Figure 1 also shows a pair of rings R. These are slipped over the tube prior to flattening and confine the flattening to the region desired. Following the flattening process they are readily removed. If excessive pressure is employed, the tube diameter will increase. In most cases this is undesirable. On the other hand, this feature can be employed to advantage if one wishes to increase the size of a tube slightly.

Figure 2 is a photograph showing three objects bent and flattened by the methods discussed above. The largest is a mass spectrometer tube formed from type 304 stainless steel tubing, 1.5-in. o.d. having 0.065-in. wall thickness. The smaller spectrometer tube was formed from type 304 stainless steel tubing, 0.5-in. o.d. having 0.020-in. wall thickness. The third object is a power vacuum tube grid bar having a rectangular cross section for most of its length. It was formed from 5/16-in. o.d., 0.035-in. wall thickness nickel tubing. The first step was to form the round tubing to give it the appropriate offset. Flattening proceeded as for the mass spectrometer tube except that one of the anvils contained a rectangular channel, the other anvil having a mating tongue.

[1] A. O. Nier, Rev. Sci. Instr. **11**, 212 (1940); **18**, 398 (1947).
[2] A. O. Nier, Rev. Sci. Instr. **31**, 1127 (1960).
[3] P. Signer and A. O. Nier, J. Geophys. Research **65**, 2947 (1960).

Fabrication of Miniature Thermocouples for uhf Acoustic Detectors*

STEPHEN A. HAWLEY, JOHN E. BREYER,† AND FLOYD DUNN
Biophysical Research Laboratory, University of Illinois, Urbana, Illinois

(Received July 12, 1962)

THE recent attainment of uhf acoustic wave generation and propagation in liquids[1,2] has necessitated the development of small acoustic detectors. It is the purpose of this note to describe the fabrication of thermoelectric probes useful in the analysis of uhf acoustic fields developed in viscous dielectric liquids. The probe has been used for determining acoustic absorption coefficients, at frequencies as high as 2 kMc, utilizing the transient thermoelectric response (to a single acoustic pulse of rectangular envelope of 0.1-sec duration) produced primarily by the viscous forces resulting from the relative motion between the thermocouple wires and the imbedding liquid.[3,4] The probe is mounted on a three-dimensional coordinate system in such a fashion that the thermoelectric junction is the nearest portion of the probe to the acoustic radiator surface (see Fig. 1).

The arrangement which has provided satisfactory output, as well as minimal construction problems, is a junction formed by butt welding a strand of copper filament to the tip of a pointed loop of Constantan wire. Prior to welding, both Constantan and copper wire are mounted on larger support leads of the same materials, respectively, that are fixed in an epoxy resin holder which, in turn, may be attached to a three-dimensional coordinate system (see Fig. 1). Annealed, commercial Constantan and copper wires 0.0005 in. in diameter, are used for the thermocouple components and are soldered to the supports with fluxless solder. With the aid of a field microscope, the copper filament may be trimmed to an appropriate length and butted against the tip of the Constantan loop prior to welding. At this point, however, it has been the practice to etch the tip of the copper strand and the extreme portion of the Constantan loop to reduce the junction size to a minimum. This is done by first "pointing" the Constantan loop with a fine blade and etching both tips, in the appropriate regions, with a droplet of concentrated nitric acid supported in an eyelet of platinum wire. The surface tension of the droplet is such that the process can be kept under suitable control. The copper tip is usually trimmed again after the etching process to facilitate the formation of a good butt junction.

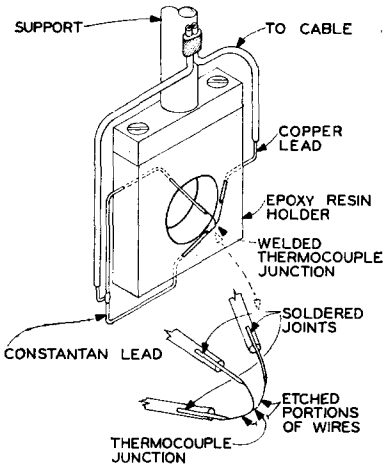

FIG. 1. Schematic diagram of miniature thermocouple for detecting uhf acoustic waves in liquids.

The strands may be butted by manipulation of the support wires and the weld is accomplished by repetitive discharges from condensers of 2-μF capacitance charged to 60 V. Best results have been obtained by continuously pulsing at the rate of 3 pps for a period of 5–10 min during which time the voltage across the capacitance is varied incrementally. Manually butting wires of this diameter is a very tedious operation. However, it has been found that by using a welding apparatus with which charge is continuously forced to exist at the filament extremities, the Coulombic attraction is sufficient, at the short distances involved, to attract the wires into the appropriate position.

The structure of the assembled unit is such that little distortion occurs in translation through viscous fluids. A particular advantage of the detector lies in the microscopic volume occupied by the junction. Thermocouples in which

the maximum dimension of the junction is less than 5 μ (approximately 10-Ω resistance) are readily constructed. This does not represent the minimum size for such probes and procedures for fabricating smaller junctions are currently being developed.

* This work was supported by the Acoustics Branch, Office of Naval Research.

† Present Address: General Electric Company, Ithaca, New York.

[1] F. Dunn, J. Acoust. Soc. Am. **32**, 1503 (1960).
[2] F. Dunn and J. E. Breyer, J. Acoust. Soc. Am. **34**, 775 (1962).
[3] W. J. Fry and R. B. Fry, J. Acoust. Soc. Am. **26**, 294,311 (1954).
[4] W. J. Fry and F. Dunn, *Physical Techniques in Biological Research*, edited by W. L. Nastuk (Academic Press Inc., New York, 1962), Vol. IV, Chap. 6.

Bonding of Lead Telluride to Pure Iron Electrodes

MARTIN WEINSTEIN AND A. I. MLAVSKY
Tyco Laboratories, Inc., Waltham, Massachusetts

(Received June 27, 1962)

A MAJOR limitation to the efficiency of lead telluride thermocouples is the contact resistance at the interface of lead telluride with metal electrodes. Various techniques have been used to reduce contact resistance, such as compression loading at the hot junction together with soft-soldering of the cold junctions.[1] Even with these mechanically complex techniques, the contact resistance still contributes excessively to the total resistance of the thermocouples. Metallurgical bonds between the thermoelements and the electrode are obviously desirable, and we describe some techniques for bonding both n- and p-type lead telluride to iron electrodes.

Standard (I_2-doped) n-type PbTe has been bonded to pure iron electrodes by interdiffusion at 858±2°C for 20 to 30 min. This temperature is 15°C below the PbTe-Fe eutectic, and therefore avoids the melting and recrystallization of the PbTe close to the junction. A dead-weight axial loading of about 500 g/cm² assists in the plastic deformation of the PbTe to conform to the contour of the electrode surface. The other experimental requirements for the bonding are that the parts be scrupulously clean, and that the bonding ambient contain less than 2 or 3 parts per million of oxygen.

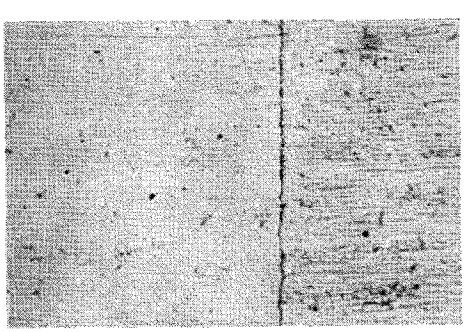

FIG. 1. Photomicrograph of Fe-PbTe (I_2-doped) bond. Deformation of PbTe to fit contour of Fe electrode is seen at junction. 300×.

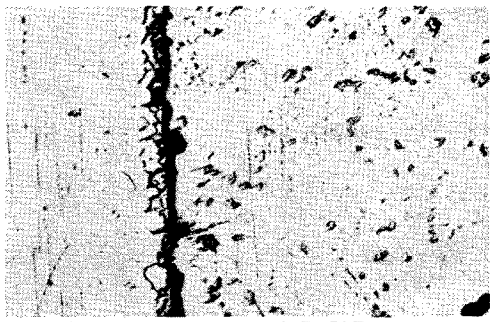

FIG. 2. Photomicrograph of Fe–SnTe–PbTe composite. Region to right is SnTe–PbTe solid solution. Intergranular attack of Fe by SnTe is seen at junction. 200×. Etched 2:1:1 15% NaOH:satd $Na_2S_2O_8$:CH_3OH 15 sec.

Figure 1 shows the interface region of a typical bond. The bond has a contact resistance of less than 10 $\mu\Omega$-cm², and is mechanically stronger than the lead telluride itself. It does not deteriorate in mechanical or thermoelectric properties on extended lifetest at operating temperatures.

Since we were unable to obtain satisfactory diffusion bonds of iron to sodium-doped p-type PbTe, an alternate approach utilizing an intermediate brazing material was explored. The criteria underlying our choice of braze were that it bond independently to iron and to p-type PbTe, that it have an intrinsically high electrical conductivity, that its melting point lie between the upper operating temperature of the thermocouple and the melting point of PbTe, that it form a graded region in the vicinity of the bond to minimize strains from the mismatch in the expansion coefficient, and that it not cause chemical or electrical deterioration of PbTe at operating temperatures. These criteria led us to investigate the use of p-type semimetals with crystal structures related to that of PbTe. Sb_2Te_3 and GeTe were both found to be unsuitable as the former yields a highly resistive two-phase region at a junction with PbTe, and the latter did not wet iron. We have, however, achieved excellent bonds using SnTe. SnTe readily wets iron and also forms a complete series of solid solutions with PbTe. It can be seen from the photomicrograph, Fig. 2, that SnTe penetrates the iron intergranularly, and that SnTe and PbTe form a completely graded solid solution.

In practice, pure stoichiometric SnTe must be used. We found it necessary to zone-level ingots of stoichiometric SnTe several times before a constant melting point was achieved. This was found to be 805±2°C, which is higher than that reported previously.[2] The bond is prepared in two steps. Firstly, powdered SnTe is applied to the iron electrode by melting it on at 850°C for about 1 h; the ex-