725
7/15/69
AEC RESEARCH AND DEVELOPMENT REPORT

889
Y-1673
Instruments

MASTER

SODIUM AND STRONTIUM SHOCK PRESSURE TRANSDUCERS

R. L. Huddleston
R. E. Sladky

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the **ATOMIC ENERGY COMMISSION** *under* **U. S. GOVERNMENT Contract W-7405 eng 26**

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

REPRODUCTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Printed in the United States of America. Available from Clearinghouse for Federal
Scientific and Technical Information, National Bureau of Standards,
U.S. Department of Commerce, Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Date Issued: July 9, 1969

Document Y-1673

TID-4500

UNION CARBIDE CORPORATION
Nuclear Division

Y-12 PLANT

Contract W-7405-eng-26
With the US Atomic Energy Commission

SODIUM AND STRONTIUM SHOCK PRESSURE TRANSDUCERS

R. L. Huddleston
R. E. Sladky

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Oak Ridge, Tennessee

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Distribution:

Air Force Scientific Command

Meeks, E. J.

Atomic Energy Commission

Keller, C. A.

Brigham Young University

High Pressure Data Center

Brown Engineering, Huntsville

Sturm, R. G.

Harwood Engineering Company

Newhall, D. H.

Lawrence Radiation Laboratory

Bonicelli, M. P.

Dorough, G. D.

Gamble, Dean

Myers, W. B.

Stone, R. G.

Oak Ridge Gaseous Diffusion Plant

Jordan, R. G.

Vanstrum, P. R.

Paducah

Winkel, R. A.

Sandia - Albuquerque

Bild, C. F.
Classen, R. S.
Narath, A.
Shuster, D. B.
Smith, L. D.

Sandia - Livermore

Gutierrez, L.
King, J. C.

University of Georgia

Giardini, A. A.

Watervliet Arsenal

Davidson, T. E.

Y-12 Plant

Ballenger, H. F. (2)
Bell, B. B.
Bernander, N. K.
Briscoe, O. W.
Burkhart, L. E.
Ellingson, R. D.
Evans, G. W.
Foulk, D. L.
Haeusler, K. R.
Hemphill, L. F.
Huddleston, R. L. (5)
Jackson, V. C.
Kahl, K. G.
Keith, Alvin
Kite, H. T.
Marrow, G. B.
McLendon, J. D.
Mitchel, G. W. (3)
Oliphant, G. W.
Phillips, L. R.

Sladky, R. E. (5)

Smith, R. D.

Stoner, H. H.

Trotter, T. C.

Wallace, S. A.

Warner, J. F.

Waters, J. L.

Wesley, R. L.

Yaggi, W. J.

Y-12 Central Files (5)

Y-12 Central Files (route)

Y-12 Central Files (Y-12RC)

In addition, this report is distributed in accordance with the category Instruments, as given in the "USAEC Standard Distribution Lists for Unclassified Scientific and Technical Reports", TID-4500.

ABSTRACT

Eight shock pressure transducers have been developed and fabricated at the Oak Ridge Y-12 Plant—four with sodium metal as the pressure-sensitive grid and four with strontium metal. These transducers operate on the principle that significant changes in electrical resistivity occur with changes in pressure.

Final shock testing is to be performed at the Lawrence Radiation Laboratory.

CONTENTS

SUMMARY	8
INTRODUCTION	9
FABRICATION AND TESTING OF THE TRANSDUCER	11
Basic Design	11
Fabrication	15
Assembly and Testing	16
Assembly Procedure	16
Hydrostatic Pressure Test	19
APPENDIX A	26
Sodium and Strontium Disc Fabrication	26
Introduction	26
Sodium Discs	26
Strontium Discs	26
Strontium Ingot Preparation	27
APPENDIX B	30
Strontium Vapor Deposition Experiments	30
APPENDIX C	32
Shock Impedance Matching of Transducer Encapsulation	32
APPENDIX D	40
Fabricating Beryllium Oxide-Loaded Epoxy Cases	40
APPENDIX E	43
Sealing Copper Caps and Pins to a Beryllium Oxide-Epoxy Case	43
APPENDIX F	45
Assembly Procedures for Sodium and Strontium Transducers	45
Introduction	45

Sodium Transducer Assembly Procedure	46
Strontium Transducer Assembly Procedure	48
APPENDIX G	50
Brazing Strontium and Sodium Pressure-Sensing Elements to the Connecting Pins.	50
APPENDIX H	52
Fabrication Environment	52
APPENDIX I	55
Corrosion Tests	55
APPENDIX J	57
Gage Resistance Calculations	57
BIBLIOGRAPHY	59

SUMMARY

Eight shock pressure transducers were designed, developed, and fabricated in response to a need for a convenient device that will measure changes in pressure under dynamic pressure conditions. Four of the transducers use sodium as the pressure-sensitive element, the other four use strontium. Basic design and fabrication work was performed at the Oak Ridge Y-12 Plant; Shock testing is to be performed at the Lawrence Radiation Laboratory.

Considerable development work was necessary in an attempt to solve such problems as: (1) forming the pressure-sensitive grids; (2) preventing contamination and corrosion of the highly reactive metals; (3) finding the proper encapsulation and sealing materials, and (4) developing a suitable testing environment.

Preliminary reports from the LRL indicate that the strontium transducers worked quite well; however, some difficulty was experienced in obtaining clear signals from the sodium transducers.

INTRODUCTION

The sensing elements for shock pressure transducers are very similar in appearance to conventional foil-type strain gages except that the foils are thicker, are encapsulated in much thicker cases, and are pressure-sensitive rather than strain-sensitive metals. The transducers also have four electrical connections, two for pulsing current to the gage and two for measuring the voltage drop across the sensor, much like the principle of the Kelvin bridge.

The transducer design is based upon the fact that all materials undergo resistivity changes when sufficient pressure is applied. Bridgman characterized the high pressure-resistivity characteristics of a large number of materials. Ideally, a pressure transducer should be made of a material which will undergo large linear changes in resistance with pressure. A graph of a few metals which show promise in this respect is presented in Figure 1. Strontium shows a large, almost linear, positive change in resistance with increasing pressure. On the other hand, sodium shows a much smaller, nonlinear, negative change in resistance with increasing pressure.

The absolute voltage drop across the sensor element depends upon the current supplied to the sensor as well as the pressure-resistance characteristics of the metal. For sensors having a small cross section, there is a limit to the steady-state current that can be supplied to the sensor without causing resistance drift due to heat or sensor burnout. One means for circumventing this problem is to provide a pulse-current input to the sensor element that will be of sufficient duration to determine the voltage drop across the sensor when the shock propagates through the transducer. Using this technique, transducer sensitivity is greatly increased. Use of a pulse input requires that the shock transducers have a very low electrical impedance. Any mismatch in shock impedance (density times Young's modulus match or density times bulk modulus match are approximations to impedance matching) results in undesirable shock reflections which bring confusion to the transducer output data.

In the study described in this report, eight shock pressure measurement transducers were designed and fabricated, four having sodium sensing elements and four with strontium elements. The transducers were to be suitable to operate in a radiation environment. Attempts have been made throughout the design and fabrication phase of the transducers to satisfy as many of the ideal design criteria as possible; however, compromises have been made in order to expedite fabrication of the transducers.

Figure 1. RELATIVE RESISTANCE OF SEVERAL PRESSURE-SENSITIVE METALS AS A FUNCTION OF THE HYDRO-STATIC PRESSURE.

The main body of this report is devoted to describing the basic sodium and strontium transducer designs and the method for fabricating and testing the transducers. The remainder of the report is divided into a series of appendixes describing various phases of the development effort leading up to a successful fabrication of the sodium and strontium transducers. Final shock testing will be performed by the Lawrence Radiation Laboratory.

FABRICATION AND TESTING OF THE TRANSDUCER

BASIC DESIGN

Geometric details of all the as-fabricated sodium and strontium transducers are the same. However, the impedance characteristics of their encapsulations, the sensor metal, and the braze joint between the sensor metal and electrical connecting pins are different for the sodium and strontium gages. An outline of the grid design is shown by the drawing of Figure 2. The grid is made in a looped, low-inductance-type pattern very similar to commercial foil-type strain gages. Strontium or sodium cross sections in the grid area are 0.028/0.030 inch wide by 0.007/0.008 inch deep. The sodium or strontium depth goes to 0.023/0.024 inch in the pin connection area. Schematic drawings of the assembled sodium and strontium transducers are presented in Figures 3 and 4. Basic transducer design includes: (1) a pressure-sensor grid made of either high-purity sodium or strontium; (2) a nominal 2-inch-outside diameter by 1 1/2-inch-long "case" made of beryllium oxide-loaded epoxy behind the sensor grid; (3) four 3/16-inch-diameter copper electrical connectors, called "pins", brazed to the sodium grid with thallium metal or brazed to the strontium grid with lithium metal; (4) a nominal 2-inch-outside diameter by 0.3-inch-thick high-purity copper "cap" in front of the sensor grid; (5) a 1/4-mil-thick disc of Mylar to insulate the copper cap from the grid, and (6) epoxy seals around the pins and between the cap and case. All sodium grids were made of reagent-grade (99.48% purity) lump metal^(a) which had the following listed impurities:

<u>Impurity</u>	<u>Concentration (%)</u>
Cl ⁻	0.0010
N ₂	0.0020
PO ₄ ⁻³	0.0002
SO ₄ ⁻²	0.0010
Heavy Metals (as Pb)	0.0005
Fe	0.0005

(a) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey, Lot Number 34283.

Figure 2. SHOCK PRESSURE TRANSDUCER BASIC GRID PATTERN.

All strontium grids were made of 99+% purity vacuum-distilled strontium bar (5/8" D)(b) which was remelted at Y-12 into a two-inch-diameter ingot. Strontium purity was not rechecked after the remelting procedure; however, it should not have changed significantly. The remelting procedure is described in Appendix A. Some work was done in an attempt to produce strontium grids by vapor deposition (see Appendix B).

Electrical connecting pins were made from 99.92% minimum-purity, oxygen-free copper as were the copper caps in front of the grid. Detail dimensions

---

(b) Obtained from the United Mineral and Chemical Company, New York.

- Notes:
1. Thallium braze (63.9°C eutectic temp) between grid and terminal
  2. See basic grid pattern, Figure 2

Figure 3. SHOCK-PRESSURE TRANSDUCER SODIUM GAGE. (Type A)

of the copper pins are given in Figure 5; nominal copper cap dimensions in Figure 6. The encapsulation material behind the grid (the "case") was a beryllium oxide-loaded epoxy. The beryllium oxide loading was based upon matching the bulk modulus times density for the sodium and strontium and their respective encapsulations according to the expression:

$$B_1 \rho_1 = B_2 \rho_2,$$

where:

B represents the bulk modulus and

ρ the density.

The resulting beryllium oxide-epoxy mixes for the sodium and strontium transducer cases, respectively, were 38 wt % BeO-62 wt % epoxy for the

Notes:

1. Lithium braze (134°C eutectic temp) between grid and terminal
2. See basic grid pattern, Figure 2

Figure 4. SHOCK-PRESSURE TRANSDUCER STRONTIUM GAGE. (Type A)

Figure 5. COPPER PIN.

sodium transducer cases and 80 wt % BeO-20 wt % epoxy for the strontium. Properties of the beryllium oxide-epoxy mixes for matching were determined using both strain-gage and sonic-velocity techniques. Some difficulties were experienced in getting shear wave velocities in the beryllium oxide-loaded epoxy material, hence the strain-gage data were used for matching. Details

Notes:

1. x and y diameters to be concentric within 0.0005 TIR.
2. Surface z to be flat within 0.0001 TIR.
3. Material: Oxygen-free, high-conductivity copper 99.92% (min) purity.

Figure 6. COPPER CAP.

of the matching and testing are summarized in Appendix C. The beryllium oxide powder used was Beryllium Corporation Type R66 powder with a particle size of less than 150 microns. The low-Z epoxy utilized was Jones Dabney Epi-Rez 5101 with a MPDA-MDA eutectic cure. Details of the beryllium oxide powder and epoxy specifications are included in Appendix D.

FABRICATION

The copper cap was insulated from the sodium and strontium grids using a 1/4-mil-thick disc of Mylar. The Mylar disc was checked for pinholes prior to use by using it to seal a vacuum of 10^{-5} inch of mercury. Conventional helium leak testing was unsatisfactory since helium diffuses through the Mylar almost instantaneously in sufficient quantities to knock a standard leak detector off scale.

The copper pins and cap were sealed to the beryllium oxide-epoxy case using an epoxy adhesive. Some difficulty was experienced in obtaining a good bond between the copper and adhesive and the beryllium oxide-epoxy and adhesive; however, a satisfactory bond was eventually obtained using Hysol epoxy patch adhesive (see Appendix E).

ASSEMBLY AND TESTING

Assembly Procedure

The transducer assembly procedure was developed by assembling the four sodium and four strontium transducers and "debugging" the procedure as required. Since the procedure was progressively upgraded throughout the program, all the transducers were not assembled exactly in the same manner. The final procedure used and the one which gave the best assembly results is summarized in Appendix F. In general, both the sodium and strontium transducers were assembled by: (1) cold forming the sodium or strontium metal into a female groove detail in the beryllium oxide-epoxy encapsulation and removing excess metal; (2) brazing the copper pins to the grids using thallium for the sodium-to-copper braze and lithium for the strontium-to-copper braze (see Appendix G); (3) capping the front of the gage with a high-purity copper cap insulated from the grid by a 1/4-mil-thick Mylar disc, and (4) sealing the transducer with epoxy. In general, all sodium gages were assembled in the same manner with the exception of the type of epoxy used to seal the transducer, whereas the procedure described for the strontium transducers was utilized on the final two transducers only.

Both the sodium and strontium transducers were assembled in a dry, high-purity argon environment. Two samples taken from the argon environment, after all modifications were made to the glove box and associated equipment, gave the following analysis by the mass spectrograph and gas chromatograph:

<u>Impurity</u>	<u>Quantity (ppm)</u>
CO ₂	14
O ₂	13
N ₂	15
CH ₄	8
H ₂	6
CO	0

An independent moisture monitor^(c) indicated ~ 3 ppm moisture.

(c) Gilbarco sorption hygrometer.

The equipment setup for providing the argon atmosphere is shown in Figures H-1 through H-3 of Appendix H. In general, it included a closed argon box with a gas recirculation system made up of a molecular-sieve and uranium-chip pass unit for removing moisture, oxygen, nitrogen, and carbonaceous gases from the box atmosphere.

Photographs were taken through the argon box window of one sodium (Specimen 6) and one strontium (Specimen 9) transducer prior to pressing the copper caps in place. Figure 7 shows the case with a sodium grid in place along with the cap. A closeup view of the sodium grid is given in Figure 8. Similar views of the strontium transducer are shown in Figures 9 and 10. Normally, the copper caps were pressed on immediately after the excess sodium or strontium was cut away; however, both the sodium and strontium transducers shown were finished during the evening work period and it was necessary to wait overnight for photographs to be taken. This delay allowed some nitriding of the exposed grid surface to occur. Colored photographs of the sodium grid give the sodium an off-color blue-white appearance which was not the case. Although the surface was slightly gray (nitrided), it had basically a silvery

123779

Figure 7. SODIUM TRANSDUCER GRID AND CAP. (Specimen 6)

123780

Figure 8. SODIUM TRANSDUCER GRID. (Specimen 6)

metallic finish. Similar circumstances exist in the strontium grid photographs. Apparently the fluorescent lighting on the argon box affected the photographs. The resistance of the transducers is a good indication of the amount of corrosion on the grid surfaces (see Appendix I). A calculated sodium transducer resistance was 27.7 milliohms at 23° C (room temperature) based upon a resistivity of 5.54 microohms-cm at 23° C; the four fabricated transducers had resistances of 30, 25.33, 29.83, and 25.30 milliohms. Calculated strontium transducer resistance was 115 milliohms at 20° C (3° C below room temperature) based upon a resistivity of 23 microohm-cm, whereas the four actual transducers had resistances of 92.8, 80.7, 106.7, and 80.3 milliohms at room temperature (23° C). Resistance calculations are summarized in Appendix J.

The four strontium transducers were radiographed after assembly to see if the grid was in intimate contact with the cap. X rays were taken perpendicular to the strontium transducer axis, one parallel to the grid and one transverse to the grid, as can be seen in Figures 11 through 14. Table 1 summarizes the X-ray analyses and indicates that Transducers 5, 7, and possibly 9 have

123950

Figure 9. STRONTIUM TRANSDUCER GRID. (Specimen 9)

good intimate contact between the grid and the copper cap; Transducer 8 could have a 3-mil gap.

Hydrostatic Pressure Test

Initial plans were to static pressure test the transducers in 100,000-psi (~ 6.9 -kilobar) mineral oil using an existing 4-inch-inside diameter piston cylinder intensifier with electrical feedthroughs. A Kelvin bridge-type setup with a 6-volt battery, a 40-ohm resistor, a Kintel milliammeter in series with the transducer (outer two pins), and a Hewlett Packard VTVM across the grid (inner two pins) was to be utilized to determine the transducer resistance change during the pressure cycle. During the first attempted pressure test (on sodium Transducer 2), the operator overpressurized to $\sim 180,000$ psi. A subsequent run to 77,000 psi indicated that a short existed in the wiring setup. Upon removal of the transducer from the pressure chamber it was found that the epoxy seal around the pins had failed, allowing oil to penetrate into the sodium grid. The transducer was evacuated several times to remove most of the oil, cleaned with hexane, and resealed with epoxy leaving some oil inside.

123949

Figure 10. STRONTIUM TRANSDUCER GRID AND CAP. (Specimen 9)

Table 1
X-RAY DATA ON STRONTIUM TRANSDUCERS

Transducer	View	Analysis (active area)
5	Parallel to Grid	Appears to be in intimate contact.
	Transverse to Grid	Appears to be in intimate contact.
7	Parallel to Grid	Appears to be in intimate contact.
	Transverse to Grid	Appears to be in intimate contact.
8	Parallel to Grid	Appears to have a gap of ~ 3 mils between the cap and grid.
	Transverse to Grid	Appears to have a gap of ~ 3 mils between the cap and grid.
9	Parallel to Grid	Appears to have a gap of ~ 3 mils between the cap and grid.
	Transverse to Grid	Appears to be in intimate contact.

During the interim, a second sodium transducer (Specimen 6) was pressed in mineral oil at 22,500 psi. It also leaked, breaking the grid. It was machined open, a new sodium grid formed, and reassembled. Although both transducers which leaked had a Shell 828 epoxy with T-1 cure, which was found later to provide a poor bond between the beryllium oxide-epoxy case and copper cap, it was decided not to expose any of the remaining transducers to isostatic oil pressure since: (1) they were likely to leak, and (2) the static data obtained were not representative of dynamic shock characteristics of the transducers and, consequently, was of little value.

(a) Transverse View

124030

(b) Parallel View

124031

Figure 11. RADIOGRAPHS OF STRONTIUM TRANSDUCER 5.

(a) Transverse View

124034

(b) Parallel View

124035

Figure 12. RADIOGRAPHS OF STRONTIUM TRANSDUCER 7.

(a) Transverse View

124029

(b) Parallel View

124028

Figure 13. RADIOGRAPHS OF STRONTIUM TRANSDUCER 8.

(a) Transverse View

124032

(b) Parallel View

124033

Figure 14. RADIOGRAPHS OF STRONTIUM TRANSDUCER 9.

Shock testing was to be done by the Lawrence Radiation Laboratory. Preliminary results obtained from the LRL indicated that the strontium transducers worked quite well, but some difficulty was experienced in obtaining clear signals from the sodium transducers.

APPENDIX A

SODIUM AND STRONTIUM DISC FABRICATION

Introduction

The sensor elements for both the sodium and strontium pressure transducers were prepared by cold forming thin discs of sodium or strontium into a female groove detail in the beryllium oxide-loaded epoxy cases and removing the excess metal leaving only that in the groove. Very little difficulty was experienced in preparing the discs; however, details of how it was done will be described.

Sodium Discs

Sodium discs were prepared by hand cutting them from a dry, 2-inch-outside diameter commercially available log of sodium metal. The sodium log was first dried by removing it from mineral oil; most of the oil was removed while outside the glove box using an absorbent cloth. It was quickly transferred into the glove box and the complete outside surface of the log cut away and removed from the glove box leaving only the inner dry log of clean sodium of ~2-inch diameter. Thin discs were cut from this log using Exacto knives and "homemade" razor blade knives.

Strontium Discs

Strontium discs were somewhat harder to prepare than sodium but not difficult. Initial efforts were to purchase 1-inch-square by 0.008-inch-thick strontium discs from a vendor; however, of the seven vendors which supply high-purity strontium, only one was willing to undertake the task. A price of \$600 per sheet was indicated, and that on a best-effort basis with no guarantee of a delivery date.

Attempts were made at Y-12 to roll strontium sheet using a hand roll and a power roll with intermediate dips in mineral oil; however, this process was found to be very difficult. In one instance, a sheet of strontium actually exploded when too large a reduction was attempted. Sandwiching the strontium between stainless steel plates helped, but the resulting strontium sheet was still of poor quality and had dark, corroded surfaces. This condition resulted because the rolling was not done in an inert atmosphere and it was practically impossible to eliminate diffusion through the thin mineral oil film.

The second attempt, which was successful, was to cut 0.050-inch-thick by 2-inch-outside diameter strontium discs from a 2-inch-outside diameter ingot using a lathe and a 0.050-inch-thick cutoff tool. These discs were then mounted in a 2-inch-outside diameter recessed groove in a vacuum chuck on a lathe and faced off until the desired thickness was reached. The discs were flooded with mineral oil during machining.

Discs as thin as 0.008 inch were prepared in this manner. This technique was used for all remaining strontium discs. The discs utilized in fabricating the four strontium transducers were ~2-inch outside diameter by 0.030-inch thick.

Strontium Ingot Preparation

Commercially available strontium is supplied in the form of extruded 5/8-inch-outside diameter rod or crystalline lump-type metal, although larger diameter rod can be supplied on special order. In order to conserve time and expense, 2-inch-outside diameter ingots of strontium were prepared at Y-12 from the readily available 99+% purity, vacuum-distilled, 5/8-inch-outside diameter strontium rod obtained from United Mineral and Chemical Company.

Commercial strontium rod generally comes shipped under mineral oil and has a heavy corrosion deposit on its surface. Since this deposit contains strontium oxide, nitride, and possible carbides, all of which melt at higher temperatures than strontium metal, it was necessary to remove these deposits prior to remelting the strontium. This step prevents the oxide or nitride particles from becoming dispersed in the strontium during melting. These deposits were removed by scraping off as much as possible by hand, then chemically cleaning the rods in acetic acid. Before chemical cleaning, the 5/8-inch-outside diameter by 20-inch-long strontium rods were cut by a hand saw into 2 3/4-inch-long rods. (A film of mineral oil was on the rods during cutting.) The short rods were degreased with hexane (at room temperature) and quickly transferred into a Pyrex reactor that had a continuous purge of plant argon. The argon entered directly the reactor and left through a mineral oil bubbler. With seven short rods in the reactor, a large volume of acetic acid was poured into the reactor and allowed to react with the strontium surface until it became a clean, metallic surface. A bottom valve was opened in the reactor and the acetic-acid-strontium acetate solution drained off quickly, then the valve was left open and a heavy flow of argon maintained until all strontium surfaces were dry. Although they appeared to be metallic, a thin film of white strontium

acetate generally remained on the surface of the bars. The clean bars were then remelted in the tantalum can shown in Figure A-1 at 850° C. The resulting melt was a clean, metallic, pore-free melt even at the top exposed surface. Remelting the strontium was not expected to reduce its purity and possibly even helped it. In going to 850° C, the strontium acetate film, $\text{Sr}(\text{CH}_3\text{CO}_2)_2 \cdot 4$ to $5 \text{H}_2\text{O}$, should decompose to form strontium oxides, carbides, and hydrides which then would settle out; whereas, any undecomposed acetate should float to form a protective top surface film, or should evaporate.

124132

Figure A-1. TANTALUM CAN AND STRONTIUM REMELTING HARDWARE.

The first attempt to remelt the 5/8-inch strontium bars into a 2-inch ingot, using only the tantalum container, resulted in an ingot with centerline porosity. During the melting operation, the tantalum can was situated in the middle of a cylindrical, wall-heated, argon induction furnace. The resulting ingot had a hole down the center of the ingot due to radial solidification and consequent shrinkage toward the tantalum container wall. This situation was corrected by forcing the strontium ingot to solidify from its bottom surface upward using the equipment in Figure A-1 as shown in Figure A-2 (ie, one-dimensional solidification). The bottom surface of the tantalum can was left exposed, whereas its sides and top were covered with alumina powder insulation. Access was left in the top insulation only such that the valve on the top flange could be opened when placed in the furnace and closed immediately upon opening the furnace. The resulting ingots came out as good, sound, clean pieces.

Figure A-2. BOTTOM-COOLED STRONTIUM INGOT SETUP.

APPENDIX B

STRONTIUM VAPOR DEPOSITION EXPERIMENTS

The object of this work was to plate approximately six mils of strontium or sodium on the beryllium oxide-loaded epoxy parts of the transducer assembly. Since these metals are very reactive, the usual vacuum evaporation procedure required modification so that the sample charge, finished product, and waste material would not be exposed to the atmosphere at any time.

The small vacuum evaporation chamber, shown in Figure B-1, was designed and built of stainless steel. Copper gaskets were used at the three flanges so that the system would remain vacuum tight regardless of the temperature reached in the confined area. The unit was small enough to pass through an isolation chamber into the argon glove box for loading. The electrodes were

124137

Figure B-1. VAPOR DEPOSITION APPARATUS.

designed to hold both baskets and boats for the metal, at currents up to 150 amperes. The distance between the plastic substrate and boat could be adjusted between 1.5 and 4 inches, if necessary, to protect the plastic from the heat of the boat. Temperature measurements made with a pyrometer showed that a current of 20 amperes through an unloaded tungsten basket would heat it to 1,000° C.

In the first attempt to plate strontium, a cube of the metal about 1/4 inch on a side was placed in the basket. However, no plating was obtained and the strontium was not melted. Following the method described in Strong's Procedure in Experimental Physics, it was found that the strontium could be melted when argon was allowed to flow through the chamber at approximately atmospheric pressure. The strontium could be evaporated from the filament in the usual manner. A special small glass chamber, also shown in Figure B-1, was used to precondition the filament in this way. The original tungsten basket could not hold enough strontium to provide plate of the required thickness, so a small molybdenum boat was fabricated to hold three to four cubes of strontium. In the various plating attempts it was found that: (1) it was necessary to rinse the plastic with methylene chloride during the cleaning procedure to get the strontium to stick; (2) hot strontium is a very active getter. All coatings deposited had a layer of the nitride, or oxide, or both on the surface. However, this coating could be scraped away leaving clean strontium. The surface of the coating in contact with the plastic was very lustrous. The cleanest coatings were obtained when the valve connecting the chamber to the pumping system was closed just before evaporation started, and a coating about 6 to 8 mils thick was finally produced using about 200 - 215 amperes for about 15 minutes.

At this time the design of the gage was changed, calling for 24 mils of strontium on the face of the gage. This modification made a larger boat necessary, and one run was made in which the thickness of the coating varied from 15 to 23 mils. A larger charge of material was conditioned in the boat, but it could not be evaporated. Currents of 390 amperes were used, greatly overloading the design of the system. Hence, two successive attempts would have to be used to coat a specimen with 25 mils of strontium, and the sample would have to be cleaned in between runs to remove the layer of oxide that would form. As an alternative, the chamber could be rebuilt for a higher current capacity.

Development of a method for forming the grids of strontium and sodium for the final transducers had already been developed and the decision was made to use that technique for the final transducers.

APPENDIX C

SHOCK IMPEDANCE MATCHING OF TRANSDUCER ENCAPSULATION

In order to reduce undesirable shock reflections at the interfaces between the sodium and strontium pressure sensing element and their respective encapsulations, their shock impedance characteristics were matched. For a longitudinal, one-dimensional shock wave, it is the usual practice to match the product of Young's modulus (E) and the material density (ρ) for the sensor and encapsulation. LRL suggested that a better match was the product ρB (ie, density times the bulk modulus). The latter method was used for selecting transducer encapsulation.

Longitudinal and shear-wave velocities and densities were determined at Y-12 for three different strontium specimens. Young's moduli, bulk moduli, and Poisson's ratios were then computed. The resulting data are summarized in Table C-1 along with similar values for sodium which were supplied by the LRL. Impedance characteristics, ρB and ρE , are also summarized. Encapsulation materials considered are listed in Table C-2.

The two materials which are compatible with strontium and sodium, and which possibly could be mixed in a ratio such that their impedance characteristic, ρB , matches that of strontium and sodium, are beryllium oxide-loaded polyethylene and beryllium oxide-loaded Epi-Rez epoxy.

Several 1/2-inch-outside diameter by 1-inch-long specimens of beryllium oxide-loaded polyethylene powder were die pressed at 150° C and 2,500 psi for testing. The resulting pellets were "flaky", heat sensitive, had little or no mechanical strength, and, consequently, were not acceptable as an encapsulation material.

A number of test pellets were also die pressed using beryllium oxide powder blended into an epoxy adhesive (see Appendix D for details of the epoxy system used). These pellets had good mechanical strength and appeared to be acceptable as encapsulation material. Initial attempts were to use commercial UOX-grade beryllium oxide powder having a particle size ranging from ~ 2 to 5 microns; however, wetting of the "fluffy" powder was difficult for high beryllium oxide loadings, resulting in < 100 percent dense pellets. Shrinkage was also excessive during die pressing. UOX beryllium oxide powder was changed to Berylco R66 powder having a particle size ranging from 2 to 100 microns (see Appendix D for powder characteristics). Pellets with beryllium oxide compositions of 0, 25, 50, 75, and 90 weight percent were easily pressed

Table C-1
IMPEDANCE MATCHING MATERIAL PROPERTIES DATA FOR STRONTIUM AND SODIUM

Material	Longitudinal Wave Velocity, V_L (in/sec $\times 10^{-5}$)	Shear Wave Velocity, V_S (in/sec $\times 10^{-5}$)	Young's Modulus, E (psi $\times 10^{-6}$)	Poisson's Ratio, μ	Bulk Modulus, B (psi $\times 10^{-6}$)	Density, ρ (gms/cc)	ρB (lb ² /in ⁵ $\times 10^{-6}$)	ρE (lb ² /in ⁵ $\times 10^{-6}$)
Sr(1) 1	1.374	0.523	1.78	0.345	1.92	2.594	0.180	0.167
Sr 2	1.377	0.531	1.66	0.36	1.95	2.593	0.183	0.156
Sr 3	1.362	0.479	1.64	0.36	1.92	2.585	0.179	0.153
Na(2)	1.1	0.63	2.25	0.295	0.876	0.97	0.060	0.080

(1) Strontium data obtained at Y-12 on specimen sizes:

Sr 1 - 0.5015" OD $\times$ 0.4994" L

Sr 2 - 0.5006" OD $\times$ 0.2505" L

Sr 3 - 0.2510" OD $\times$ 0.2495" L

(2) Sodium data obtained from the LRL.

Table C-2
ENCAPSULATION MATERIALS CONSIDERED

Material	Density, ρ (gms/cc)	Poisson's Ratio, μ	Young's Modulus, E (psi $\times 10^{-6}$)	Bulk Modulus, B (psi $\times 10^{-6}$)	ρE (lb ² /in ⁵ $\times 10^{-6}$)	ρB (lb ² /in ⁵ $\times 10^{-6}$)	Remarks
KAPTON (polyimide)	1.42 - 1.79	0.45	0.25 - 0.43	0.83 - 1.43	0.013 - 0.028	0.043 - 0.093	Available in thin films only.
MgO	3.6	0.16	40	20	5.203	2.601	ρB mismatch.
Al ₂ O ₃	3.97	0.25	56	38	8.032	5.450	ρB mismatch.
BeO	3.01	0.25	49	33	5.329	3.589	ρB mismatch.
BN	2.27	0.30	5 - 13	4.2 - 10.8	0.410 - 0.886	0.344 - 0.886	Surface H ₂ O; ρB mismatch.
VESPEL (polyimide)	1.42 - 1.59	0.45	0.46 - 0.61	1.53 - 2.03	0.024 - 0.035	0.078 - 0.117	Compatible with Na and Sr. Poor ρB match with Sr. Fair ρB match with Na.
MYKROY (glass-bonded mica)	3.1 - 3.9	0.30	6 - 7	5 - 5.83	0.672 - 0.986	0.560 - 0.821	Reacts with alkalis; formed at 1,800° F, 60,000 psi.
Polyethylene	0.91 - 0.96	0.36	0.015 - 0.14	0.018 - 0.17	0.0005 - 0.0049	0.0006 - 0.0060	Compatible with Na and Sr. ρB mismatch.
BeO-Polyethylene ⁽¹⁾	0.91 - 3.01	0.36 - 0.25	0.015 - 49	0.018 - 33	0.0005 - 5.329	0.0006 - 3.589	Difficult to press with high BeO loadings.
Epirez (epoxy)	1.20	0.40	0.36	0.43	0.016	0.019	Compatible with Na and Sr. ρB mismatch.
BeO-Epirez ⁽¹⁾	1.20 - 3.01	0.40 - 0.25	0.36 - 49	0.43 - 33	0.016 - 5.329	0.019 - 3.589	ρB , can be matched to Na and Sr.

(1) BeO-loaded material with weight percent beryllium oxide ranging from 0 - 100 percent.

and were essentially 100 percent dense up to loadings of $\sim 50 - 75$ weight percent, as shown in Figure C-1.

Figure C-1. BERYLLIUM OXIDE-EPOXY DENSITY AS A FUNCTION OF THE WEIGHT PERCENT BERYLLIUM OXIDE. (Densities by "Mike")

Initial efforts were to determine the shock-impedance characteristics, density, and bulk modulus using sonic techniques; however, difficulty was experienced in determining the shear-wave velocity using standard techniques although longitudinal values could be determined. (The standard technique for determining wave velocity was to input a wave, reflect it off the rear surface, and using the specimen length and delay times determine wave velocity.) As a secondary method, biaxial strain gages were applied to 1/2-inch-outside diameter beryllium oxide-epoxy test cylinders, shown in Figure C-2, and plots of stress

124134

Figure C-2. STRAIN-GAGED BERYLLIUM OXIDE-EPOXY TEST SPECIMENS FOR IMPEDANCE MATCHING.

versus strain determined. Young's modulus (E) and Poisson's ratio (μ) were determined from the stress-strain curves. Bulk modulus (B) was calculated using the equation:

$$B = \frac{E}{3(1 - 2\mu)}.$$

Hopefully, the bulk modulus obtained is representative of the material even though the material is by its very nature a homogeneous, nonisotropic medium since it has a system of discrete particles suspended in a matrix material (see Figure C-3 for the microstructure of a 50/50 beryllium oxide-epoxy mix). Neither the standard wave velocity nor strain-gage calculations for B are correct in a theoretical sense.

During completion of the strain gage tests, a technique for obtaining shear-wave velocity was determined whereby the shear wave was passed through the beryllium oxide-epoxy cylinder and into an aluminum cylinder rather than reflecting from the rear beryllium oxide-epoxy surface. The resulting data for both the strain gage and sonic tests are summarized in Table C-3. Beryllium oxide-epoxy and impedance characteristics (ρB and ρE) as a function of the weight percentage of beryllium oxide are summarized in Figures C-4 and C-5. Beryllium oxide-epoxy encapsulations of ~ 80 and ~ 38 weight percent beryllium oxide are required for the strontium and sodium transducers,

Figure C-4. DENSITY-BULK MODULUS IMPEDANCE MATCHING OF STRONTIUM AND SODIUM TO BERYLLIUM OXIDE-EPOXY ENCAPSULATION.

Figure C-3. MICROSTRUCTURE OF A 50 - 50 WEIGHT PERCENT BERYLLIUM OXIDE-EPOXY MIX. (As Polished; Bright Field Illumination; 150X)

Figure C-5. DENSITY-YOUNG'S MODULUS IMPEDANCE MATCHING OF STRONTIUM AND SODIUM TO BERYLLIUM OXIDE-EPOXY ENCAPSULATION.

Table C-3
SUMMARY OF STRAIN GAGE AND SONIC PROPERTY VALUES FOR BERYLLIUM OXIDE-LOADED EPOXY

Epoxy-Beryllium Oxide Composition (%)	Longitudinal Wave Velocity, V_L (in/sec $\times 10^{-5}$)	Shear Wave Velocity, V_S (in/sec $\times 10^{-5}$)	Density, ρ (gms/cc)	Young's Modulus, E (psi $\times 10^{-6}$)	Poisson's Ratio, ν	Bulk Modulus, B (psi $\times 10^{-6}$)	ρE $\times 0.03613$ (lb ² /in ⁵ $\times 10^{-6}$)	ρB $\times 0.03613$ (lb ² /in ⁵ $\times 10^{-6}$)
<u>Strain Gage Results</u>								
100 - 0	-	-	1.200	0.361	0.400	0.605	0.016	0.026
75 - 25	-	-	1.412	0.463	0.402	0.787	0.024	0.040
50 - 50	-	-	1.713	1.124	0.359	1.331	0.070	0.082
25 - 75	-	-	2.142	2.174	0.316	1.965	0.168	0.152
10 - 90	-	-	2.405	6.393	0.279	4.825	0.555	0.419
<u>Sonic Results</u>								
100 - 0	1.034	0.336	1.203	0.366	0.44	1.03	0.016	0.045
75 - 25	1.140	0.383	1.498	0.593	0.44	1.54	0.032	0.083
50 - 50	1.206	0.504	1.614	1.07	0.39	1.68	0.062	0.098
25 - 75	1.546	0.552	2.055	1.67	0.43	3.81	0.123	0.283

respectively, based upon the strain-gage density-bulk modulus match. Density-Young's moduli match curves are shown for reference only. The latter ρE plot indicates compositions of 73.9 and 54.2 weight percent beryllium oxide for strontium and sodium transducers, respectively.

APPENDIX D

FABRICATING BERYLLIUM OXIDE-LOADED EPOXY CASES

All beryllium oxide-loaded epoxy cases were made by: (1) pressure curing beryllium oxide-loaded epoxy against a die face which contained the transducer grid detail; (2) sizing the case outside diameter on a cylindrical grinder using the flat case end with groove detail as a reference surface; (3) sizing the case length on a lathe using carboloy tools, and (4) drilling and counter-boring pinholes using a Bridgeport mill with carboloy tools. Both the sodium and strontium cases have the same dimensions although their beryllium oxide loadings were 38 weight percent for the sodium transducer and 80 weight percent for the strontium.

The beryllium oxide-epoxy mix for each case was prepared by hand mixing beryllium oxide powder with the uncured epoxy in a stainless steel beaker until it was thoroughly mixed. This step required ~ 2 hours per case. (The epoxy was evacuated prior to mixing and again after mixing.) The 38 weight percent mix was liquid enough to pour; however, the 80 weight percent mix was very stiff, much like modeling clay. The die face, die cavity, and top piston surfaces (see Figure D-1) were mold released with Garan 225 mold release to prevent the epoxy from adhering to the steel surfaces during curing. The beryllium oxide-epoxy mix was transferred to the die and evacuated again after the top piston was inserted. The die assembly was then placed in a 100-ton press, heated to 150° C, and a load of 50,000 pounds applied as soon as practical without excessive extrusion of epoxy out of the die (generally, 45 minutes after reaching temperature for the 38 weight percent cases and immediately for the 80 weight percent cases). Both load and temperature were held for a minimum of 12 hours, the heat was cut off, and the load removed when the die temperature cooled to 50° C. The case could be removed from the die with no difficulty.

The die cylinder used for making the cases was made of D-3 grade steel hardened to R_c 65 - 66. Both the top piston and die were made of annealed AISI 4340 steel. The 4340 die which had a male grid pattern was machined on a Lucas 3-inch-diameter, numerically controlled horizontal milling machine.

Beryllium oxide powder used in both the sodium and strontium cases was Berylco R66 grade commercial powder, approximately 96 - 98 percent purity, with the typical particle-size distribution given in Figure D-2. Typical spectrographic analyses are reported in Table D-1.

Figure D-1. DIE HARDWARE FOR PRESSING AND CURING BERYLLIUM OXIDE-LOADED EPOXY CASES.

124130

Figure D-2. BERYLCO R66 PARTICLE SIZE DISTRIBUTION ANALYSIS. (By Coulter Counter)

Table D-1
TYPICAL SPECTROGRAPHIC ANALYSES OF BERYLCO R66
GRADE BERYLLIUM OXIDE POWDER

Element	Concentration (ppm)(1)	Element	Concentration (ppm)(1)	Element	Concentration (ppm)(1)
Al	0.3%	Ho	< 0.20	Ru	1
As	3	I	< 0.16	S	530
Au	< 0.20	In	0.5	Sb	2
B	2.0	Ir	< 0.38	Sc	30
Ba	1	K	290	Se	2
Bi	< 0.16	La	< 0.16	Si	0.45%
Br	6.0	Lu	< 0.22	Sm	< 0.68
Ca	20	Mg	1.2%	Sn	0.002
Cd	< 0.002	Mn	< 4	Sr	0.4
Ce	< 0.20	Mo	2	Tb	< 0.20
Co	< 0.80	Na	5.0	Te	< 0.44
Cr	< 4	Nb	0.4	Th	< 0.28
Cs	< 0.16	Nd	< 0.75	Ti	10
Cu	20	Os	< 0.56	Tl	< 0.36
Dy	< 0.70	P	720	Tm	< 0.22
Er	< 0.62	Pb	< 0.0002	U	< 0.30
Eu	< 0.36	Pd	2	V	2.0
Fe	110	Pr	< 0.18	W	< 0.74
Ga	2	Pt	< 0.56	Y	0.40
Gd	< 0.88	Rb	0.5	Yb	< 0.66
Ge	1	Re	< 0.36	Zn	192
Hf	< 0.82	Rh	1	Zr	120
Hg	< 0.84				

(1) Unless otherwise noted.

The epoxy resin used was Jones Dabney Epi-Rez 5101 resin purchased on Bendix Specification SG 223056. Curing agent used was a 60/40 eutectic mixture of MPDA and MPA agents purchased on Y-12 Specifications 00-M-051 and 00-M-052, respectively. The resin-curing agent ratio used was 100/20.

APPENDIX E

SEALING COPPER CAPS AND PINS TO A BERYLLIUM OXIDE-EPOXY CASE

The environment surrounding the transducers is kept away from the highly reactive sodium and strontium grids by sealing both the pins and cap to the beryllium oxide-loaded epoxy encapsulation using an epoxy adhesive. A counterbore in the beryllium oxide-epoxy case was provided around the pins for sealing purposes. The fabricated transducers have the counterbore filled with epoxy giving an annular seal 1/16 inch thick by 3/8 inch deep. The copper cap has an annular seal around its periphery of ~ 0.17 inch wide radially by 0.100 inch deep longitudinally.

Some difficulty was experienced in finding an epoxy system which would provide a good bond to copper and the beryllium oxide-loaded epoxy. Initial attempts were to use flat, 0.300-inch-thick copper caps bonded down using a 100/20 mixture of Shell 828 epoxy and T-1 curing agent. However, the resulting bond was very poor and the copper caps came off after final assembly. Several other epoxy systems were tried including:

1. A mixture of 100 parts Shell 828 resin to 40 parts Versamide 140 flexibilizer to 10 parts T-1 curing agent.
2. Hysol Epoxy Patch.
3. A mixture of 100 parts Hysol Epoxy Patch to 40 parts Shell 815 resin with TETA curing agent. The 815 and TETA were mixed in a ratio of 100 parts 815 to 20 parts TETA.
4. A mixture of Epi-Rez 5101 resin with MPDA-MDA eutectic curing agent mixed in the ratio 100 parts 5101 to 20 parts eutectic curing agent.
5. A mixture of 100 parts Shell 815 epoxy resin to 25 parts Versamide 140 flexibilizer to 10 parts Shell U curing agent.

Of the five systems tried, only the Hysol Epoxy Patch and the Shell 815/Versamide 140/Shell U systems provided adequate bonds to both the copper and the beryllium oxide-loaded epoxy case. An adequate bond was considered to be: (1) one which would stand up under the thermal shock induced in the bonded copper caps and beryllium oxide-loaded epoxy case when dropped into cold water from a 150° C oven, and (2) one which would pull a portion of the beryllium

oxide-loaded epoxy case apart prior to rupturing the bond to the copper cap or the case.

Although the Epoxy Patch adhesive provided a good bond, it is a highly filled epoxy system and cannot be evacuated properly (too thick), whereas the Shell 815/Versamide 140/Shell U system was not loaded and could be evacuated as long as vacuum was not taken below $\sim 20 - 22$ inches Hg such that excessive foaming occurred. For sealing purposes, the latter system also provided the best bond.

APPENDIX F

ASSEMBLY PROCEDURES FOR SODIUM AND STRONTIUM TRANSDUCERS

Introduction

Several pieces of hardware were used inside the argon box when assembling the transducers. These items are shown in Figures F-1 and F-2 and are referred to as follows in the assembly procedures:

<u>Item</u>	<u>Description</u>
1	Pin Wetting Fixture
2	Piston-Cylinder Press
3	"Homemade" Razor-Blade Knives
4	O-Frame Screw Press
5	C-Frame Screw Press
6	Teflon Clamp Fixture

124131

Figure F-1. MISCELLANEOUS ASSEMBLY HARDWARE.

124133

Figure F-2. PISTON CYLINDER PRESS.

Sodium Transducer Assembly Procedure

The assembly procedure developed for the sodium transducers is as follows:

1. Measure and record the copper cap thickness and outside diameter and scribe a number on the cap outside diameter.
2. Sandblast the outside ring around the copper cap which will be epoxy bonded to the case, then carefully clean with methylene chloride.
3. Weigh the copper cap on an analytical balance and record the weight, then tape the outside diameter and outer flat face of the copper cap with Mylar tape to prevent scratching the cap finish during assembly.
4. Clean the beryllium oxide-epoxy case and copper pins with methylene chloride.
5. Using a Q tip, apply Garan 225 epoxy mold release to the outside diameter and bottom surface of the case. Then tape the outside diameter of the case with Mylar tape.
6. Transfer the case, pins, and cap to the argon box and allow one to two hours for the surface moisture to evaporate.

7. Scratch the end of each copper pin down to clean copper, then place one pin at a time in the copper wetting fixture preheated to 310 - 400° C on a hot plate and wet the end of the pin with thallium metal (melts at ~ 302° C).
8. After wetting the pins, use a razor-blade tool to trim the soft thallium down to a thin layer (varies ~ 2 to 10 mils).
9. Use a Q tip moistened with mineral oil to apply a very thin film of oil to the flat face of the beryllium oxide-epoxy case which stands above the female groove detail. (This step prevents the soft, gummy sodium from adhering to the case in the remaining steps.)
10. Place the case with the pins in place in the piston-cylinder press.
11. Using a homemade razor-blade knife, cut a clean, dry, sodium disc ~ 1/16 to 1/8 inch thick by 1 1/2 to 2 inches in diameter from a dry sodium log. (Cut away all contaminated sodium prior to cutting the clean disc.) Place the sodium disc over the case groove detail inside the piston cylinder press, lay one silicone rubber pad ~ 1/32 inch thick by 2 inches outside diameter on top of the sodium, then insert the top piston. (Note: Both the bottom plate and top piston have O-ring seals.)
12. Transfer the piston-cylinder press out of the argon box, quickly press the sodium into the groove using a 10,000-pound force, and transfer the press back into the argon box and remove the case from the press.
13. Using the homemade razor-blade knives, trim away any excess sodium leaving only that sodium in the groove detail.
14. Immediately lay a 1.67-inch-outside diameter by 1/4-mil-thick disc of Mylar over the sodium grid and gently brush it down until it adheres to the sodium and beryllium oxide-epoxy surface.
15. Immediately place the copper cap on the case and press the cap down in place using the O-frame press, then place the case inside the O-ring sealed piston-cylinder press, transfer the unit out of the argon box, press the cap down with a 20,000-pound force, and transfer the unit back into the argon box.
16. Preheat a hot plate to 70° C inside the argon box and heat the transducer to 65 - 70° C to form the eutectic braze between the thallium-wetted copper pins and sodium (eutectic at 63.9° C).

17. Place the transducer with the pins in the vertical position and fill each of the counterbores around the pins with epoxy (100 parts Shell 815 resin, 40 parts Versamide 140 flexibilizer, to 10 parts Shell U curing agent). Allow 10 to 12 hours to set up at room temperature.
18. Place the transducer in the Teflon fixture with the cap on top and pour epoxy seal (same epoxy as used on pins) around the cap-to-case sealing groove. Place the fixture and transducer in the C-frame press and load while the epoxy sets up. Evacuate down to < 20 inches Hg approximately three times to pull the epoxy into the seal groove, then place the transducer and fixture on a 50 to 60° C hot plate inside the argon box and heat the unit for ~ 12 hours until the epoxy seal cures.
19. Remove the transducer from the argon box, obtain a rough resistance check using a Simpson 260 VOM, then remove the Mylar tape and machine off any excess epoxy around the outside diameter.
20. Check the final gage resistance using a Kelvin bridge applying power (1 amp maximum discontinuous current) at the outer pins and checking the resistance between the inner two pins.
21. Radiograph the transducer perpendicular to its axis to check for any gap between the copper cap and sodium grid. Get one shot parallel to the grid and one shot across the grid.
22. Package the transducer in cotton to prevent scratching the cap surface.

The procedure described for assembling sodium gages can be used with little or no difficulty. In future sodium transducers it may be possible and is desirable to eliminate the thallium braze joint. This change could probably be made by first wetting the copper pins with melted sodium, allowing the surface of the sodium to pick up the usual amount of corrosion during the wetting operation. Just prior to pressing the sodium into the groove, one thin layer of corroded sodium on the pins could be trimmed down to clean sodium metal. (Clean sodium will weld to itself instantly upon contact.) When the clean sodium disc is pressed into the groove, it should weld itself to the sodium-wetted copper pins making a good low-resistance joint.

Strontium Transducer Assembly Procedure

The procedure developed to assemble strontium transducers is very similar to that for the sodium ones except that the following steps differ:

<u>Step</u>	<u>Change</u>
7	Copper pins are wet with lithium instead of thallium (Li melts at 186° C).
9	Eliminate.
11	One side of a 0.030-inch-thick by 2-inch-diameter disc of strontium (see Appendix A for preparation of the disc) is scratched until it has a clean metallic finish. It is then placed over the case female groove detail with the case in the piston-cylinder press. Two 1/32 by 2-inch silicone rubber discs are placed between the strontium disc and the top piston.
12	Same as for sodium except a load of 40,000 pounds is used to form the strontium disc into the groove detail.
13	Excess strontium is filed away using hand files. The case and strontium grid are then faced off ~ 1/2 - 1 1/2 mils in steps of 0.0002 inch using a lathe in a dry argon atmosphere and tungsten carbide tools. (This step requires a new carbide tool after approximately three passes.)
16	A 3/16-inch drill with its tip ground flat is inserted down each pin-hole and the strontium surface scratched clean again. The pins are inserted and a hot plate is preheated to ~ 150° C inside the dry box. Heat the transducer above 134° C to form the eutectic braze between the lithium-wetted pins and the strontium grid (eutectic at 134° C).

APPENDIX G

BRAZING STRONTIUM AND SODIUM PRESSURE-SENSING ELEMENTS TO THE CONNECTING PINS

In order to fabricate a low-resistance shock transducer with a stable resistance, it was desirable that the sodium and strontium pressure sensor elements be brazed to the electrical connecting pins. A number of experiments were run to determine suitable brazing metals that would wet both the pins and strontium and sodium at a temperature below the melting points of strontium and sodium and at a temperature compatible with the beryllium oxide-loaded epoxy encapsulation. Tests were run to determine which of the metals (Be, Al, Mg, or Cu) could best be brazed to strontium and sodium using one of the pure low-melting metals (Li, K, Na, or Tl). These tests were run inside

Table G-1
TEST DATA ON BRAZING METALS

Sample 1		Sample 2		Remarks
Metal	Melting Point (° C)	Metal	Melting Point (° C)	
Al	660	Li	186	Forms a black, hard, brittle state.
		Na	97.5	Difficult to wet due to a surface film developing on the sodium.
		K	62.3	Difficult to wet due to a surface film developing on the potassium.
Be	1,284	Li		Difficult to wet even by scratching the beryllium surface under the lithium melt.
		Na		Difficult to wet.
		K		Difficult to wet.
Cu	1,063	Li		Very easy to wet.
		Tl	303.5	Very easy to wet.
		Na		Sodium wets copper but sodium blackens rapidly even in argon with < 50 ppm impurities.
Sr	770	Li		Forms eutectic melt at 134° C. The lithium-strontium alloy is brittle.
		Na		Not recorded. ⁽¹⁾
		K		Not recorded.
Na	97.5	Tl		Forms a eutectic melt at 63.9° C.
		Li		Forms a eutectic melt at ~ 92° C, too close to the melting point of sodium.
Mg	651	Li		Not recorded.
		Na		Not recorded.
		K		Not recorded.

(1) Some of the magnesium and strontium wetting data were not recorded; however, the results at the time of the tests were apparently negative enough to warrant their rejection as a pin metal.

the argon glove box using a hot plate and/or a 250-watt soldering gun as a heat source. These tests are summarized in Table G-1. As a result of the tests, copper was selected as the best pin material for both the sodium and strontium transducers. Lithium (mp - 186°C) was selected to braze strontium (mp - 770°C) to copper (mp - $1,063^{\circ}\text{C}$), and thallium (mp - 303.5°C) was selected to braze sodium (mp - 97.5°C) to copper. The copper is easily wet by both liquid lithium and thallium, whereas strontium forms a eutectic melt with lithium at $\sim 134^{\circ}\text{C}$ and thallium forms a eutectic melt with sodium at $\sim 64^{\circ}\text{C}$. Each system provides a means for brazing the pressure sensor element to the pins.

Electrical pin connectors made of such metals as beryllium, which are plated on the tip with a thin film of copper to aid in brazing to strontium and sodium, were considered but were not used since such a pin may result in an unnecessary thermoelectric emf which will be generated as a result of the dissimilar metals.

APPENDIX H

FABRICATION ENVIRONMENT

Due to the highly reactive nature of the alkali and alkaline earth metals, a high-purity argon atmosphere was required for transducer fabrication and assembly. Both sodium and strontium are readily attacked by even a few parts per million of moisture, oxygen, or nitrogen in argon. To reduce the exposure to these elements, the transducers were assembled inside the argon glove box, shown in Figure H-1. Box volume was $\sim 26 \text{ ft}^3$. The box was supplied with plant argon normally having $< 50 \text{ ppm}$ total impurities (3 ppm O_2 , 15 ppm N_2 , 5 ppm moisture, 9 ppm CO_2 , 8 ppm CH_4 , and 7 ppm H_2). Entrance into the glove box was made through a vacuum interlock. When the project was first initiated, the box was continuously purged with a high argon flow while the box argon was also continuously cycled through a 3-inch-diameter by 3-foot-long molecular sieve filled with sodium-alumina silicate, Type

124136

Figure H-1. ARGON GLOVE BOX.

13X, 1/16 inch pellets, to remove moisture. Diffusion of nitrogen and oxygen through the butyl gloves and through the window seals was sufficient to produce a "dirty" environment (0.25% O₂, 1.01% N₂, and 7 to 10 ppm moisture). A uranium-chip pass unit with clamshell-type heaters and with a water after-cooler (shown in Figure H-2) was added to the system, as shown in the flow schematic in Figure H-3. The uranium-chip pass unit was operated at 700° C. The chip unit was made of a 304L stainless steel, Schedule 105 pipe, 4 feet, 2 inches long with an inner 304L stainless steel, 3 1/2-inch Schedule 105 pipe. Argon comes in along the outer annulus, is preheated going down the annulus, and then flows up through the uranium-chip bed. The aftercooler is simply a 3-inch Schedule 40 pipe, 26 inches long, with an inner 3/4-inch Schedule 40 pipe. Argon flows through the 3/4-inch pipe and water through the annular space between the pipes. Glove ports were also blocked off overnight when the box was not in use. The argon environment improved (to 5 ppm O₂, 0.15% N₂, and 3 to 4 ppm moisture), but was still a "dirty" atmosphere in terms of sodium and strontium corrosion. A final effort was made by doubling the pulley size on a motor-driven argon blower. This change caused the argon

124135

Figure H-2 ARGON RECIRCULATION AND PURIFICATION SYSTEM.

Figure H-3. GLOVE BOX PURIFICATION SYSTEM.

flow to slow down to approximately half speed, allowing more resonance time for the argon in the uranium-chip pass unit. The resulting box atmosphere improved to a total impurity count of ~ 56 ppm (13 ppm O_2 , 15 ppm N_2 , < 3 ppm moisture, 14 ppm CO_2 , 8 ppm CH_4 , and 6 ppm H_2).

These were the final modifications made to the environmental equipment. At this point the argon atmosphere was good enough to hold a relatively clean finish on strontium for a few hours, on lithium for days or even weeks, and on sodium for 1 to 2 hours (if they were not heated). Heating lithium or sodium to $200 - 400^\circ C$ resulted in producing an almost instantaneous gray or black film (probably nitride).

During transducer assembly, the transducers were assembled as quickly as possible to reduce the amount of corrosion of the sensor metals.

APPENDIX I

CORROSION TESTS

The alkali and alkaline earth metals are, in general, very reactive. Any reaction involving the pressure sensor grid of sodium or strontium will result in resistance drift as well as a change in the resistive-pressure characteristics, and will eventually render the transducer useless as the sensor disappears through corrosion. It is important that the encapsulation not react with the sensor or that it react at such a slow rate that it can be tolerated. Due to the highly reactive nature of sodium and strontium, corrosion testing is very difficult since they react with only a few parts per million impurities even in an inert gas such as argon. Unless corrosion test equipment becomes very sophisticated, requiring extremely high-purity argon and exotic handling techniques, the sodium or strontium will, in general, be exposed to a gaseous environment during sample preparation or removal. Such exposure will probably corrode the sample. Any corrosion resulting from the environment during sample preparation, test, and inspection, leads to difficulties in determining the amount of corrosion of sodium or strontium due to contact with the encapsulation materials.

One of the best corrosion test procedures and what is probably one of the simplest to perform is to test the assembled transducers themselves. This can be done in two steps:

1. Carry out crude, but simple, corrosion tests in argon with as few impurities as practical by contacting sodium and strontium against possible encapsulation materials and checking for obvious corrosion at room and/or slightly elevated temperatures.
2. Then follow these tests by fabricating several transducers using materials which passed Test 1 and monitor and watch for room and/or elevated temperature-resistance changes over a period of time. Due to the short delivery date for the transducers, this method was used as a means for corrosion testing. The first set of transducers can serve to check corrosion and also be used for shock testing.

A simple corrosion test setup was made where 3/4-inch-diameter discs of strontium or sodium could be stacked against 3/4-inch discs of possible encapsulation materials inside a glass tube. Plant argon (< 50 ppm impurity level) was continuously purged through the glass tube and out through a mineral oil bubbler. The samples and tube were placed on a hot plate where they could be heated from 0 to 350° C.

Table I-1 summarizes the samples tested and basic results. One additional test was run testing sodium and strontium against Mylar. Both metals were placed on a Mylar sheet on a hot plate inside the argon glove box and were held there for 24 hours at each of three temperatures—room, 60° C, and 80 - 90° C, with no visible signs of corrosion which could be attributed to the corrosive action of the Mylar. Normal glove box environmental corrosion of both samples did occur, however.

Table I-1
DATA ON SAMPLES TESTED

Sample 1	Sample 2(1)	Test Temperature (° C)	Test Duration (hrs)	Remarks
Copper	Sr	150	360	Normal environmental corrosion only.
Aluminum	Sr	150	360	Data not recorded.
Beryllium	Sr	150	360	Data not recorded.
Vespel	Sr	150	360	Normal environmental corrosion only.
Epirez 5101 Epoxy	Sr	150	360	Normal environmental corrosion only.
Polyethylene	Sr	150	360	Normal environmental corrosion only.
Beryllium Oxide	Sr	150	360	Normal environmental corrosion only.
Type 304 Stainless Steel	Sr	125	72	Normal environmental corrosion only.
Fluorocarbon Mold Release	Na	Rm	24	Normal environmental corrosion only.
Fluorocarbon Mold Release	Sr	Rm	24	Normal environmental corrosion only.
Fluorocarbon Mold Release	Sr	~100	1	Normal environmental corrosion only.
Fluorocarbon Mold Release	Na	~100	1	Sodium began turning black.

(1) Samples of sodium and strontium against Mylar in the glove box argon were tested for 24 hours at room temperature, 60° C, and 80 - 90° C with no visible signs of corrosion other than normal box environmental corrosion.

Corrosion tests were not run between sodium and its possible encapsulation. In order to conserve time, it was decided by the LRL and Y-12 to use the same type of encapsulation for both the sodium and strontium transducers, if possible (ie, beryllium oxide-loaded epoxy). Based on prior work at Y-12, the epoxy system used was known to be relatively compatible with the alkali metals.

APPENDIX J

GAGE RESISTANCE CALCULATIONS

Transducer resistance was approximated prior to fabrication using resistivity values of sodium and strontium taken from the literature. The resistivity of strontium was taken as 23×10^{-6} ohm-cm, whereas the literature indicates widely separated values for sodium, two of these being 4.3×10^{-6} and 9.7×10^{-6} ohm-cm. Transducer resistance based upon both sodium resistivities were calculated.

Calculated resistances were based upon the equation:

$$R = \rho \sum_i \left(\frac{l_i}{A_i} \right),$$

where:

R represents the transducer resistance in ohms,

ρ the sodium or strontium resistivity in ohm-cm,

A the grid cross-sectional area in cm^2 , and

l the grid length in cm.

Mean grid lengths and areas were used for resistance calculations. The sensor element was divided into three zones: (1) the looped grid pattern with a constant cross section that is 0.030 inch wide by 0.008 inch deep, and a mean length of 2.7534 inches; (2) the 0.030-inch-wide tapered grid section going

Table J-1
CALCULATED STRONTIUM AND SODIUM
TRANSDUCER RESISTANCES

Grid Type	Resistivity (ohm-cm $\times 10^6$)	Temperature ($^{\circ}$ C)	Grid Resistance (milliohms)
Strontium	23	20	115
Sodium	4.3	0	21.5
Sodium	9.7	100	48.5
Sodium	5.54 ⁽¹⁾	23	27.7

(1) Linear interpolation between 0 and 100 $^{\circ}$ C resistivities to obtain an approximate room-temperature value.

from 0.008 to 0.024 inch with a length of 0.1875 inch; (3) the 0.030-inch-wide by 0.024-inch-deep section with a length of ~ 0.128 inch. The resulting grid resistance becomes:

$$R = 0.005 \times 10^{-6} \rho,$$

where the units of R and ρ are still in ohms and ohm-cm. The resulting grid resistances are listed in Table J-1.

BIBLIOGRAPHY

Bridgman, P. W.; Collected Experimental Papers, III, pp 1341 - 1438; Harvard Press (1964).

Bridgman, P. W.; Collected Experimental Papers, V, pp 3203 - 3250; Harvard Press (1964).

Bridgman, P. W.; Collected Experimental Papers, VII, pp 4113 - 4197 and 4213 - 4230; Harvard Press (1964).

Hampel, C. A.; Rare Metals Handbook, 2nd Edition; Reinhold Publishing Corporation, London (1961).

Lange; Handbook of Chemistry, 8th Edition; Handbook Publishers, Inc, Sandusky, Ohio (1950).

Elliot, R. P.; Constitution of Binary Alloys, First Supplement; McGraw-Hill Book Company, New York (1965).

Hansen, P. M.; Constitution of Binary Alloys, 2nd Edition; McGraw-Hill Book Company, New York (1958).

Jayaraman, Klement, and Kennedy; "Phase Diagrams of Calcium and Strontium at High Pressure", Physical Review, 132 (4), pp 1620 - 1624; November 15, 1963.

Gmelins Handbuck Der Anorganischen Chemie, Strontium-Barium-Radium; Verlag Chemie, Berlin (1931).

Gmelins Handbuck Der Anorganischen Chemie, Strontium; Verlag Chemie, Weinheim/Bergstrasse (1960).