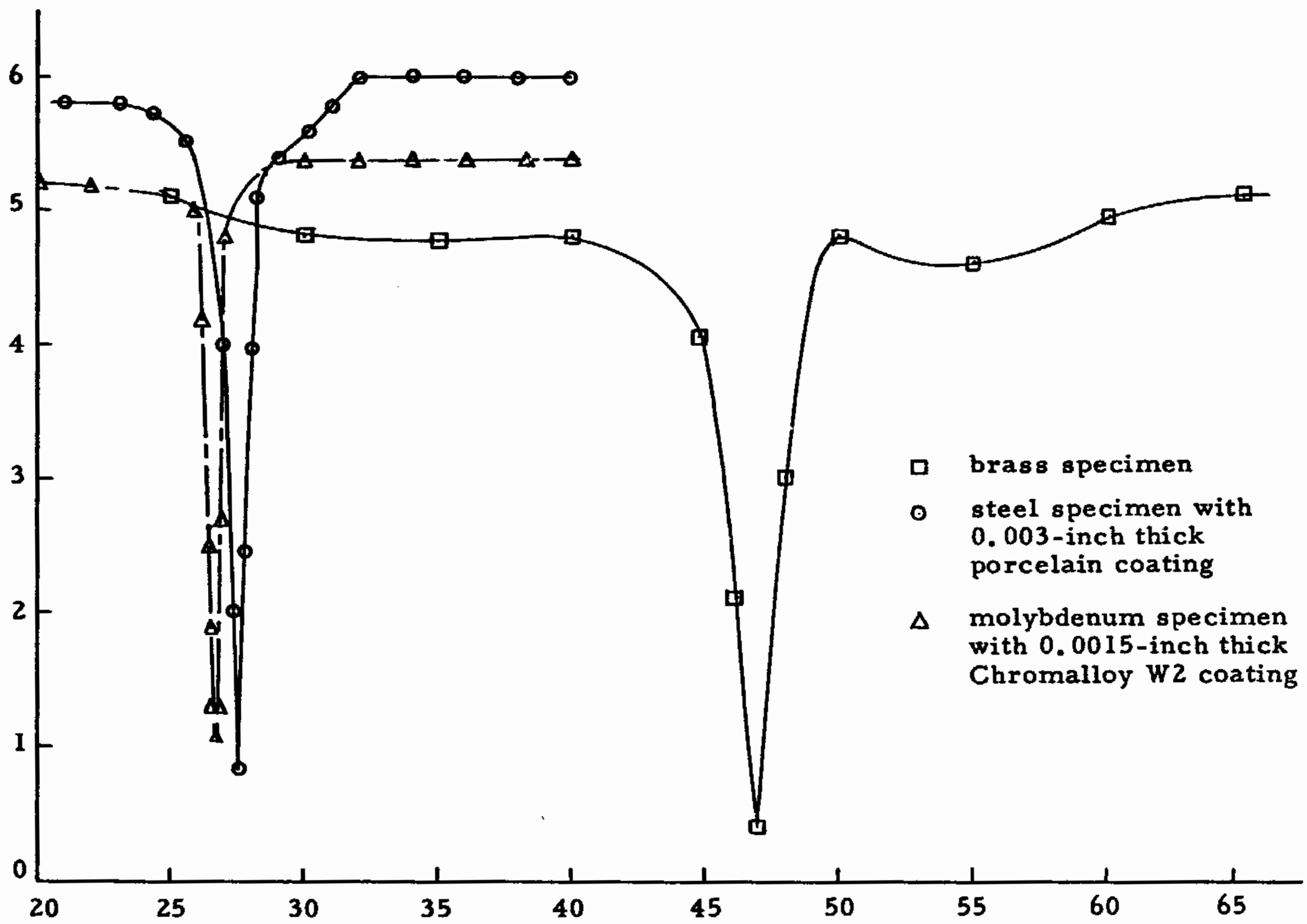


Fig. 14 - VARIATION OF AMPLITUDE OF REFLECTED COMPRESSIONAL WAVE WITH ANGLE OF INCIDENCE. Results are shown for brass, porcelain-coated steel, and Chromalloy molybdenum specimens.

Section V

INTERMODULATION MEASUREMENTS

The intermodulation method of detecting bond defects in a sandwich specimen was described previously.[5] In this method, a longitudinal vibrator is constructed so that the ceramic-metal bond forms a transverse plane at the longitudinal center of the specimen. The vibrator is driven at its resonant frequency (about 14 kc/s) and simultaneously, continuous high frequency energy (5-10 mc/s) is propagated longitudinally through the specimen between X-cut quartz crystals mounted on the end surfaces. If the ceramic-metal bond is defective in the region intersected by the high frequency beam, the electrical output voltage amplitude of the receiving crystal is no longer uniform in time, but is modulated at the frequency of the bond stress.

It has been found that quartz crystals cannot be coupled directly to a solid specimen with sufficient repeatability to give reproducible output voltages from the receiving crystal. Consequently, if large areas are to be scanned, necessitating movement of the crystals over the ends of the specimen, variations in output due to defects might well be masked by variations due to changes in coupling. If the intermodulation method is used, the variations in amplitude caused by coupling changes create no difficulty. The absolute amplitude is not measured; only the percentage modulation is determined experimentally.

PULSE MEASUREMENTS

Additional experiments have been conducted to provide a better understanding of the intermodulation phenomenon. In the first experiment pulsed high frequency energy was applied simultaneously with continuous low frequency energy. The variable delayed trigger feature of one of the oscilloscopes used for obtaining data, was used to trigger the high frequency pulsed oscillator at selected phases of the low frequency stress wave. The apparatus used is shown schematically in Fig. 15.

The results of this experiment are shown in Fig. 16. The top curve shows the stress across the bond as a function of time. Although it has been drawn as a pure sinusoid it should show harmonic distortion. Since the effective area of a defective bond is different during the tensile and compressive portions of the stress cycle these stresses will be unequal.

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[5] WADD TR 60-157, page 33.

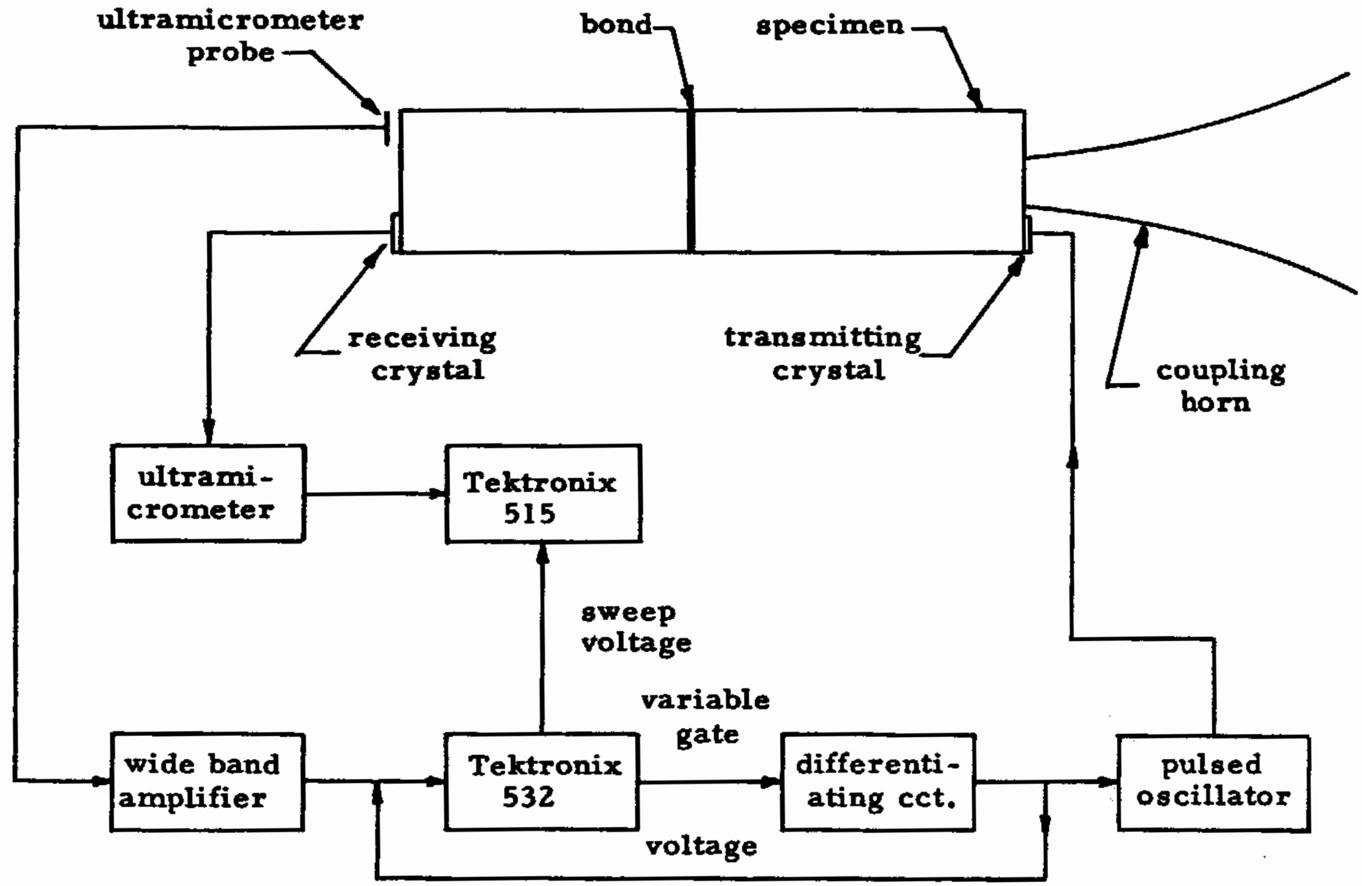

Fig. 15 - BLOCK DIAGRAM OF APPARATUS FOR STUDYING INTERMODULATION USING PULSED HIGH FREQUENCY ENERGY

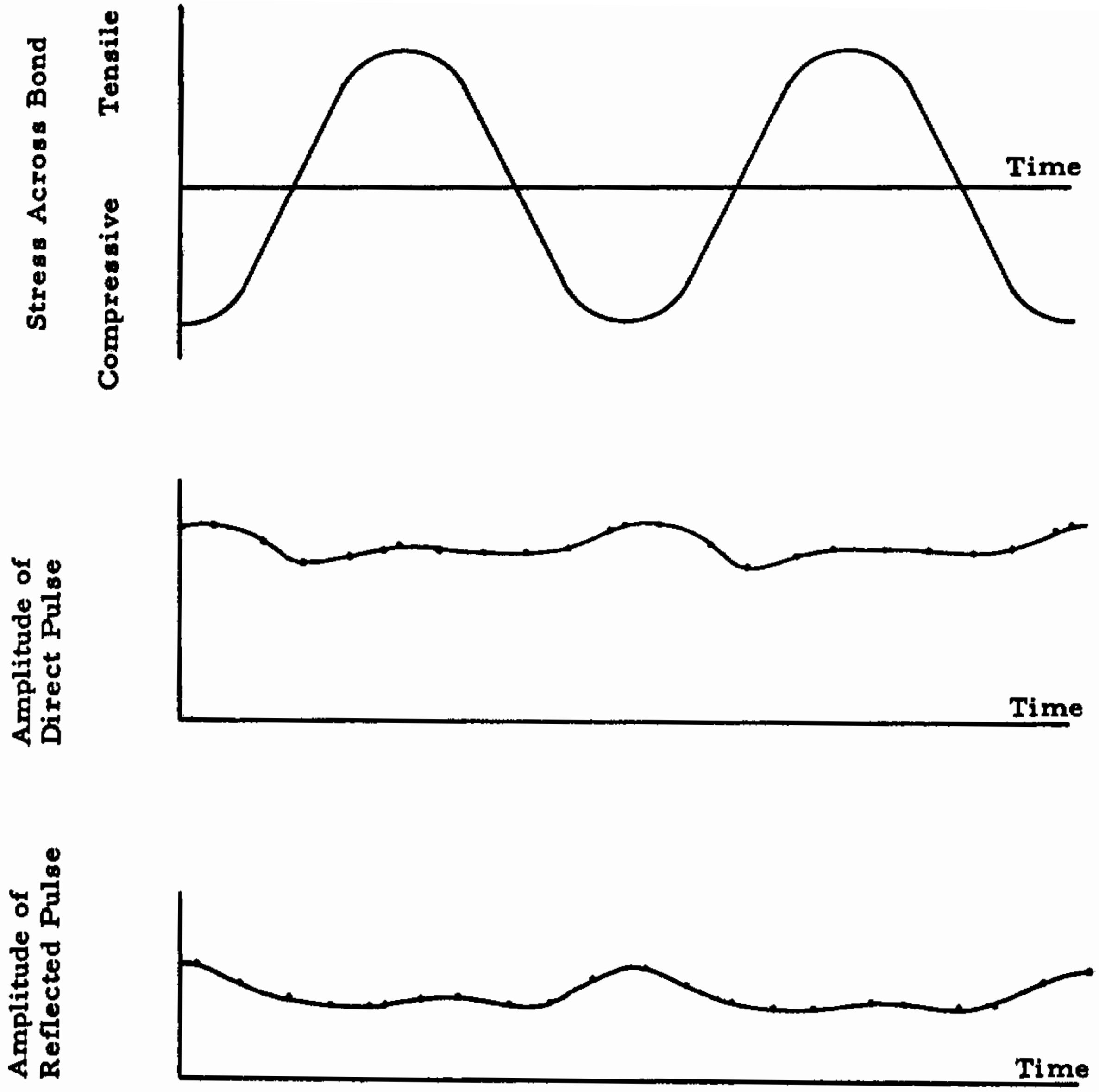

Fig. 16 - VARIATIONS OF BOND STRESS AND RECEIVED PULSE AMPLITUDES WITH TIME. Pulse amplitudes are shown directly below stress that existed when pulse was propagated through bond.

The second and third curves show the amplitude of the received pulses, also as a function of time. The data has been corrected for the phase change that occurs in the bond stress while the pulse is transmitted through the specimen. For both directly transmitted and multiply reflected pulses, the maximum amplitude occurs when the pulse crosses the bond during a peak compressive stress. A second smaller maximum occurs for pulses crossing during a tensile stress.

The empirical results permit a hypothesis to be advanced to account for the intermodulation. During both the peak compressive and tensile stresses, the low frequency stress tends to lock all particles in position. This locking prevents the small high frequency stresses from causing relative motion of particles and hence reduces dissipation. Thus, during the peak stresses the amplitude of the received high frequency pulses would be greater than during periods of low stress. When the peak stress is compressive, the effective bond area is greater than when the stress is tensile. Consequently, the amplitude is greatest for pulses that cross the bond during a peak compressive stress.

This hypothesis would also predict the absence of intermodulation when the bond is free of defects.

CONTINUOUS WAVE MEASUREMENTS

Additional measurements were also made using continuous high frequency energy. As was reported in WADD TR 60-157, the percentage modulation is dependent on the frequency of the high frequency continuous wave. Small frequency changes in the high frequency signal produce large changes in the percent modulation. The output voltage of the detecting crystal also depends on frequency because of the rapid variation of impedance presented to the transmitting crystal with frequency. The dependence of output voltage and percentage modulation on frequency are different since in one specimen, maximum modulation and maximum output voltage occurred together and in a second, maximum modulation occurred with minimum output voltage.

The output voltage of the receiving crystal depends upon the impedance presented by the specimen to the transmitting crystal, or upon the position of the nodes relative to the source end of the specimen. The modulation percentage depends upon the position of the nodes relative to the bond. As the frequency is varied, one node moves away from the bond and is replaced by the adjacent node, causing a complete cycle in the modulation depth. Since the bond is near the center of the specimen, the replacement of one node by the adjacent node at the bond is accompanied by a change in the acoustic length of the specimen (length divided by wavelength) of unity. Consequently, the average amplitude of the receiving crystal would go through two cycles of alternate maxima and minima. If, with a given specimen, maximum modulation occurred with minimum output voltage, then the shortening of the specimen length by one-quarter wavelength (0.005 inch at 10 mc/s) will cause maximum modulation to occur with maximum output voltage.

The operating Q of the two quartz crystals is relatively low when tightly coupled to the specimen. Consequently, small changes in frequency can easily be accommodated by the crystals with negligible loss in efficiency. In using the intermodulation system it is always possible to make slight changes in the high frequency to obtain maximum modulation.

The results of the pulse measurements and the continuous high frequency measurements are not in disagreement with the results reported in WADD TR 60-157. However, the measurements reported above do contribute to a better understanding of the phenomenon.

CHARGE SCANNING AND PERTURBATION OF DEFECTS

The intermodulation method is only one member of a class of techniques that might be designated "perturbation of defects". The application of a stress to the bond, which perturbs any defects present, provides a change in transmission coefficient at a defective region with no change at a bonded region of the ceramic-metal interface. The use of an alternating stress produces a dynamic variation in transmission coefficient at defective regions, which simplifies the experimental measurement. However, a static stress can also enhance the detectability of defects providing appropriate experimental methods are used.

One method that uses a static rather than a dynamic stress is illustrated in Fig. 17. The coated specimen, which incorporates a simulated defect, is cemented to a steel cylinder. X-cut quartz crystals are coupled to the free surface of the specimen and the free end of the cylinder. The composite specimen is supported by a plate so that the application of a force to the yoke will cause a stress to be developed across the bond.

A pulsed oscillator is used to excite the lower crystal and the charge scanning mechanism is used to measure the potential distribution on the back of the upper receiving crystal. By using a thin specimen, diffractive effects occurring between the defect and the crystal are not serious.

This system was used with a zirconium oxide coating. The transmission coefficient through the coating is sufficiently low that a usable signal could not be obtained with the probe. The use of a narrow band amplifier to cut down the noise, combined with a tuning circuit for the transmitting crystal, should improve the signal to noise ratio enough to obtain useful results. The substitution of a Chromalloy W2 coating on a molybdenum specimen should also improve the results by providing a better impedance match at the interface.

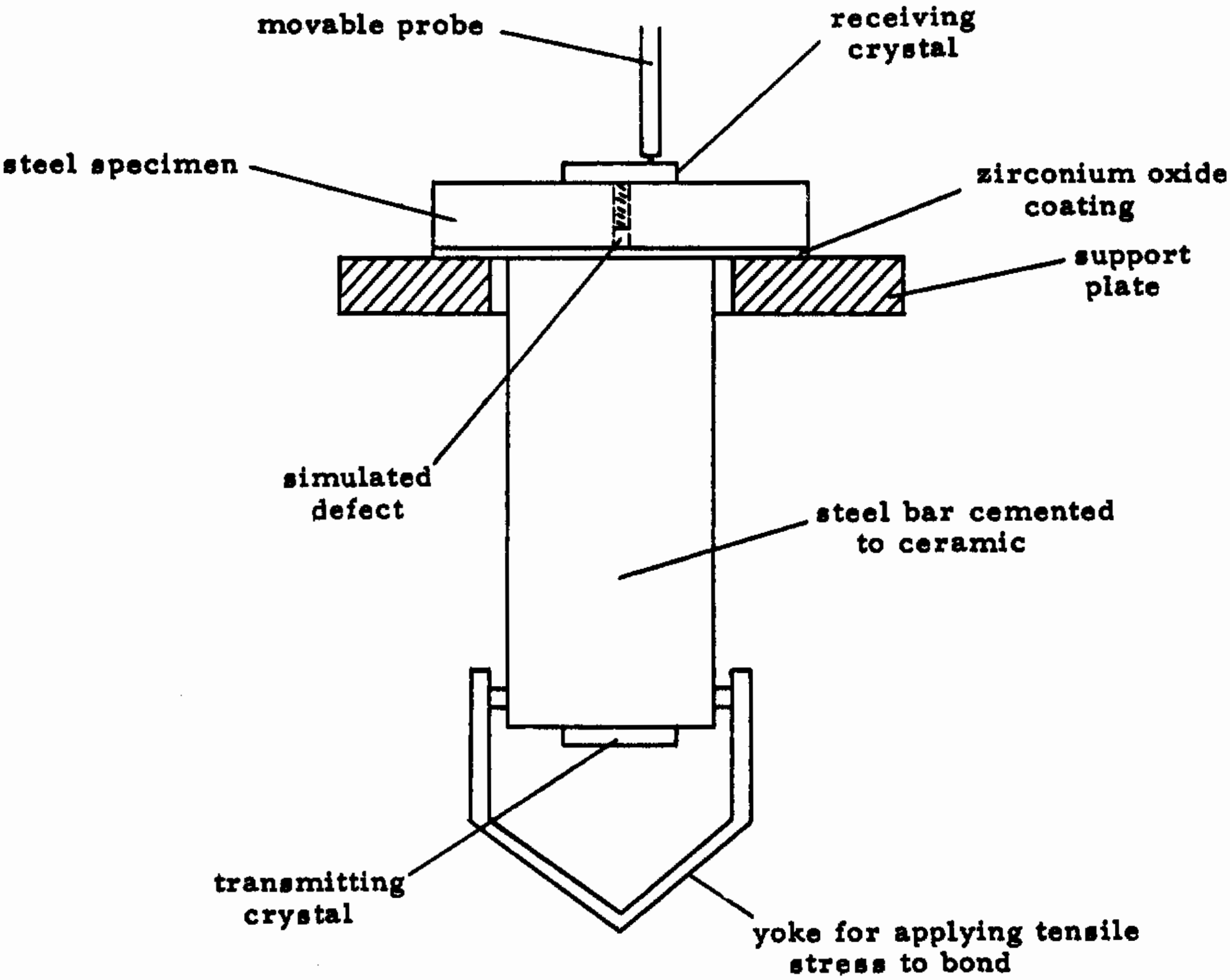

FIG. 17 - APPARATUS TO COMBINE CHARGE SCANNING WITH PERTURBATION OF DEFECTS

Section VI

LOW FREQUENCY DAMPING MEASUREMENTS

The majority of methods to detect bond defects have used high frequency beams to probe for the defect. Although low frequencies have been previously used to determine typical bond strengths and to perturb the defect in the intermodulation technique, little use has been made previously to determine the presence of defects by low frequency measurements. A relatively simple concept has been investigated, using two different experimental methods, with some success.

When a coated specimen is excited so that the coating-substrate bond is strained, it is expected that in regions where the bond is defective frictional losses would be greatly increased. If the losses increase sufficiently, the logarithmic decrement, indicative of the decay rate of the motion, would increase and might even become stress-dependent. By correlating the decrement and bond condition, the presence of defects method would not indicate the position of defects as is done using high frequency techniques, the method might provide a simple means of qualitatively assessing coatings.

SANDWICH SPECIMEN

The first of the low frequency techniques employed longitudinal, half wavelength, specimens resonant at 14 kc/s, with a transverse bond at the longitudinal center of the specimen. The constructions of these specimens is described in a previous report.[6] The specimen is driven at its resonant frequency by a magnetostrictive transducer which is excited by a pulsed amplifier. The pulses are longer than the rise time of the vibrating system so that dynamic equilibrium is attained. At the end of the pulse, the motion decays at a rate dependent upon the mechanical Q.

Typical results are shown in Fig. 18 for two different specimens. In each case the decay curves were first recorded without any initial stressing of the bond. Then, the bond stress was increased until partial failure occurred, and the decay curves remeasured. A marked contrast is clearly evident between the decay curves without the initial stressing, and the curves obtained after the stressing. Both quantitative and qualitative changes occur, and the experiments indicate that as bond failure progresses, the average decrement increases, and becomes more erratic.

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[6] WADD TR 60-157, page 24.

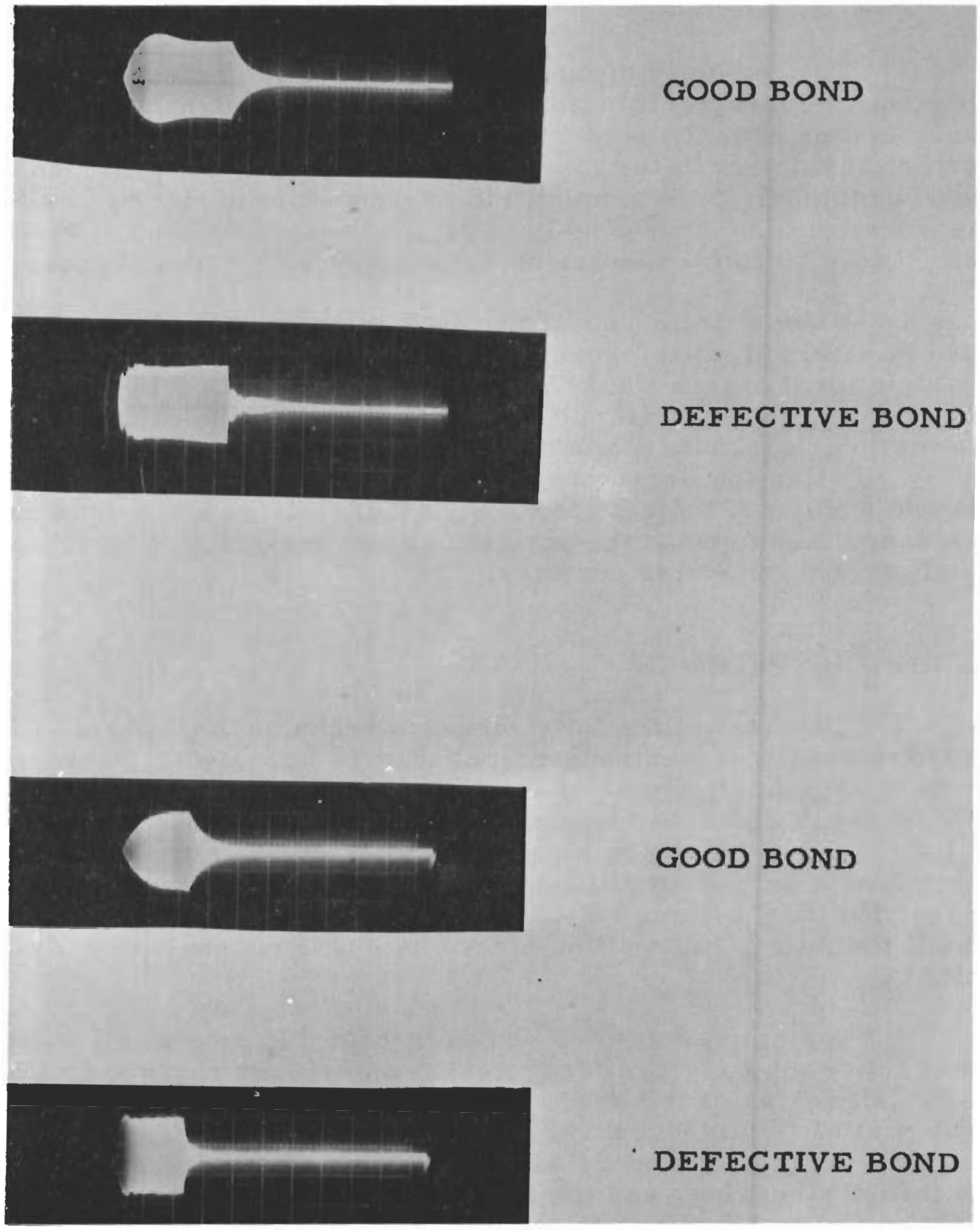


FIG. 18 - CHANGE IN MOTIONAL DECAY CHARACTERISTICS CAUSED BY PARTIAL FAILURE OF TRANSVERSE BOND. (Note both the quantitative and qualitative changes in decay.)

Although a quantitative change does occur in the decrement, the qualitative change presents a better indication of partial bond failure. Consequently, no attempt has been made to compute the relative change in decrement.

FLEXURE SPECIMENS

A study was made on a number of spring steel strips 4.5" x 1.0" x 0.02", having a 2 to 3 mil coating of zirconium oxide on one surface. The dimensions were chosen to provide resonant flexure frequencies of approximately 30 to 60 cps depending on the effective free length. The strips were rigidly clamped, as shown in Fig. 19 and caused to vibrate in the fundamental flexure mode. An ultramicrometer was used to measure the decay time of several types of vibrating specimens. The specimens were:

1. 4 inch effective free length
   a. uncoated
   b. 2 to 3 mil zirconium oxide coating
   c. coating removed by sandblasting
2. 3 inch effective free length
   a. uncoated
   b. 2 to 3 mil zirconium oxide coating
   c. coating removed by sandblasting

By using thin strips of spring steel, a known stress could be applied to the ceramic-metal bond by bending the coated specimen to a predetermined curvature, without permanently deforming the strip. This would presumably damage the bond, and allow a correlation to be made between the measured decrement and the prestress.

Typical decay curves are shown in Figs. 20 and 21 for the specimens enumerated above. The double decay curves are produced by the ultramicrometer used to measure the amplitude. The ultramicrometer detects capacity variations and hence produces an instantaneous output which is independent of which direction the vibrator is displaced. The double curves thus indicate unequal amplitudes in the positive and negative half cycles of vibrator displacement.

Comparison of the decay curves for either the 3-inch or 4-inch specimens shows that a significant difference exists in the decrements for the uncoated and coated specimens (see Figs. 20A and B and Figs. 21A and B). It will be noted that no significant difference in decrement exists between the coated specimens (Figs. 20B, 21B) and the sandblasted specimens (Figs. 20C, 21C). As mentioned previously the sandblasted specimens were completely free of ceramic. Consequently,

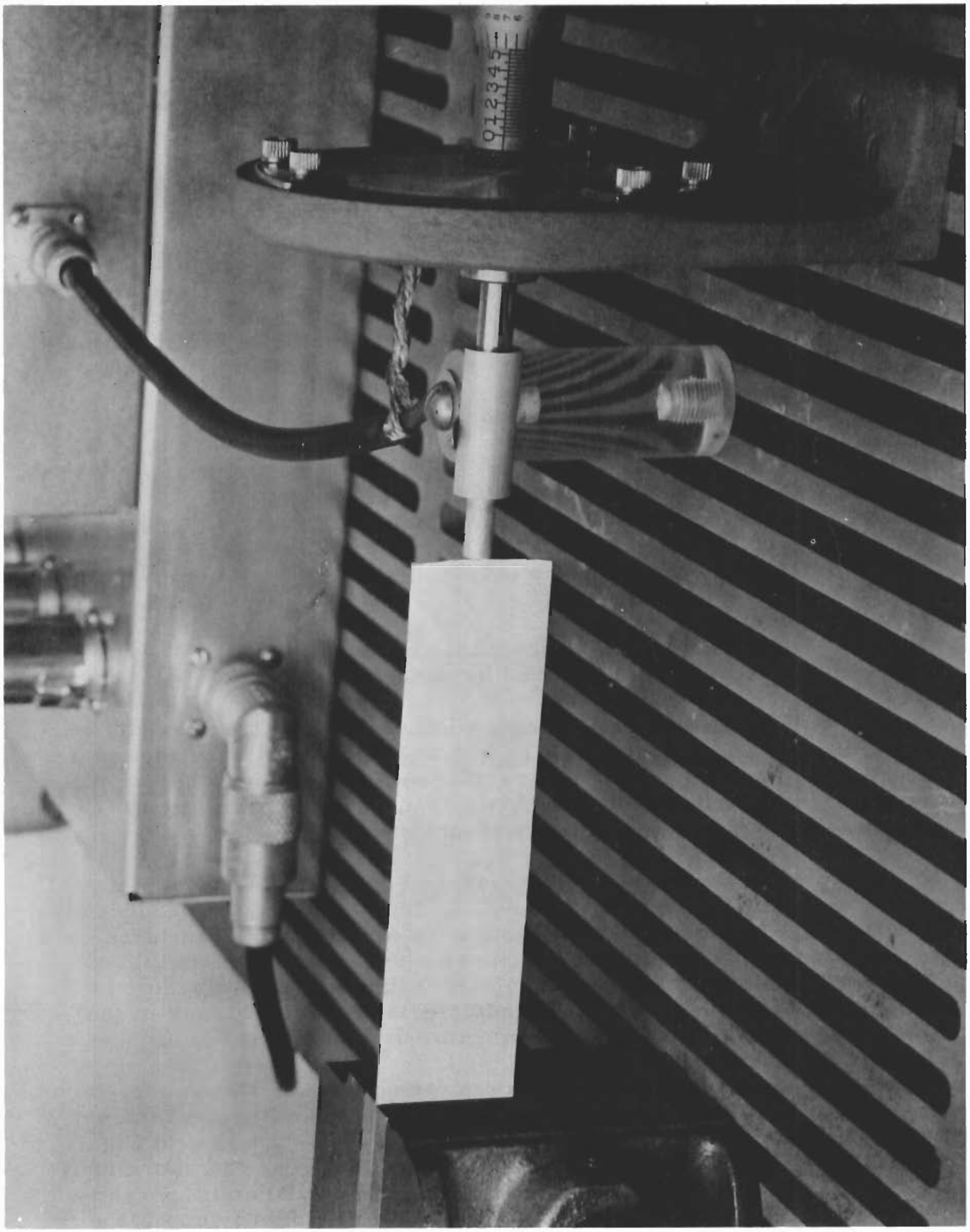
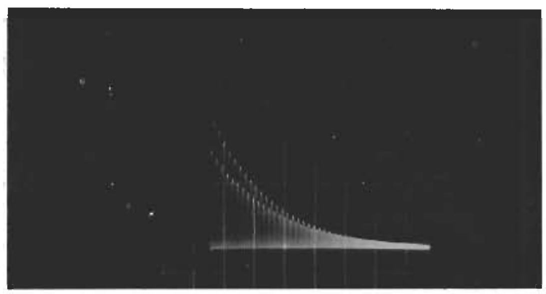


FIG. 19 - EXPERIMENTAL APPARATUS FOR FLEXURE MEASUREMENTS

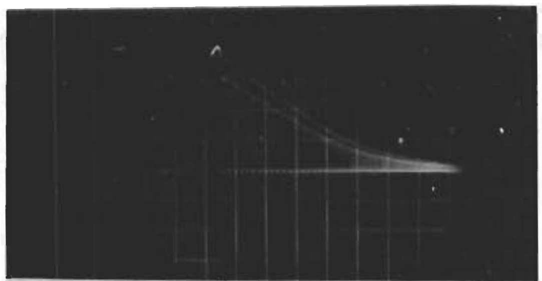
(A) UNCOATED SPECIMEN



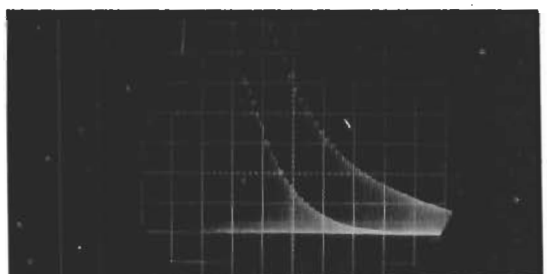
(B) COATED SPECIMEN

(C) SANDBLASTED SPECIMEN

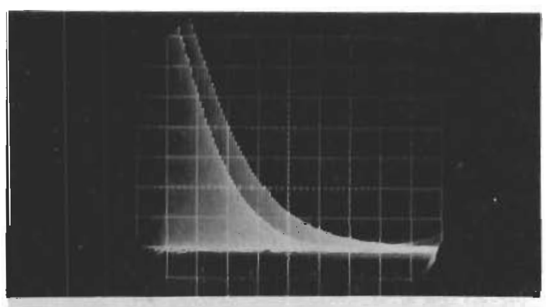
FIG. 20 - DECAY CURVES FOR FOUR-INCH SPRING STEEL SPECIMENS



(A) UNCOATED SPECIMEN



(B) COATED SPECIMEN



(C) SANDBLASTED SPECIME

FIG. 21 - DECAY CURVES FOR THREE-INCH SPRING STEEL SPECIMENS

a close agreement might be expected between the decay curves for the uncoated and sandblasted specimens with little agreement between these decay curves and the curves for the coated specimens. The empirical evidence discussed above indicates that the presence of the coating makes only a small difference in the decrement. The governing factors are the change in properties of the steel caused by the temperature induced during flame spraying, and the change in surface properties caused by surface preparation prior to coating.

The experiments show that the use of spring steel strips does not provide a satisfactory method of detecting poor bonds.

Section VII

SUMMARY AND CONCLUSIONS

The continuity of the bond between various types of ceramic coatings and refractory metal substrates is determined using one of three methods. In the first method, the continuity is determined from measurements of the acoustic energy transmitted through the coating material. Using compressional waves, the spatial distribution of the transmitted energy normal to the sound beam is measured using the charge scanning technique in which the electrical charge distribution is determined at the rear of the receiving quartz crystal. By combining the effects of high frequency and low frequency waves, each wave being simultaneously transmitted through the bond, a second method of defect detection is obtained having the merit of high detection sensitivity. The low frequency signal amplitude modulates the high frequency beam when any defect structure lies in the path of high frequency sound beam. This technique—the intermodulation technique—obviates difficulties caused in some systems by variable crystal coupling. The third technique exploited is that in which surface waves are propagated in the coating-substrate surface.

Three different combinations of materials were used to fabricate test specimens; zirconium oxide flame-sprayed onto a mild steel substrate, porcelain on a mild steel substrate and Chromalloy W2 vapor-deposited onto a molybdenum substrate. The zirconium oxide coating has poor ultrasonic properties. The other two coatings, porcelain and Chromalloy, have good properties and moreover, the properties of the two coatings are similar.

The direct transmission and charge scanning technique offers excellent sensitivity and resolution. Similated defects down to 1/32-inch diameter have been clearly displayed. Successful application of this method to display a defect demands that the defect modify the ultrasonic transmission coefficient. This creates no difficulties with impervious coatings, but might mean that the porous zirconia coatings should be treated to prevent saturation with the liquid couplants. Saturation could conceivably prevent the defect from altering the transmission coefficient.

The surface wave method is capable of resolving small changes in surface elastic constants. Consequently, this method appears promising as a technique to detect variations in the effective constants induced by defects in the surface layer. The method is useful only with materials having good ultrasonic properties. Satisfactory results are obtained with Chromalloy-coated molybdenum specimens or porcelain-coated steel specimens. The method is not applicable to zirconium-coated specimens since the porous, granular structure of the zirconia presents poor ultrasonic properties.

Further experiments have yielded more data on the intermodulation phenomenon. The modulation of a high frequency sound beam is produced by cyclic variations both in the effective bond area and in the frictional losses. Since the amplitude of the transmitted high frequency wave is dependent on the effective bond area, the application of a static tensile stress to a defective bond should alter the effective area and thus enhance the detectability of the defects. The use of the charge scanning display system to display defects while simultaneously applying a static stress to the bond should provide a defect detection system with high sensitivity and resolution.

No previous use has been made of low frequency energy to detect defects since low frequency ultrasonic waves inherently provide low resolution. Investigations have shown that the presence of bond defects can produce a marked change in the logarithmic decrement. This decrement is easily measured. The simplicity of the system might compensate for the poor resolution if only the presence of defects need be determined without detailed knowledge of the location of defects.

More consideration should be given to the type of defects that occur in each of the coatings since the type of defect determines the most appropriate technique to use. The direct transmission and charge scanning method is most applicable to laminar type defects in a plane normal to the axis of the sound beam. For surface cracks, a simple transmission technique using high frequency surface waves might be most appropriate.

The experimental results discussed in this report justify further development of the charge scanning method, the intermodulation method and the surface wave method. The investigations should now be concentrated less on the study of ultrasonic phenomena and more on the application of these techniques to the detection of typical defects in various ceramic-substrate systems.