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Status Report on Ultrasonic Examination Techniques for Multicouple Thermoelectric Subcomponents and Assemblies

K. V. Cook
R. W. McClung
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R. A. Cunningham, Jr.

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K. V. Cook, R. W. McClung, W. A. Simpson, Jr.,
and R. A. Cunningham, Jr.

NOTICE: This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

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STATUS REPORT ON ULTRASONIC EXAMINATION TECHNIQUES FOR MULTICOUPLE THERMOELECTRIC SUBCOMPONENTS AND ASSEMBLIES*

K. V. Cook, R. W. McClung, W. A. Simpson, Jr.,
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ABSTRACT

Since the early 1960s, the United States has been using radioisotope thermoelectric generators (RTGs), developed by the U.S. Department of Energy and its predecessors, for a variety of environments including space. A recent concept in building RTGs uses modular components to allow for an advanced, lighter space mission generator that provides scaled power level outputs. The scalability feature of the modular isotopic thermoelectric generator (MITG) represents a major advance over earlier RTG designs. However, special problems are encountered in applying nondestructive testing methods because of the miniature size of parts that require inspection. This report describes the status of ultrasonic inspection methods being developed to examine the subcomponents and assemblies of the MITG. The principal emphasis has been to evaluate various bonds between subcomponents using ultrasonic techniques that employ focused search units and a pulse-echo method.

INTRODUCTION

The Nondestructive Testing Group of the Metals and Ceramics Division at Oak Ridge National Laboratory (ORNL) has been conducting studies, developing methods, and applying nondestructive testing techniques to evaluate multicouple thermoelectric subcomponents and assemblies for a modular isotopic thermoelectric generator (MITG). The principal distinguishing feature of the MITG space-applicable generator design [as compared with other radioisotope thermoelectric generators (RTGs)[1,2]] is its modular construction. Our task is to provide additional process

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control tools to be used in assessing the quality of the subcomponents. For that reason, the emphasis of this study has been placed on testing at various appropriate stages of subcomponent fabrication. Several areas of interest and benefit have been identified for examination, and agreement has been reached in meetings with personnel from General Electric Company* and the U.S. Department of Energy† on techniques for examining the various bonds between the subcomponents of the multicouple thermoelectric device. Good bonding is needed to ensure good heat transfer, structural strength, and, in the case of the germanium bond and cold-side contacts, good electrical conductivity. Examining and detecting inadequate bonds at the subcomponent stage is advantageous for several reasons: (1) the bonds are usually more accessible for examination, (2) information about poor bonding can provide feedback for correcting the fabrication process, and (3) detection and removal of unacceptable subcomponents from the fabrication stream will prevent further expensive processing on faulty material and will help ensure acceptable assemblies. This report discusses the status of ultrasonic developments in nondestructive testing at the end of 1985.

PRIOR ACTIVITIES

It was determined that the multicouple bonding should be examined at selected optimum stages of the fabrication process. These optimum stages are the N/P sandwich with bonded silicon-molybdenum (Si-Mo) hot shoe, five-couple slices, thermopile assemblies, the heat collector/thermopile bonded assembly, cold-side contacts, and power lead attachments. For convenience in identifying the various fabrication steps and multicouple interfaces, see Appendix A (prepared by General Electric Company*). Feasibility studies have established ultrasonics as a primary examination method for developing specific techniques for the various bonds. Because

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*General Electric Company, Space Systems Division, Valley Forge Space Center, P.O. Box 8555, Philadelphia, PA 19101.

†U.S. Department of Energy, Division of Special Applications, NE-522, Germantown, Washington, DC 20545.

of the need for technology transfer for use of the developed techniques, commercial or modified commercial equipment was used to simplify the transfer. In general, commercial ultrasonic instruments were preferred, but with recognition that circuitry modifications would be necessary to improve frequency response, electronic gating, and other necessary features to attain the needed sensitivity and resolution. In addition, it was recognized that special ultrasonic transducers with selected frequency and focusing for specific bonds would be required. The ultrasonic scanning system was modified to allow preparation of magnified (5×) recordings (x-y maps) of the very small bonded areas.

RECENT TECHNICAL ACTIVITIES

The emphasis in FY 1985 was on developing specific ultrasonic techniques for multicouple subcomponents to be used in process control at the early stages of fabrication. The techniques have been developed using limited, rejected samples supplied by General Electric Company. The following subsections briefly describe the techniques for the subcomponents.

It should be noted that during the course of the study a change in material occurred. The GaP was deleted from the silicon-germanium (Si-Ge) P-type material, causing accompanying dimensional changes in the subcomponents. This change required adjustments in ultrasonic techniques and standards for some of the subcomponents. A few hybrid subcomponents have been obtained, and additional specimens are to be received. Drafts of procedures for several techniques have been prepared. An example is included in Appendix B. The primary ultrasonic method employed for bond inspection uses focused search units and pulse-echo flaw detection schemes. We are currently using an Automation Industries* Reflectoscope (S-80) flaw detector with a 25-MHz pulser-receiver. Figure 1 shows the small research tank* with the S-80 unit sitting on top of the x-y bridge controller

*Automation Industries, Inc., Sperry Division, Shelter Rock Road, Danbury, CT 06810.

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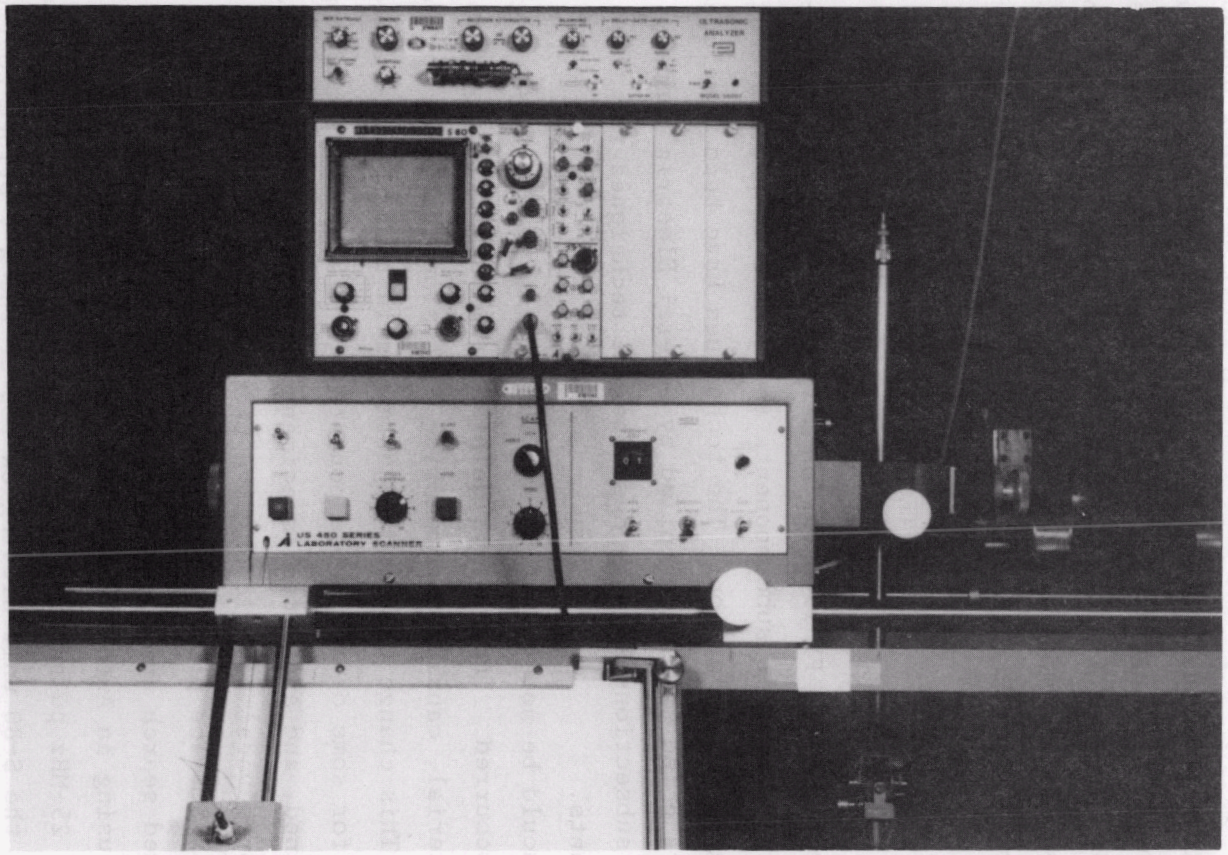

Fig. 1. Ultrasonic immersion tank with ultrasonic flaw detection equipment on the controller bridge mechanism.

mechanism. An additional Panametrics Ultrasonic Analyzer* (model 5600T flaw detector) is sitting on top of the S-80 and could be used instead of the S-80. The search unit and minimanipulator are shown in the water-filled tank (lower right of Fig. 1). Modifications to the basic equipment and additions of peripheral devices will be discussed in the subsections on multicouple subcomponents.

N/P SANDWICHES

The hybrid N/P sandwich is composed of a slice of Si-Ge (GaP) N-type material [19 × 8.38 × 0.831 mm (0.75 × 0.33 × 0.0327 in.)] bonded with 0.05 mm (0.002 in.) of high-temperature glass to a slice of Si-Ge P-type material [19 × 8.38 × 0.734 mm (0.75 × 0.33 × 0.0289 in.)]. An ultrasonic technique has been developed to examine the glass bond between the two slices using prehybrid specimens. The prehybrid samples have P- and N-slice thicknesses of 0.883 and 0.734 mm (0.0348 and 0.0289 in.), respectively; however, we feel that the prehybrid specimens are adequate to use in developing the procedure and calibrating the technique. Figure 2 shows the basic pulse-echo flaw detection scheme as applied to the glass bond in an N/P sandwich subcomponent. An electronic time-amplitude gate is used to monitor the flaw signal, as illustrated in Fig. 2(*b*). The details of the sandwich construction are given in Appendix A.

We developed a silicone-rubber mold to hold several N/P sandwiches in proper alignment during ultrasonic scanning (Fig. 3). The scanning is performed in an x-y raster pattern [limited to a 44- by 44-mm (1.7- by 1.7-in.) zone] with the focused ultrasonic beam entering the major surface of the thickest slice. Since the examination is for the bond quality only 0.831 mm (0.0327 in.) below the entry surface for the hybrid subcomponent, special ultrasonic techniques were required to separate the entry-surface reflection from the reflections from the bond interface (and the back surface of the N/P sandwich). Circuits were modified to improve the high-frequency (25-MHz) performance, improvements were made to the electronic

*Panametrics, 221 Crescent St., Waltham, MA 02154.

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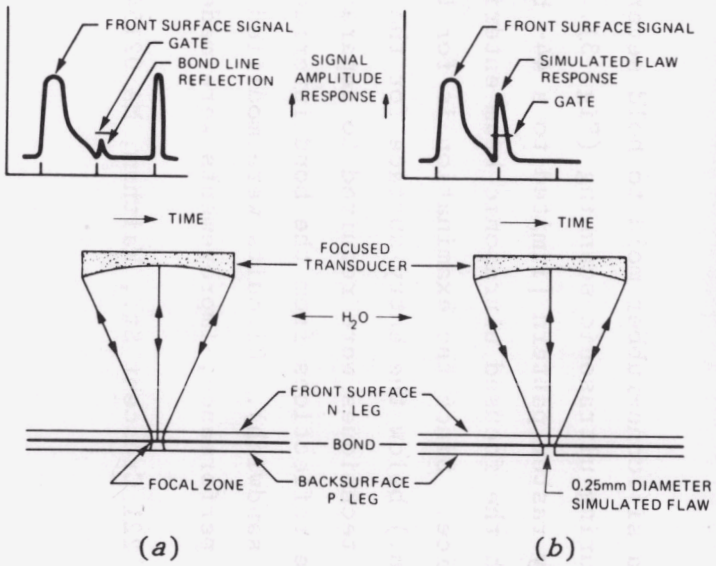

Fig. 2. Ultrasonic pulse-echo technique using a focused transducer to examine (*a*) good bond and (*b*) simulated delamination in bond in an N/P sandwich. The flaw detection video readout is shown above each illustration.

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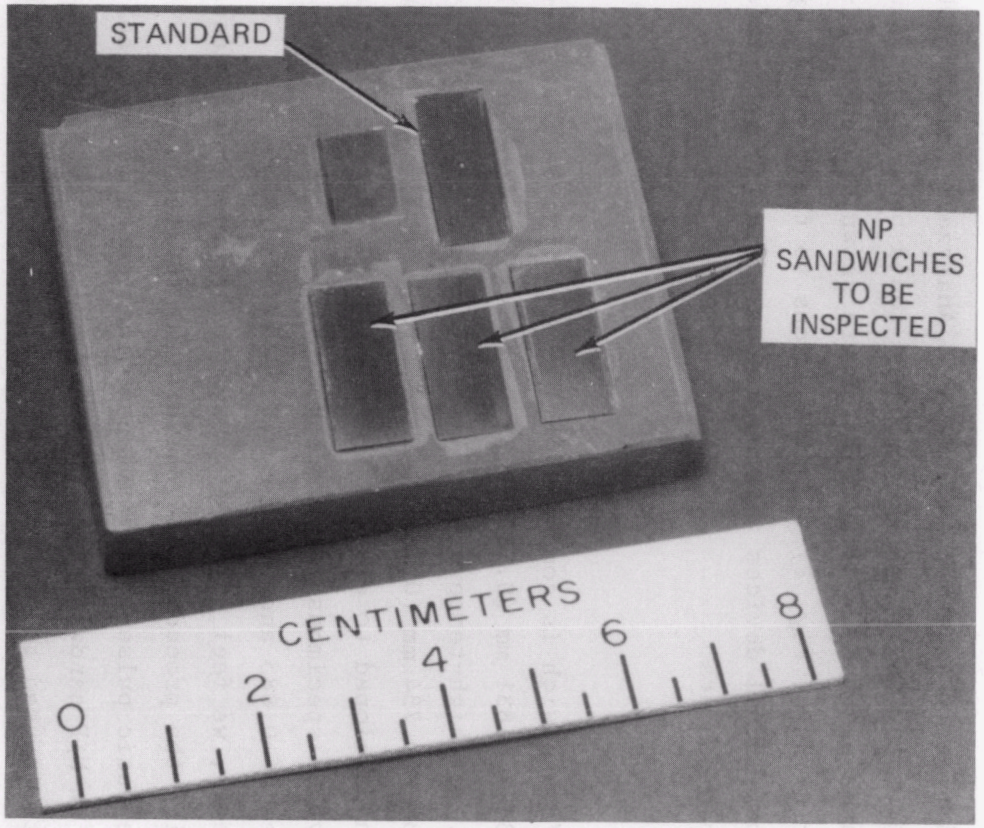

Fig. 3. N/P sandwich specimens in a rubber holder used for pulse-echo ultrasonic inspection of the glass bond between legs.

gate (for isolating the signals from the bond region), and 25-MHz focused search units [25-mm (1-in.) focused length and 0.24-mm (0.009-in.) focal spot] were designed and procured. Figure 4 shows the signature spectrum analysis readout for this particular 25-MHz unit when used with Panametrics equipment and our ModComp II computer analysis system.*

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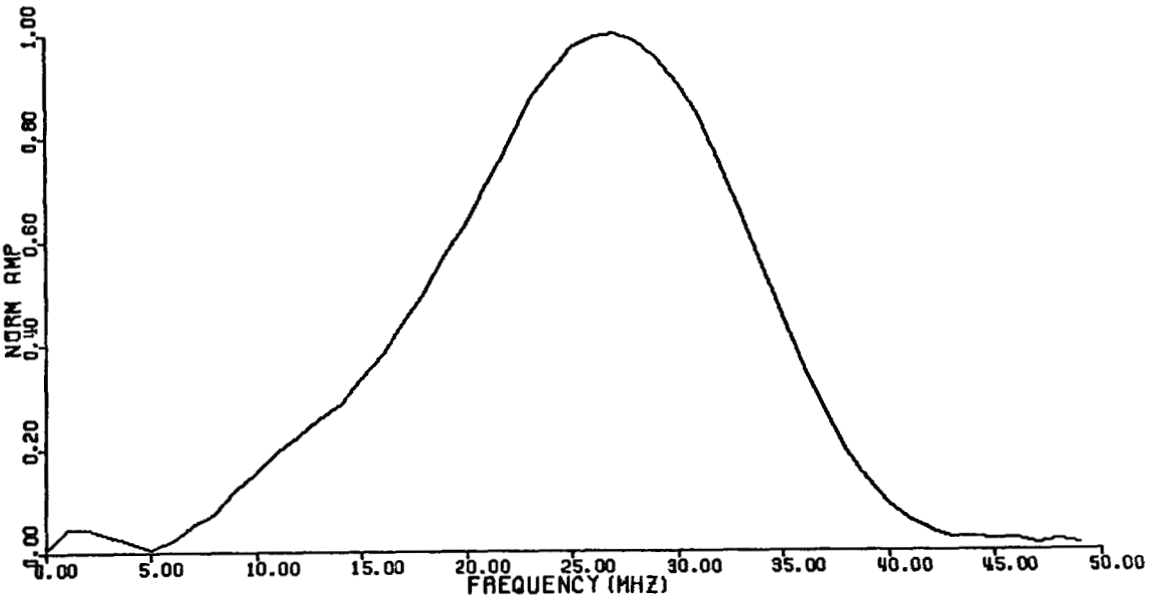

Fig. 4. Spectrum analysis of 25-MHz transducer using front-surface reflection at 25.4-mm (1-in.) focal distance (water couplant)

We also assembled a real-time frequency monitoring system for the interrogation of radio-frequency (rf) pulses from front surfaces, flaws, and back surfaces (when appropriate). Monitoring ensured that the necessary high frequencies were being maintained with the inspection system as required for high-sensitivity examination response. This frequency monitoring system is basically a Tektronix 7704A oscilloscope† with a 7L12 spectrum analyzer plug-in module; however, ancillary electronic components were added for application with commercial S-80 equipment. Figure 5 shows the real-time frequency monitoring equipment along with

*Modular Computer Systems, Inc., 1650 West McNab Road, P.O. Box 6099, Fort Lauderdale, FL 33310.

†Tektronix, Inc., P.O. Box 1700, Beaverton, OR 97075.

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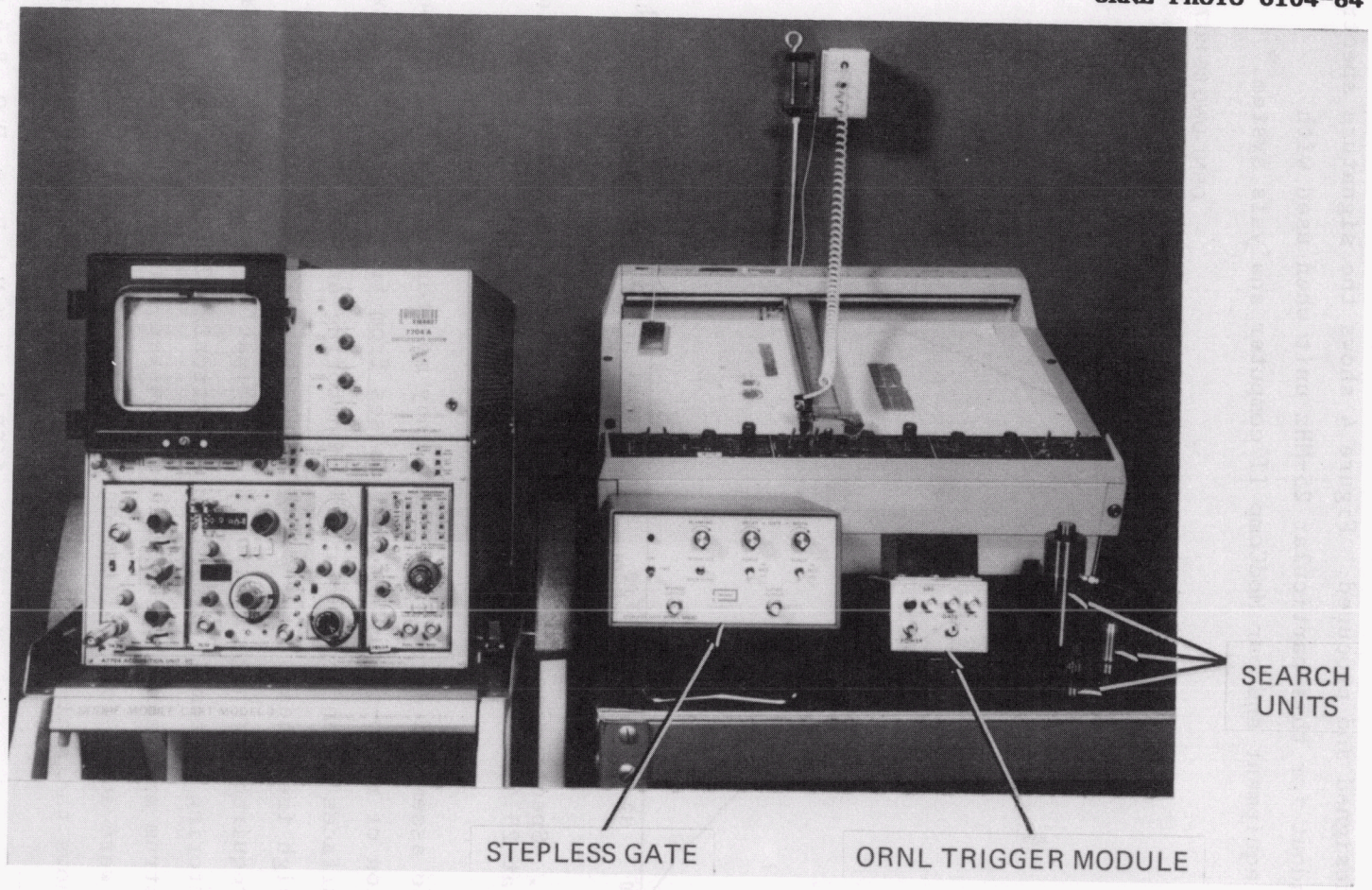

Fig. 5. Real-time ultrasonic frequency monitoring systems, 5× C-scan recorder, and three focused search units.

a modified Hewlett-Packard 7000 5× C-scan recorder* (upper right) and three different focused transducers (right corner). Two ancillary units, required for real-time frequency analysis, are also shown. These are a Panametrics stepless gate (near the center) and an ORNL-fabricated interface (trigger) module (the smaller box located between the transducers and the stepless gate).

Sensitivity and resolution studies were performed on a prehybrid design N/P sandwich specimen. Flat-bottom holes of three diameters [0.13, 0.23, and 0.66 mm (0.005, 0.009, and 0.26 in.)] were drilled through the N slice (with diamond-coated microdrills) to a depth such that the flat bottoms of the holes would be in the plane of the glass bond. The holes served as reference reflectors simulating nonbonds at an approximate 0.883-mm (0.0348-in.) depth. After developing the details of the ultrasonic technique, we demonstrated that all three of the holes in the bond were detectable. We also developed a capability to make analog 5× C-scan (map) recordings to inspect and record the presence of nonbonds as small as 0.13 mm (0.005 in.). Application of the technique to four prehybrid samples supplied by General Electric Company disclosed only one spot of apparent nonbond, and it had an area smaller than the 0.13-mm (0.005-in.) reference reflector. Figure 6 shows the simulated† C-scan 5× data for the three full-size specimens (nothing detected), the one shortened specimen (showing the small flaw indication), and the reference specimen (showing the 0.13-, 0.66-, and 0.23-mm simulated flaws). These data were collected in accordance with the draft procedure MET-NDT-MITG-UT-1 in Appendix B.

HOT-SHOE BOND

The next stage of fabrication is the bonding (with germanium) of the Si-Mo hot shoe [19 × 1.64 × 0.81 mm (0.75 × 0.0645 × 0.0320 in.)] to the 19-mm (0.75-in.) edge of the N/P sandwich. Ultrasonic techniques have been developed to detect areas of nonbond.

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*Hewlett-Packard, 820 Embarcadero Road, Palo Alto, CA 94303.

†Simulated C-scan data are used to provide maximum quality illustrations; however, very good quality C-scan data are obtained by the test and are copied or photographed directly in some instances, especially for less formal reports.

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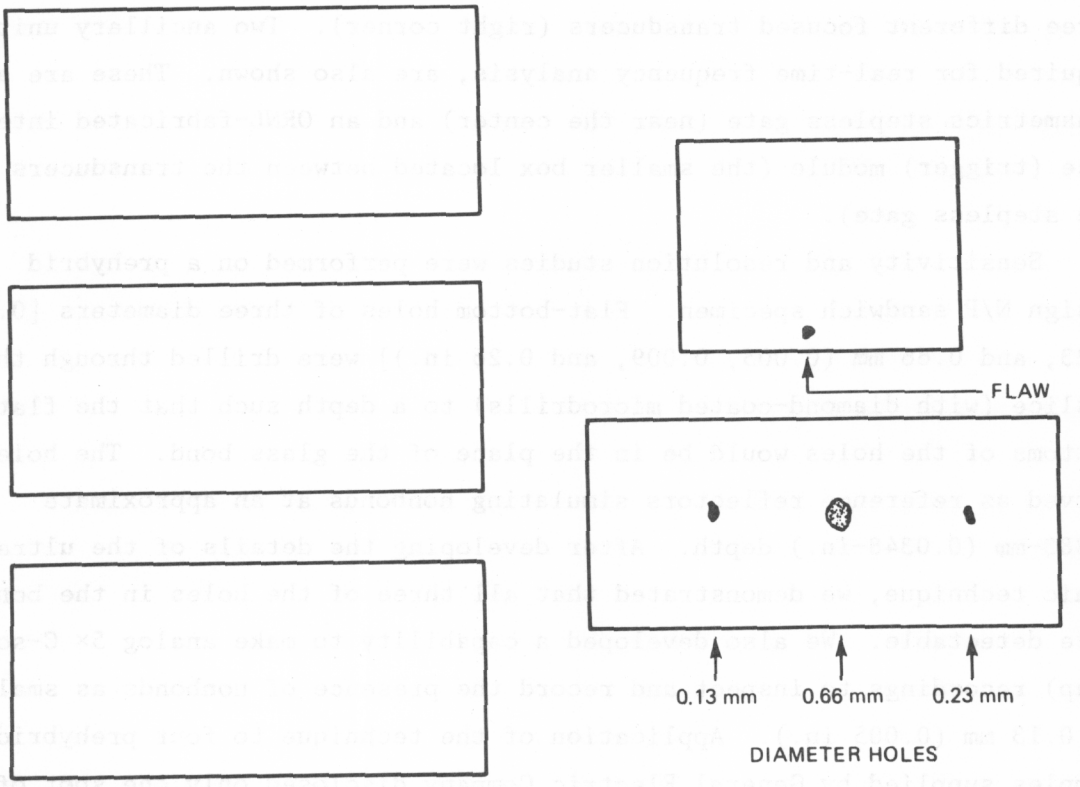

Fig. 6. Ultrasonic data on glass bond of N/P sandwiches (5× recording).

A silicone-rubber mold was developed to hold the prehybrid design N/P sandwich with the hot-shoe bond in proper position for ultrasonic x-y scanning with the focused ultrasonic beam entering the major surface of the Si-Mo hot shoe. This holder was fabricated when only the five prehybrid specimens discussed above were available; therefore, we included two thermopile specimens as well as various calibration pieces. Figure 7 shows the holder with the hot-shoe surfaces located parallel to the holder's major surface. As in the examination of the N/P sandwich bond, special ultrasonic techniques were used to separate the entry-surface reflection from the germanium bond interface reflection only 0.81 mm (0.032 in.) beneath. Instrument modifications noted earlier were also used for this examination. Several different designs were evaluated along

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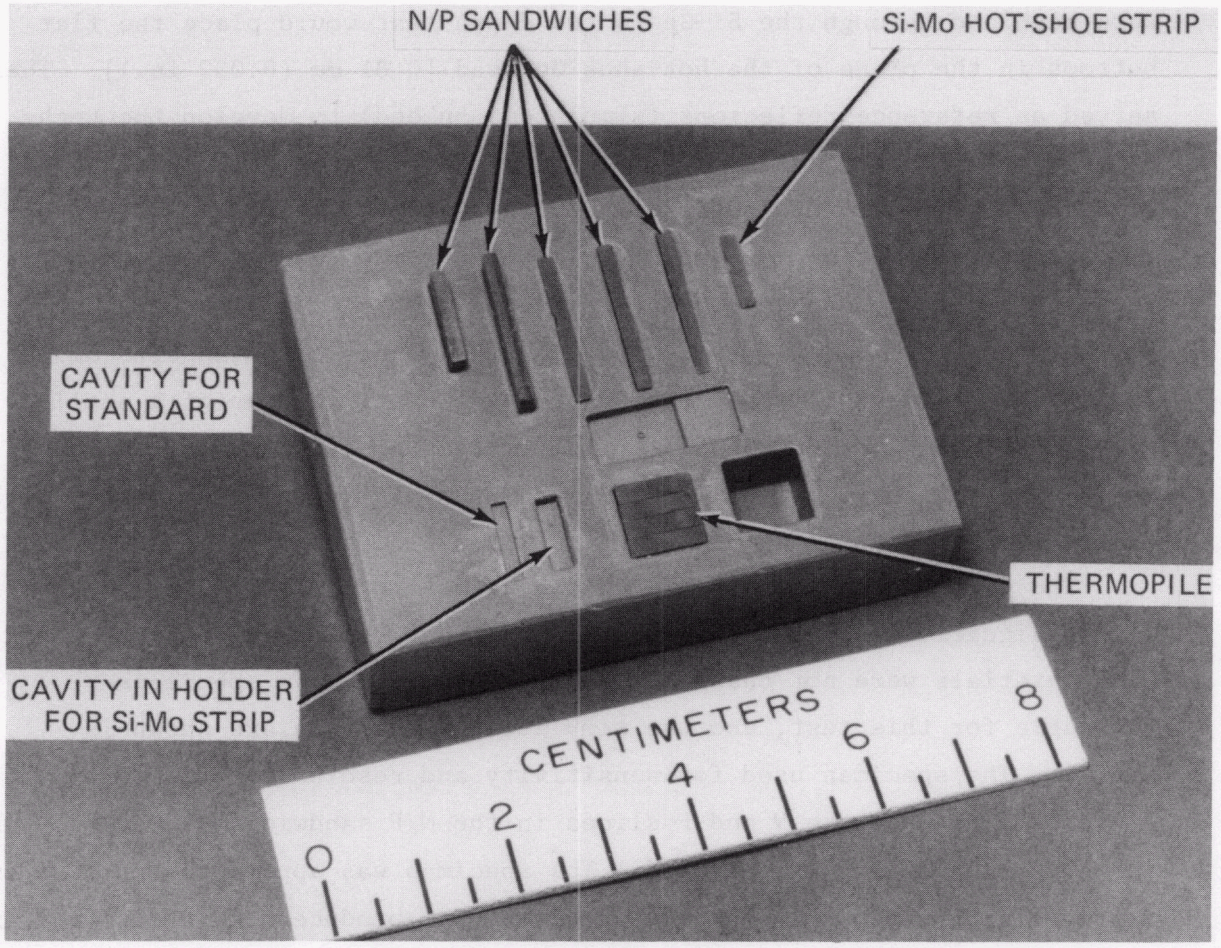

Fig. 7. Rubber holder for the germanium bond inspection with pulse-echo ultrasonic immersion techniques.

with special-order, focused ultrasonic search units. One 25-MHz unit with a 25-mm (1-in.) focal length and a 0.24-mm (0.009-in.) focal spot size had the best performance. The sensitivity and resolution studies were performed on a specimen machined from a five-couple slice subcomponent. This standard would be located in the cavity between a strip of Si-Mo (also removed from its cavity) and a thermopile in the holder shown in Fig. 7 (near the scale in the photograph). This was a reasonable substitute since the accessibility and ultrasonic technique were anticipated to be very similar for the hot-shoe bond at each of these two stages of fabrication. All but 0.81 mm (0.032 in.) of the length of the Si-Ge N and P legs

was removed from the selected five-couple slice. Three flat-bottom holes were machined through the Si-Ge to the depth that would place the flat bottoms in the plane of the hot-shoe Ge bond [0.81 mm (0.032 in.)]. These served as reference reflectors (simulated nonbond) to develop the technique and determine sensitivity. The diameters of the holes were 0.25, 0.38, and 0.89 mm (0.010, 0.015, and 0.35 in.). With the 25-MHz inspection system with 5× map recording, all three simulated nonbonds were detected and recorded. On this calibration sample, the presence of smaller reflected signals [i.e., smaller than those from the 0.25-mm (0.010-in.) reflector] would have prevented identification of a smaller hole [e.g., 0.12 mm (0.005 in.)]. The small signals could be caused by the acoustic mismatch of the dissimilar materials in the bond or by small discrete discontinuities.

The fact that the available specimens were of prehybrid design has little significance for the Ge bond development because the hot-shoe and bond materials were not changed. We evaluated the five specimens available for this test, and the simulated C-scan data are shown in Fig. 8. The specimen used for sensitivity and resolution studies of the glass bond between the N and P slices in the N/P sandwich was the only one in which nonbonding was detected. The specimen was apparently cracked in the machining process in an initial attempt to produce simulated flaws in the hot-shoe bond. Visual examination suggests that the crack propagates through the Si-Mo shoe in the thickness direction, changes direction near the bond line, penetrates into the sandwich, and then runs nearly parallel to the bond line at a depth of about two to three times the hot-shoe thickness. Thus, as expected, we detected a small area of nonbond in the bond line area where the crack penetrates to the N/P slices.

FIVE-SANDWICH STACK

Five of the N/P sandwiches with hot-shoe bonds are combined with four new glass bonds to produce a five-sandwich stack with nominal dimensions of 19 × 8.59 × 8.28 mm (0.75 × 0.338 × 0.326 in.). In the original recommendations, nondestructive examination techniques (other than visual examination) were not to be performed. It was felt that better examination could be performed at the next stage on the thinner sections

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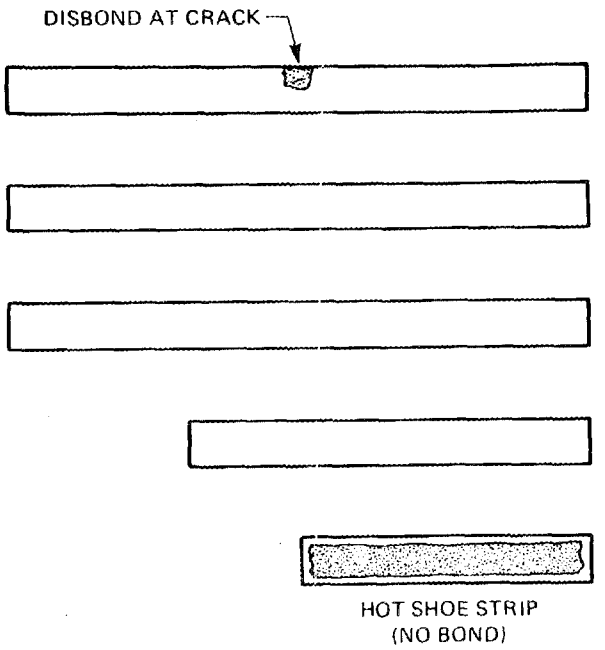

Fig. 8. C-scan ultrasonic data for germanium bond inspection for N/P sandwich subcomponents. The rectangular boxes outline the approximate 5× area occupied by the N/P sandwiches and the hot-shoe strip.

of the five-couple slice. However, the very low X-ray attenuation by the inter- and intracouple glass indicated that ultrasonics would be preferable. The ultrasonic method can be applied more efficiently through the larger areas of the five-sandwich stack using the same high-frequency ultrasonic capabilities with electronic gating and 5× recording. The five-sandwich stack is mounted in a molded rubber holder (very similar to those shown earlier) to maintain the alignment of the ultrasonic beam. The focused ultrasonic beam (from search units procured for this examination) impinges on the major surface of the uppermost thickest slice and an x-y raster scan is performed. Because of the depth of the specimen [0.828 mm (0.326 in.)] as the focused beam is transmitted through the nine glass bonds, it is not possible to establish peak focusing at all levels simultaneously. Therefore, several ultrasonic examinations are required with the ultrasonic beam focused at different depth zones. Figure 9 illustrates the condition where focusing is near bond levels 8 and 9 from the surface of entry. If the glass bonds were examined at the N/P

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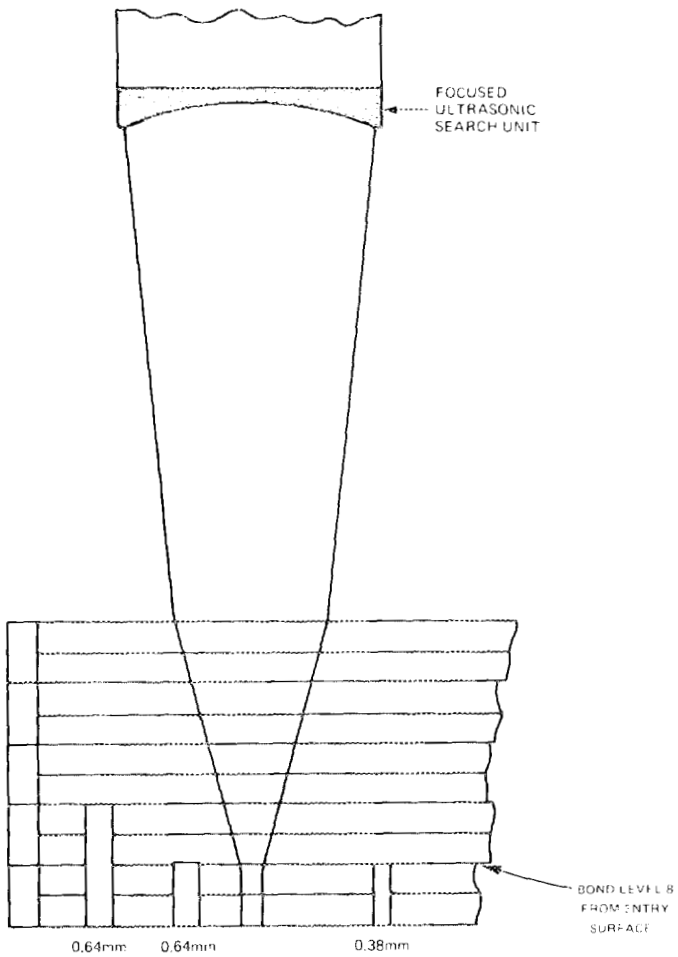

FIVE - SANDWICH STACK CROSS SECTION
WITH THREE DIFFERENT HOLES

Fig. 9. Cross section of a five-sandwich stack with ultrasonic beam focused near bond levels 8 and 9.

sandwich stages, only the four new bonds between N/P sandwiches would need examining. An old (prehybrid) specimen has been used in the study. Flat-bottom holes (nonbond simulators) were machined into the specimen to provide reflecting surfaces in several of the bond lines. The sizes include 0.25-, 0.38-, and 0.64-mm (0.010-, 0.015-, and 0.025-in.) diam. With the available specimen it appears that sensitivity to 0.25-mm-diam (0.010-in.) nonbonds may be possible in the upper half of the scanned specimen (through the first four inter- and intraleg glass bonds). Because of the interaction of the beam passing through each bond, sensitivity in the lower half of the specimen may be reduced to 0.38 mm (0.015 in.). Of

course, if the 0.25-mm (0.010-in.) level of sensitivity is desired, the specimen could be turned over, allowing all bonds to be near the search unit for the entire examination. Because of the many bonds in this specimen, special care and gating techniques are necessary to avoid ambiguity and confusion. If desired, the hot-shoe bonds could also be examined at this stage of fabrication using the techniques discussed earlier. However, if examined at the earlier stage, there should be no need for reexamination at the five-sandwich stack stage.

FIVE-COUPLE SLICE

The five-sandwich stack is sliced into a number of five-couple slices each 2 mm (0.080 in.) thick and containing five Si-Ge N and P pairs with hot shoes. Original recommendations were for nondestructive examination of the four new glass bonds (introduced during fabrication of the five-sandwich stack) and the germanium hot-shoe bond for potential damage introduced during slicing. Ultrasonic techniques are to be developed as a means of confirming and backing up radiography studies.

Glass Bonds

The most practical technique transmits the ultrasound perpendicular to the plane of the glass bonds. However, because of the narrowness [2 mm (0.080 in.)] of the specimen, effective focusing (required for adequate resolution) cannot be obtained below the first two bond lines. For that reason we initiated the previously discussed activities developing techniques for the five-sandwich stack because of the improved capability for application of the focused ultrasonic beam through the large area of the Si-Ge slice.

Hot-Shoe Bond

Except for the design and fabrication of the molded rubber holder (shown in Fig. 10) for the five-couple slice, the ultrasonic technique and attainable sensitivity for the examination of the hot-shoe bond is the same as that discussed earlier for examination of the hot-shoe bond on the N/P sandwich. The technique has been applied to several rejected specimens with good success. Figure 11 shows the 5× C-scan data for five

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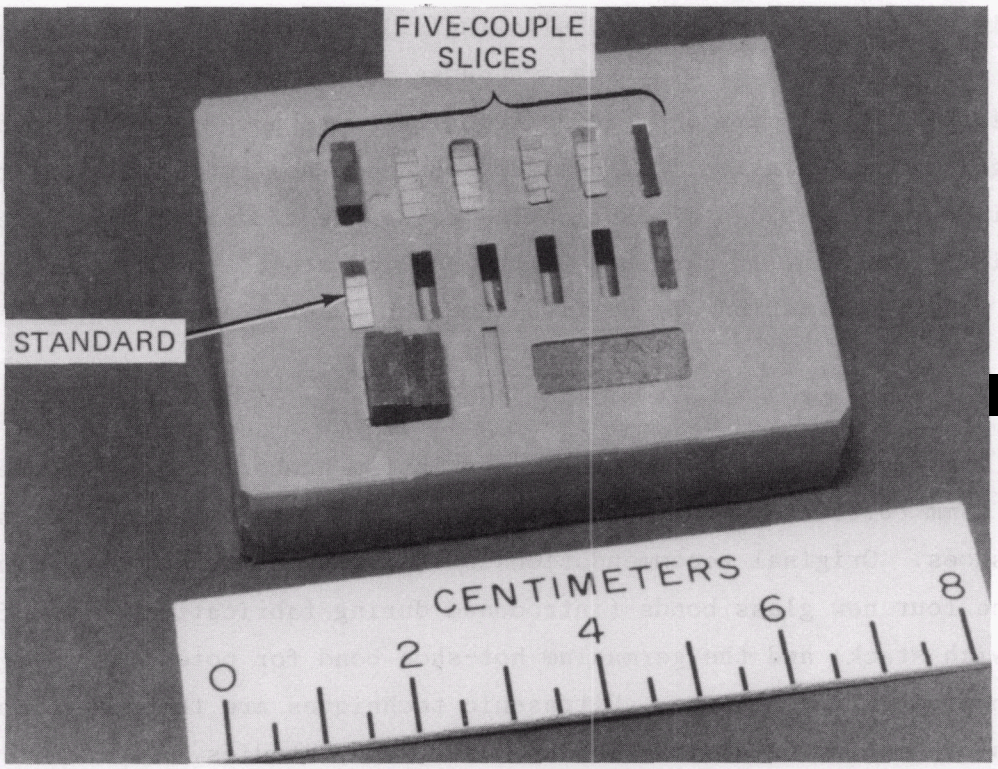

Fig. 10. Holder for five-couple slices, one thermopile, and calibration specimens.

of the specimens, along with calibration standards. Most of the specimens displayed indications equivalent to the 0.25-mm (0.010-in.) reference; two indications were as large as that from the 0.89-mm (0.035-in.) reference. Metallographic sectioning was performed on two specimens, and results are discussed below.

Metallography

Two five-couple slices (specimens 11 and 12) were selected for metallographic analysis. Each contained areas of nonbond detected in the hot-shoe bond both by ultrasonics (Fig. 11) and by radiography. The first specimen (11) was used to establish the procedure for cutting, mounting, and polishing. In Fig. 11, sample 11 is not outlined as clearly as the other samples, probably because of a slight misalignment in the holder. The nonbond detected by both ultrasonics and radiography was observed and

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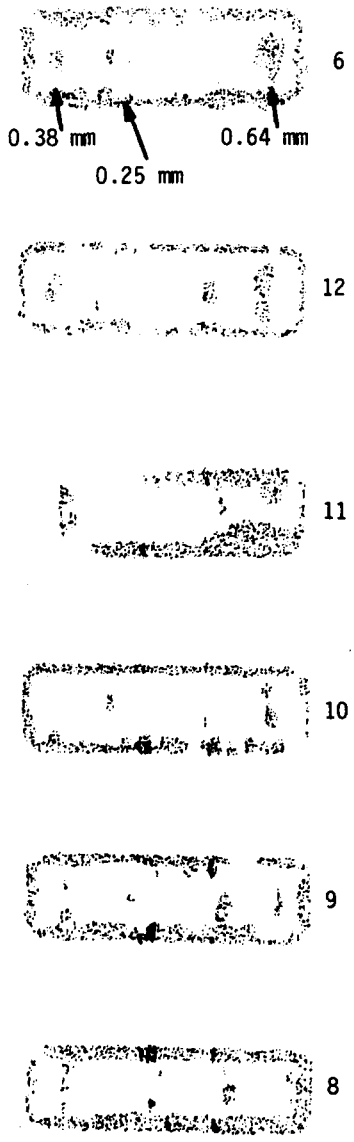

Fig. 11. Ultrasonic data on hot-shoe bond of five-couple slices. The standard is sample 6 and contains 0.25-, 0.38-, and 0.64-mm-diam (0.010-, 0.015-, and 0.025-in.) holes.

confirmed by scanning electron microscopy. The second sample (12) was subjected to sequential polishing and photographic documentation in 0.05-mm (0.002-in.) increments for a total length of about 0.66 mm (0.026 in.). Scanning electron microscopy again confirmed the void in the bond. Excellent correlation was obtained with the location and size predicted by the nondestructive examination. A letter report documenting this correlation was prepared and distributed to General Electric Company. This report is Appendix C.

THERMOPILE ASSEMBLY

Four of the five-couple slices are joined together with three new glass bonds to produce the thermopile assembly with nominal dimensions of 8.28 × 8.43 × 8.28 mm (0.326 × 0.332 × 0.326 in.). If the other bonds have been examined, the principal need is to evaluate only the new glass bonds. Ultrasonic techniques developed for the five-sandwich stack are similar to those needed for the thermopile. The principal differences are in the need for a calibration standard made from a thermopile and the fine tuning in the ultrasonic equipment because of the different dimensions (depths below the ultrasound entry surface) and the effect on focusing. A new hybrid thermopile that probably can be used for the preparation of the hybrid thermopile calibration standard was recently received from General Electric Company.

HEAT COLLECTOR/THERMOPILE BONDED ASSEMBLY

The next stage in fabricating the multicouple is bonding the thermopile assembly to the graphite heat collector with a high-temperature glass with a nominal thickness of 0.05 mm (0.002 in.).

Ultrasonic examination of the hot-side glass bond is performed by transmitting a focused ultrasonic beam from the cold end along the Si-Ge legs to the hot end where nonbonds cause reflections of ultrasound back through the Si-Ge to the search unit. The machined configuration and acoustic properties of the graphite heat collector prevent examination from the heat collector side. Ultrasonic studies with the improved ultrasonic instruments and recording system established optimum

operating parameters of approximately 18 MHz with a 20-MHz search unit, a 51-mm (2-in.) focal length, and a 0.51-mm (0.021-in.) focal spot. The attenuation in the Si-Ge prevents the use of higher frequencies. Similar techniques can be applied to examine either the germanium hot-shoe bond or the hot-side glass bond, with the principal difference being in the calibration standard and the setting of the electronic gate to compensate for the 0.81-mm (0.032-in.) difference in distance from the entry surface to the bond being examined. Attainable sensitivity for either bond (based on studies in Si-Ge calibration standards) is for nonbonds equivalent in area to 0.31 mm (0.012 in.) in diameter. The ultrasound passes through the germanium hot-shoe bond before reaching the hot-side glass bond. Therefore, if there is a nonbond in the hot-shoe bond, the sound will be reflected rather than transmitted. Those areas of the glass bond directly under a nonbond in the hot-shoe bond would therefore not be examined. Initially, specimen holders (plastic) were made to hold several heat collector/thermopile bonded assemblies in proper alignment during scanning; however, with the current 44-mm scanning capability, only one specimen and the calibration standard can be scanned and recorded at 5× because of the large area occupied by the heat collector. Ultrasonic scanning requires an immersion bath (usually water) to couple the ultrasound between the search unit and the specimen. Inquiry was made to General Electric Company about the acceptability of immersing the graphite heat collector in water, and it was determined that distilled water would be acceptable.

COLD-END CONTACTS

A tungsten-foil cold-end contact [with a thickness of 0.0025 mm (0.001 in.)] defining the electrical circuit pattern is bonded to the cold end of the thermopile with a silver plating. Experimental work for nondestructive examination of the bond quality has not been performed. Specimens that are representative of the current fabrication method have been requested. High-resolution ultrasonics (necessary because of the very small thickness) are being considered. The necessary high-frequency ultrasonic instrumentation (e.g., 100-MHz capability) has been ordered for

this (and other applications) but has not been delivered (original expectation of delivery was June 1985), and these factors have prevented the desired investigation.

POWER LEAD ATTACHMENTS

The two power leads [0.38-mm-diam (0.015-in.) nickel-clad molybdenum wire] are brazed into two holes [0.41-mm diam by 1.27 mm deep (0.016 by 0.05 in.)] drilled into an N and P leg, respectively, near the center of the cold side of the thermopile. An initial investigation has been conducted with ultrasonics to determine the feasibility of examining the power lead attachment.

An initial investigation was made using ultrasonics to detect nonbonding of the power lead attachments in one of the available thermopiles. Both 20- and 25-MHz focused search units were used to compare the reflection from the side of drilled holes to the reflection from the hole containing the power lead attachments. A 0.5-mm (0.021-in.) side-drilled hole, which served as a reference reflector, was placed in the same Si-Ge leg containing a power lead attachment with a radiographically detected void. Ultrasound detected the void found by radiography; however, the glass bond between the Si-Ge legs adjacent to the power lead attachment produced a reflected signal approximately as large as that from the 0.5-mm (0.021-in.) drilled hole (the reference reflector) and so close in time that electronic separation of the signals was difficult. If such interface signals are present in production multicouples, establishment of such a technique will be difficult, and the effectiveness may be questionable.

RECOMMENDATIONS

Several different (albeit similar) ultrasonic examination techniques have been investigated and developed for evaluating various bonds in the subcomponents of the multicouples. The bonds examined include glass bonds between the N and P Si-Ge, germanium hot-shoe bonds, hot-side glass bonds, cold-end contacts, and power lead attachments. Most of the development activity has been conducted on rejected prehybrid subcomponents supplied

by General Electric Company (but in general the specimens were not rejected because of bonding problems). We are convinced that these techniques should be applied by ORNL to a larger number and variety of specimens as part of pilot plant production. The specimens could represent a statistical sampling of the entire production run, depending on the total volume. This approach would allow additional confirmation and validation of techniques and would supply feedback concerning the fabrication process. The most information could be obtained by applying each of the techniques that have been described in this report at each fabrication stage. Large-scale application of these methods would allow more informed decisions to be made on optimum techniques and time of application for full-scale production. However, if logistical consideration and schedules preclude full-scale application, the following subsections outline a compromise position based on technique developments to date.

GLASS BONDS BETWEEN N AND P Si-Ge

The most sensitive examination at this time for this bond is ultrasonic examination at the N/P sandwich stage, and this is recommended for process control. The next step for glass bonds is the five-sandwich stack where four new parallel glass bonds are added, and the ultrasonic technique is again recommended. If examination at the N/P sandwich stage is deemed logistically impractical, both the inter- and intraleg glass bonds can be evaluated in the five-sandwich stack but with less sensitivity than is attainable in the N/P sandwich. The last of these glass bonds is added in the thermopile assembly, and ultrasonic techniques are recommended.

GERMANIUM HOT-SHOE BONDS

Ultrasound provides useful examination of the hot-shoe bond and should be applied during the pilot steps of fabrication. Ultrasonic examination provides a map showing the location of discontinuities within the bond area. The optimum stage in the fabrication process for examination is on the N/P sandwich with hot-shoe bond (the earliest stage) for process control information. If there are sufficient logistical or

scheduling benefits in delaying the examination, the ultrasonic technique can be applied to the hot-shoe bond at the five-sandwich stack stage or, as a secondary choice, on the five-couple slice. If slicing damage is surface-related, it should be detectable by the 10× magnified visual examination. If damage is completely internal (not extending to an exterior surface), ultrasonic examination would be in order.

HOT-SIDE GLASS BONDS

Ultrasound is recommended for the examination of the hot-side glass bonds following the fabrication-stage bonding of the heat collector to the thermopile. Ultrasonic techniques provide data on the location and size of nonbonds within the area of the bond (of interest for process control and particularly useful during early production stages).

BULK MATERIALS

Ultrasonic techniques should be developed and applied for the characterization of Si-Ge and Si-Mo materials at the compact or slice stage to ensure uniformity of other measurable properties. For example, ultrasonic techniques that have been developed and demonstrated on compact sections of Si-Ge should be modified as necessary to determine variations in elastic properties (variations were observed in limited available compact samples).

SUMMARY

Drafts of nine different ultrasonic procedures have been prepared. These procedures list the parameters and steps that must be established before testing is performed on production assemblies. Appendix B contains one of the procedures provided to the U.S. Department of Energy for possible use in the MITG Program.

ACKNOWLEDGMENTS

The encouragement and support of W. J. Barnett, DOE Division of Special Applications, is gratefully acknowledged. The authors thank R. S. Crouse, B. E. Foster, and G. C. Marsh for their contributions to

the destructive testing analysis. We thank B. E. Foster and M. M. Martin for their discussions and for reviewing the manuscript, and J. L. Bishop for her assistance in compiling the manuscript. We also thank P. G. Greeson and O. A. Nelson for editing and M. R. Upton for the final preparation of the report.

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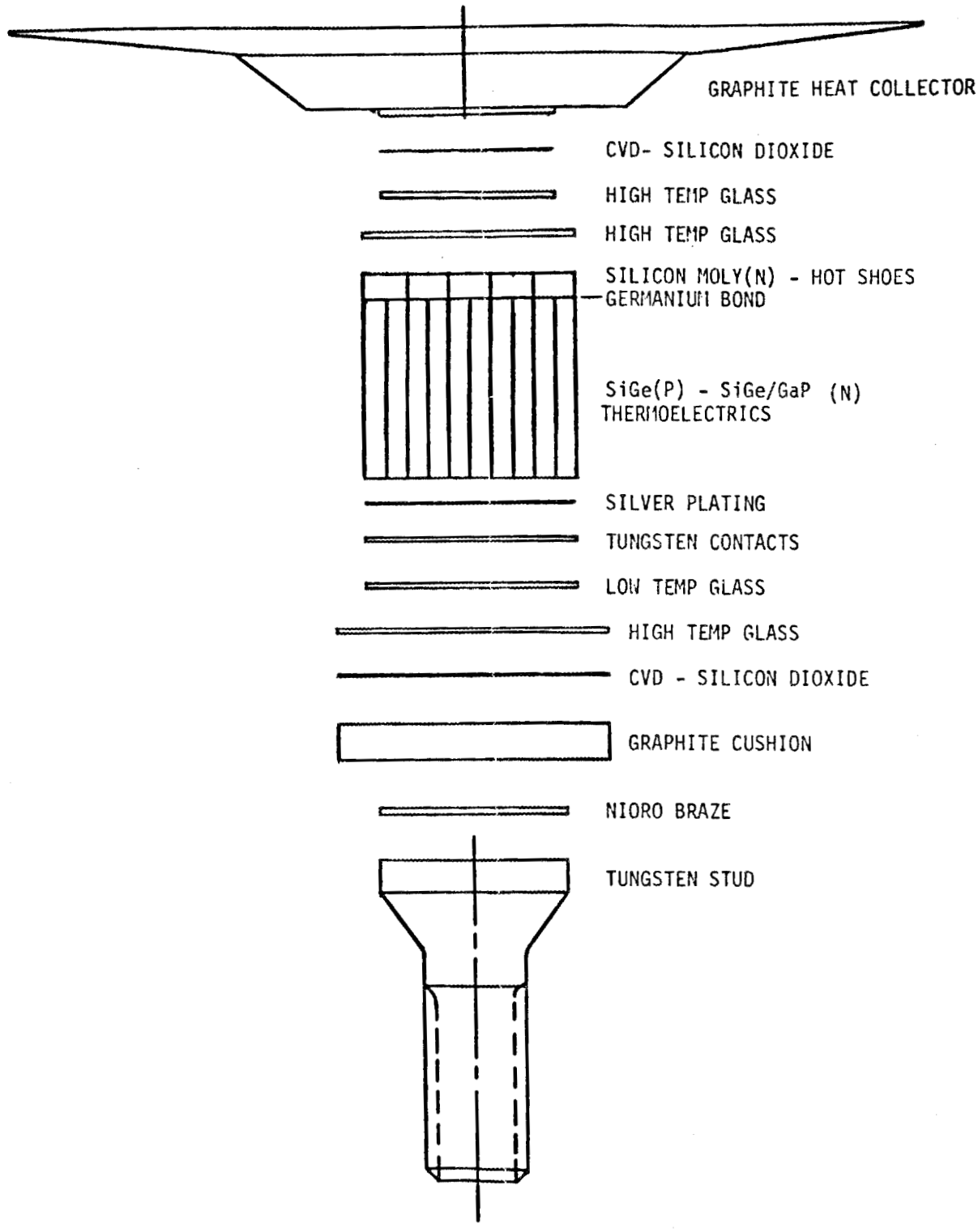
1. G. L. Bennet, J. J. Lombardo, and B. J. Rock, "U.S. Radioisotope Thermoelectric Generators in Space," *The Nuclear Engineer* **25**(2), 49–58, (March–April 1984).

2. A. Schock, "Design Evolution and Verification of the General Purpose Heat Source," Paper 809203 in *Proceedings of the 15th Intersociety Energy Conversion Engineering Conference*, Aug. 18–20, 1980, Seattle, American Institute of Aeronautics and Astronautics, New York, 1980.

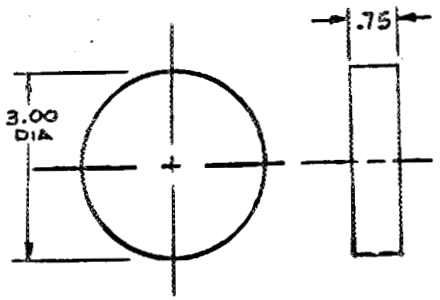
Appendix A

SUBCOMPONENT AND ASSEMBLY DRAWINGS FOR THE MODULAR ISOTOPIC THERMOELECTRIC GENERATOR DESIGN

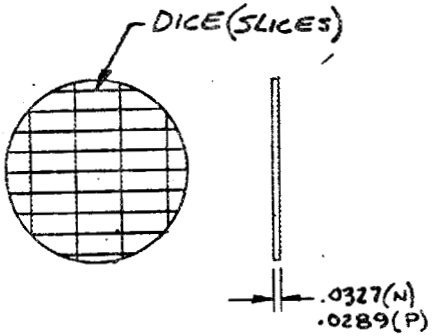
(Prepared by General Electric Company)



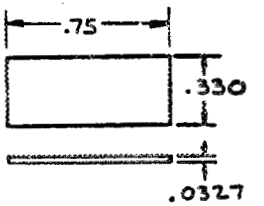
MULTICOUPLE INTERFACES



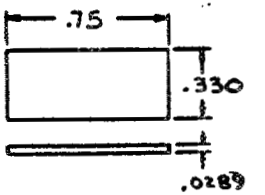
SiGe/GaP COMPACT
'N' & 'P' TYPE



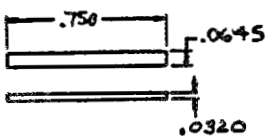
WAFER (N & P)



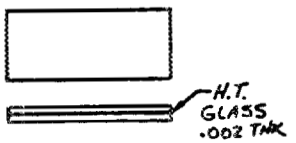
'N' SLICE
47C306536



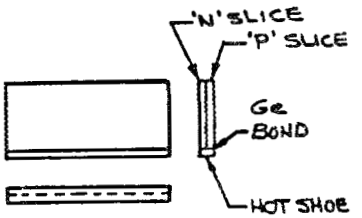
'P' SLICE
47C306537



HOT SHOE
47C306535



N/P SANDWICH
47C306538



HOT SHOE BOND
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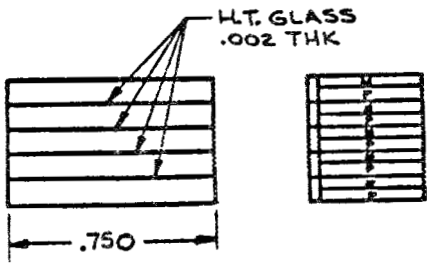
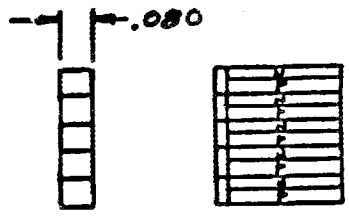
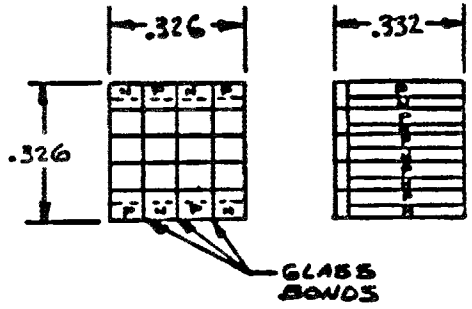


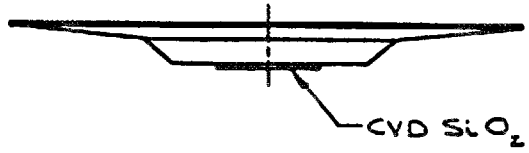
FIVE SANDWICH STACK
47C306542



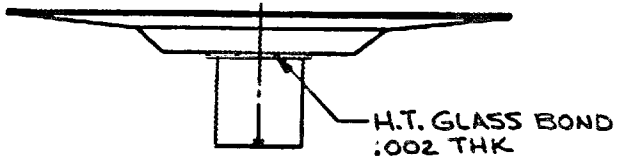
FIVE COUPLE SLICE
47C306540



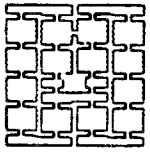
THERMOPILE ASSY
(CONTACTED HOT SIDE)
47C306541



HEAT COLLECTOR
47D306510



HEAT COLLECTOR/T/P
BONDED ASSY
47C306544



COLD END CONTACTS
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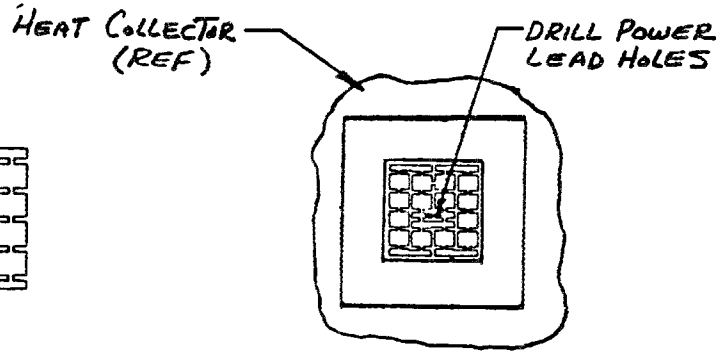

CONTACT BOND
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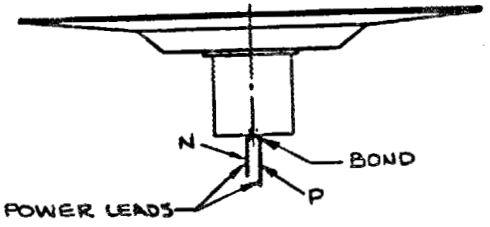

POWER LEAD ATTACH.
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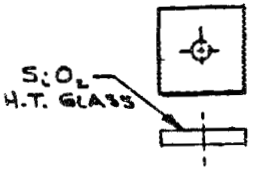
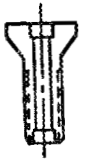


CUSHION
47B306690

BRAZE
SHIM
47B306556



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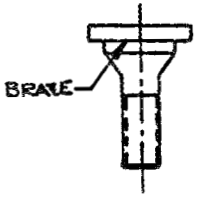
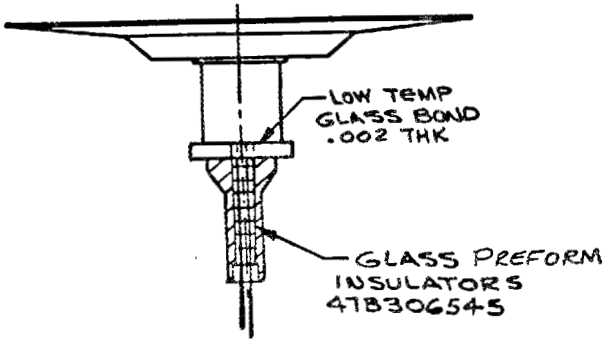
47C306512



CUSHION/STUD ASSY
47C306548



MULTICOUPLE PRE-ASSY
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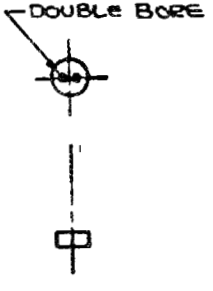
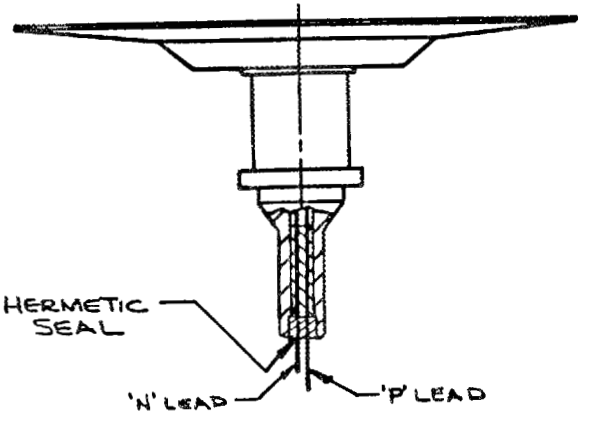


GLASS PREFORM
47B306545



MULTICOUPLE ASSY
47C306550

Appendix B

DRAFT PROCEDURE FOR ULTRASONIC INSPECTION OF N/P SANDWICH ASSEMBLY FOR LAMINAR DISCONTINUITIES IN GLASS BOND

D R A F T

Nondestructive Testing Laboratory Metals and Ceramics Division OAK RIDGE NATIONAL LABORATORY Oak Ridge, Tennessee 37831	Procedure No. MET-NDT-MITG-UT-1 Revision No. Draft Date September 1985 Page 1 of 3

PROCEDURE

Title: ULTRASONIC INSPECTION OF N/P SANDWICH ASSEMBLY FOR LAMINAR DISCONTINUITIES IN GLASS BOND

Prepared by: K. V. Cook

Approved by: R. W. McClung, Group Leader; M. M. Martin, Program Manager; W. H. Miller, Quality Assurance

A. SCOPE

This procedure outlines the steps necessary for the ultrasonic evaluation of glass bonds between the silicon-germanium (SiGe) N-type and SiGe P-type slices that are used to fabricate the N/P sandwich assembly. This assembly is a subcomponent for a multicouple fabricated in accordance with General Electric* Drawing 47C306538, Revision B. The inspection is to be performed with a pulse-echo immersion flaw detection technique using a focused search unit. The ultrasonic inspection beam shall be introduced perpendicular to the major surface of the N slice [i.e., the 19- by 8.3-mm (0.075- by 0.327-in.) area]. Focusing of the search beam is maximized on the nominal N-slice-to-glass interface and laminar flaws in the bond zone of the glass provide reflections with amplitudes dependent on size. Evaluation of each glass bond shall be based on the interpretation of C-scan (plan-view) recordings by comparing them with simultaneously generated C-scan data for a proper reference containing simulated flaws.

B. SURFACE CONDITION

All surfaces shall be clean and free of dirt, grease, or other foreign material. Alcohol shall be used to rinse parts after ultrasonic flaw detection tests in clean demineralized water.

*King of Prussia, Pennsylvania.

PROCEDURE

C. EQUIPMENT

1. The instruments and accessory equipment shall be of the pulse-reflection type with sufficient sensitivity and response (including variable time and amplitude electronic gating) to distinguish the reference holes as required during inspection. In addition, a real-time spectrum analysis system shall be used to establish the inspection frequency (as a minimum for the original inspection system assembly).

2. A C-scan (plan-view) recorder capable of at least five-to-one recording shall be used to document these results. Recording scan-line indexing shall be 0.026 mm (0.003 in.) or less.

3. A special holder shall be used to hold as many as five hot-shoe-to-N/P-sandwich assemblies plus the proper standard for the inspection. The x-y area of scan is currently limited to 51 by 51 mm (2 by 2 in.) in order to maintain a 5× capability; therefore multiple holders may be utilized to provide semiproduction capabilities. The nominal 19- by 8.3-mm (0.75- by 0.327-in.) surfaces of the N slices shall be precisely located (via a 50× microscope) in respect to each other (i.e., the major surfaces exposed to the ultrasonic beam and immersion fluid shall be parallel). In addition, the holder must maintain a precise horizontal plane position in the ultrasonic couplant fluid so that a constant water column is maintained [within about 0.25 mm (0.010 in.) over the entire x-y scan field].

4. An ultrasonic scanner with 0.025-mm (0.001-in.) indexing capabilities and variable scanning speeds shall be used in conjunction with an immersion tank (containing distilled water).

D. CALIBRATION STANDARDS

1. A calibration standard made from an appropriate subcomponent shall be used.

2. The primary reference shall be a 0.25- ± 0.05-mm-diam (0.010- ± 0.002-in.) flat-bottom drilled hole, machined with a diamond-tipped drill to a depth that locates the bottom at the nominal glass-to-slice interface for the N slice.

3. In addition to the primary reference hole, two flat-bottom holes shall also be machined in the reference with the same nominal depth as the 0.25-mm hole. These holes shall have diameters of 0.13 and 0.66 ± 0.05 mm (0.005 and 0.026 ± 0.002 in.).

PROCEDURE

E. EQUIPMENT CALIBRATION

1. The equipment shall be calibrated at the same indexing steps and longitudinal speed that will be used for the inspection.

2. The sensitivity of the equipment shall be established to show easily discernible indications from the reference holes on a plan-view recording.

F. INSPECTION PROCEDURE

1. Each sample shall be completely scanned for discontinuities comparable to the reference hole. A 5× or greater plan-view recording of test results will be made for each inspection.

2. Since alignment of samples, holders, and transducer is critical, edge recording due to the holder surface can occur. Small bubbles can also produce false indications of flaws. If either one occurs, the subcomponent may be subsequently retested at the discretion of the Level II or Level III inspector.

3. A focused transducer operating near 25 MHz (established by utilizing real-time spectrum analysis) shall be used for this inspection. The focusing of the transducer shall be accomplished and maximized on the 0.66-mm-diam reference hole. The nominal focal distance of the transducer in water shall be 25 mm (1 in.).

4. Any time the primary reference hole is not recorded properly, recalibration shall be made and the test rerun.

5. Inspectors shall be certified in accordance with SNT-TC-1A, Supplement C.

G. REPORTS

1. A copy of the C-scan inspection data shall be provided for insertion into an inspection data package (as required by General Electric).

2. Copies of inspection records shall be retained in an identified file cabinet for 1 year by the Nondestructive Testing Group.

Appendix C

LETTER REPORT DETAILING RESULTS OF EXAMINATION OF GERMANIUM BOND IN FIVE-COUPLE SLICES WITH RADIOGRAPHY, ULTRASONICS, AND METALLOGRAPHY

Letter 0528-08-85

INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

May 28, 1985

To: R. W. McClung

From: B. E. Foster
K. V. Cook
R. S. Crouse

Subject: **Examination of the Germanium (Ge) Bond in Five-Couple Slices with Radiography, Ultrasonics, and Metallography**

Following the visit of GE and DOE personnel in late January, we received approval to pursue metallographic analysis of the Ge bond in one or two five-couple slices for correlation with specific ultrasonic and radiographic data. This study was mutually desired to identify the type of indications (apparent nonbonds) that were being detected by ultrasonics and radiography.

Radiography

The radiographic examination of the Ge hot-shoe bond in the five-couple slice is done with an energy of 125 kVcp. The samples are placed in the X-ray tube axis and displaced laterally from the beam centerline (as determined by a plumb line) approximately 9.5 mm (3/8 in.). After one exposure is made, the sample is turned over such that the side that was next to the film is then source side and a second exposure is made. Both views are interpreted for nonbond indication and one view usually shows significantly more indication of nonbond than the other view.

A total of eleven (11) five-couple slices were radiographed and the data is shown in Table 1. The difference in the length of apparent nonbond in the two views is caused by the X-ray beam being less parallel in one of the views. The Ge bond may not be precisely perpendicular to the sample surfaces or the surfaces may not be parallel, which could cause the change in parallelism. Samples 11 and 12 showed two areas with apparent nonbond that was significantly wider than other indications.

Table 1. Ge hot-shoe nonbond indications in samples of five-couple slices

ORNL assigned sample identity	Approximate length of apparent nonbond in respect to sample length (%)	
	View 1	View 2
1	30	90
2	60	20
3	40	20
4	10	90
5	10	60
6	30	95
8	70	85
9	20	80
10	30	60
11	95	75
12	40	80

Ultrasonics

The ultrasonic examination of the Ge-hot-shoe bond for delaminations was performed with a 25 MHz search unit. The search unit was focused at the Ge bond line [i.e., 0.76 mm (0.030 in.) below the SiMo entry surface] and an ultrasonic plan view ("C"-scan) recording was made at approximately 5X magnification. The five-couple slices were located in the center section of our modular rubber holder to maintain proper inspection location. Figure 1 (Photo 8505-84) shows the modular rubber holder with five-couple slices 6 and 8 located in the center inspection zone. Specimens 9, 10, 11 and 12 had been removed from the inspection zones and inserted in the thermopile location at the near left hand corner of Fig. 1 when this picture was made. The "C"-scan data (~5×) for these six specimens are shown in Fig. 2 (only the center inspection zone data is included).

Sample #6 is the standard specimen that contains simulated nonbonds. It was sectioned [leaving only the hot shoe and about 0.81 mm (0.032 in.) of the N/P sandwich] to allow subsequent machining of simulated nonbonds into the Ge hot-shoe bond. Three flat-bottom holes were machined through the N/P sandwich section to a depth of about 0.81 mm (0.032 in.). The flat-bottoms of these holes are positioned at depths near or in the Ge bond at the approximate physical center of a nominal 1.7-by 2.2 mm (0.067-by 0.087 in.) hot shoe section. A nominal 0.89-mm-diam. (0.035 in.) hole is located in the couple at one end of sample 6; a 0.38-mm-diam. (0.015 in.) hole is located in the couple on the opposite end, and a 0.25-mm-diam. (0.010 in.) hole is located in the couple adjacent to the 0.38 mm hole. All three holes can be detected and recorded as is evident in the top of

the recording in Fig. 2. The edges of all six specimens are outlined in black, and flaw indications fall within the sample outline. The data from Fig. 2 for specimens 12 and 11 were used to correlate with radiography and metallography results. The couple with the P-leg exposed is on the right hand side for specimen 12; whereas, for sample 11 the P-leg position is on the left hand side. Thus the metallography sections described later for both samples 12 and 11 (initiated from the P-leg positions) correspond to the long vertical black indication on the right side of number 12 data and to the intermittent black indications at the left side of number 11 (the outline box for sample 11 was not recorded, probably because of slight sample misalignment in the rubber holder).

Metallography

We selected sample 12 from the population of five-couple slices for metallographic analysis. This sample had shown a large nonbond area in the Ge bond with ultrasonics in the outer couple where the P-leg is outermost (see Fig. 2). Radiographically we had imaged an apparent nonbond in the same area (see Fig. 3).

Sample 11 contained a similar indication (both ultrasonically and radiographically) in the same general area but smaller in magnitude. We submitted this sample to the ORNL Metallography Laboratory so that the technique could be established to examine the Ge bond. There were no problems encountered in the mounting and polishing methods used.

Using the ultrasound printout as a guide, the assembly was mounted in epoxy resin so that the plane of polish would be parallel to the SiGe legs, and perpendicular to the Ge bond. The objective of the procedure was to produce planes of polish for photomicrography at nominal depth intervals 2 mils (0.002") apart. This would allow us to build a visual model of the void in the joint to correspond to the ultrasound and X-ray observations.

The mounted specimen was measured with a micrometer before the first grinding step to establish a base-line dimension. Grinding consisted of abrading the specimen on a series of wet silicon carbide papers with 180, 240, 320, 400, and 600 grit. This was followed by two polishing steps on vibratory polishers; 0.3 μm alumina on Texmet cloth and 0.5 μm diamond paste on nylon cloth. This provided the first polished plane at 5–10 mils. Each succeeding increment was removed by a very light grind on 600 grit paper, followed by the two polishing steps. This removed 0.002 in. each time. Photomicrographs of 50 and 100× magnification were made at each polishing interval to document the void pattern through the assembly.

Results

The nonbond detected by both ultrasonics and radiography was observed metallographically. Only three positions of test sample 11 (the one used

to establish the metallographic bond examination technique) were photographed (documented) and are shown in Fig. 4 (labeled S, T, and U). The locations were approximately 1.25, 1.3 and 1.35 mm (0.050, 0.052 and 0.054 in.) from the outer surface of the P-leg. It was clearly identified as a void with the scanning electron microscope (SEM) and is shown in Fig. 5.

Sample 12 received successive polish and photographic documentation from approximately 0.1 mm (0.004 in.) to 1.5 mm (0.060 in.) in nominal 0.05 mm (0.002 in.) increments. Figure 6 (positions labeled G through O) cover the area from 0.9 mm to 1.3 mm (0.032 to 0.052 in.). Obviously, these voids (detected and documented in excellent fashion by G. C. Marsh) correlated quite well with the nonbond indications previously noted with ultrasonics and radiography.

KVC:mr

Attachments

cc: K. V. Cook/File
R. S. Crouse/File
B. E. Foster/File
M. M. Martin
G. M. Slaughter

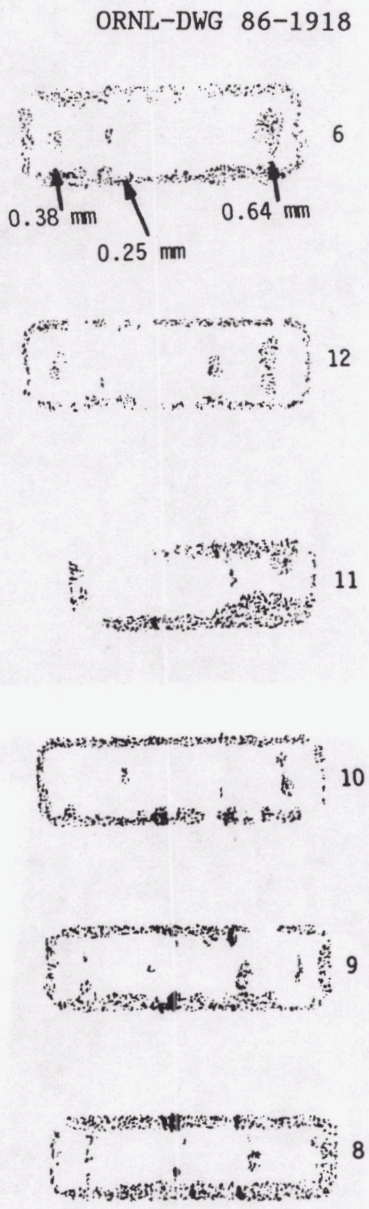


Fig. 1. Modular rubber holder used to position five-couple slices for ultrasonic inspection.

ORNL-PHOTO 8505-84

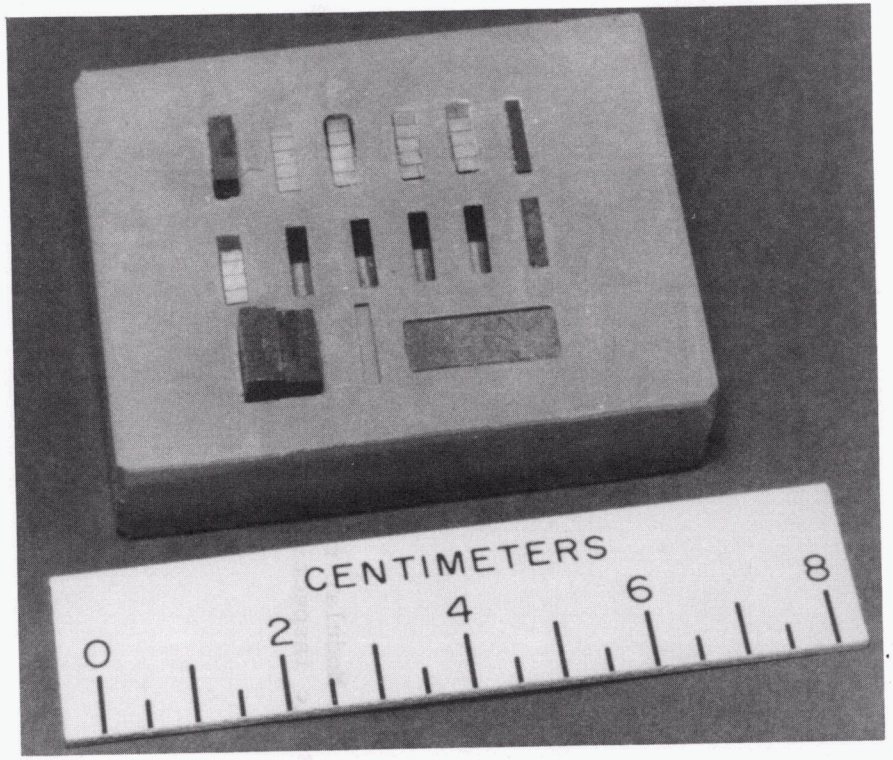


Fig. 2. Ultrasonic data on hot-shoe bond of five-couple slices 6, 12, 11, 10, 9, and 8 (top to bottom).

XP2412

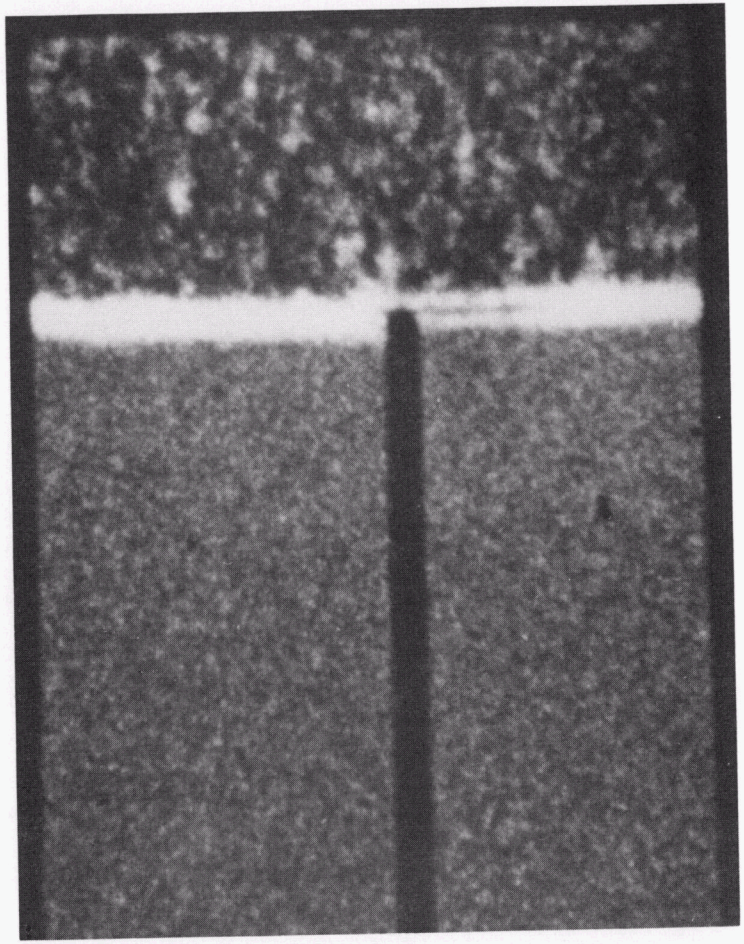


Fig. 3. Photograph enlargement (45×) of radiograph of sample 12 showing nonbond (black horizontal line) in Ge bond (white horizontal line).

Y201449

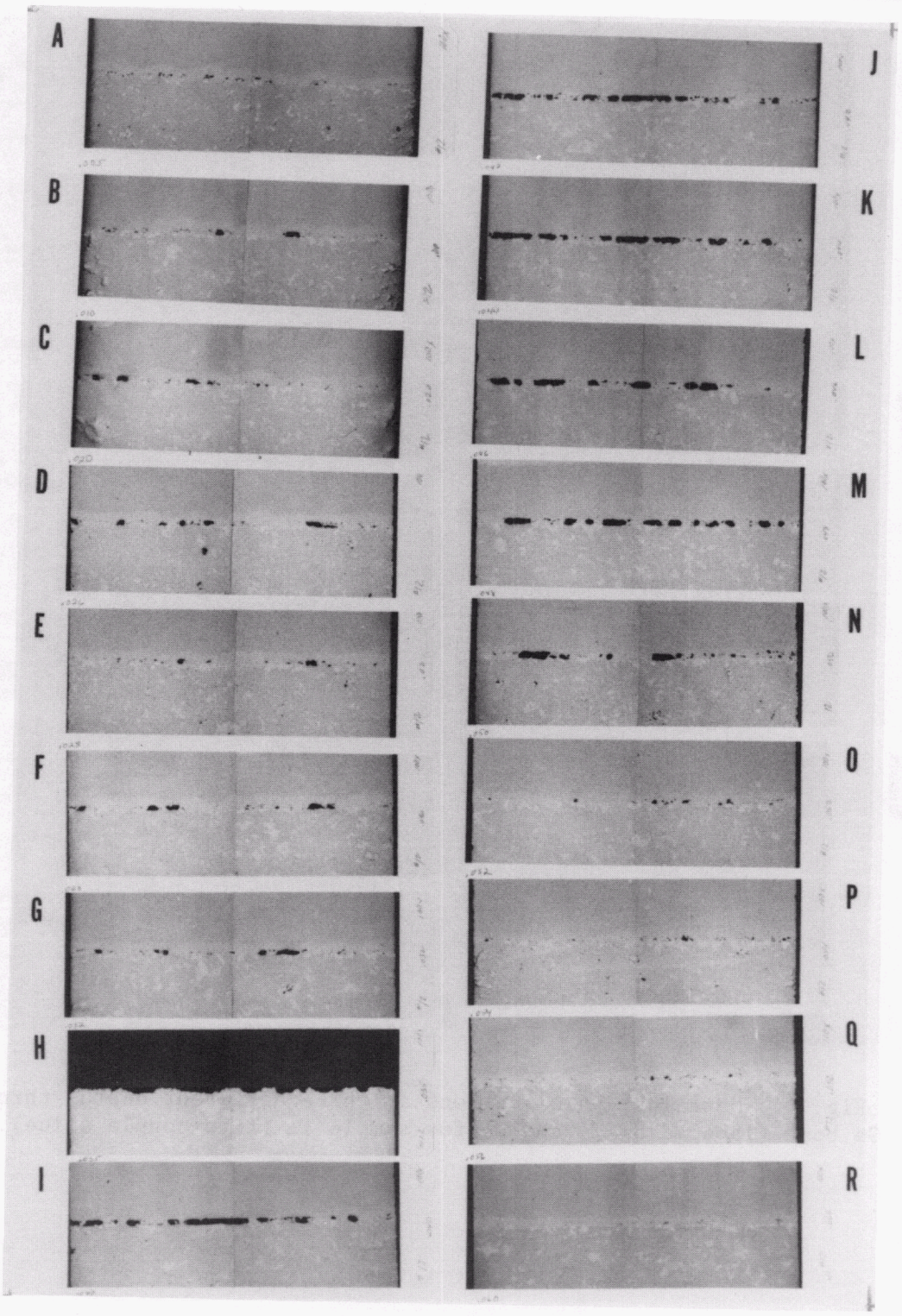


Fig. 6. Metallographic sections at different depths through the Ge bond (labeled A-R) for sample 12 (five-couple slice).

Y201448

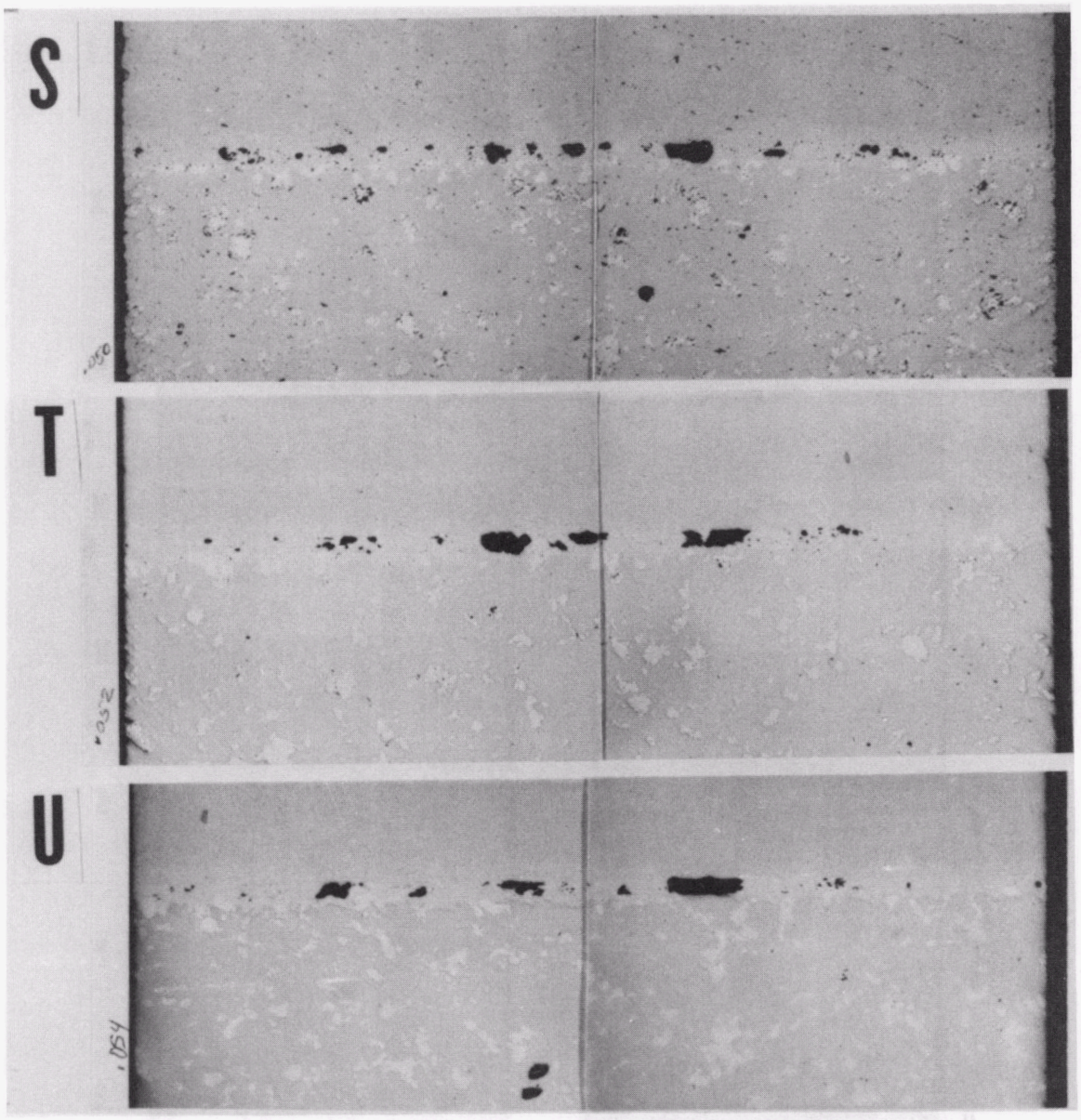


Fig. 4. Metallographic sections at three different depths through the Ge bond (labeled S, T, and U) for sample 11 (five-couple slice).

M-21164

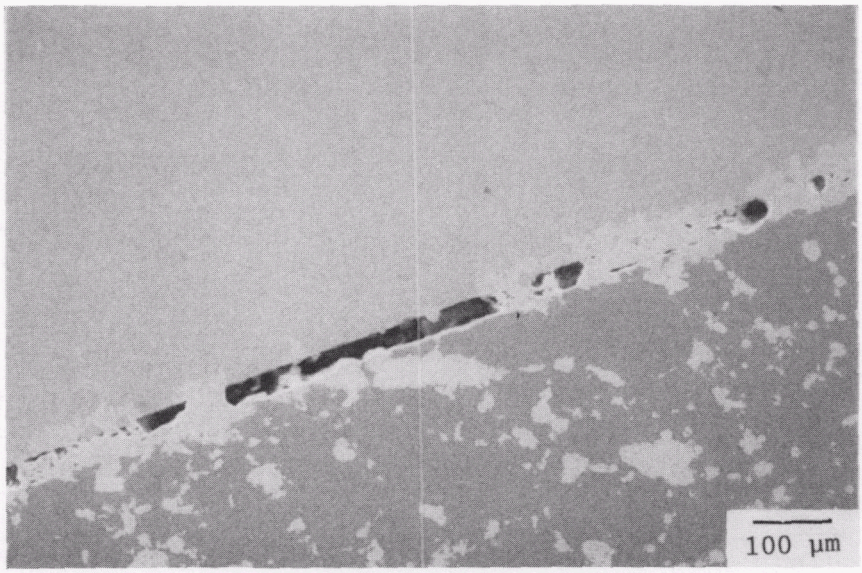


Fig. 5. Photograph of nonbond (identified as a void with the scanning electron microscope) in the Ge bond of sample 11 (five-couple slice).

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