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PROCESS DEVELOPMENT AND FABRICATION
OF
PbTe THERMOELECTRIC ELEMENTS

AEC Research and Development Report

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OF
PbTe THERMOELECTRIC ELEMENTS

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ABSTRACT

The development and fabrication of lead telluride pellets, contacted to iron end caps, for use as elements in thermoelectric converters for the SNAP 10A program, is described.

The method adopted consists of contacting, by hot pressing in a controlled atmosphere, 24 pellets simultaneously, using 4-layer, 6-cavity graphite dies.

The optimum hot pressing parameters were found to be 5000 psi at 1550°F for 30 min.

Several thousand elements were produced by this relatively high-volume process, modifying the normally slow and expensive hot pressing by the use of multilayer, multicavity dies and automatic controls.

I. INTRODUCTION

This report describes a process for making lead-telluride thermoelectric elements for the vacuum-design thermoelectric converter for the SNAP 10A Program. The thermoelectric device converts heat from a sodium-cooled nuclear reactor directly to electrical energy without the customary rotating machinery. Figure 1 shows section drawings of the assembly configurations

a. Cross Section of Thermoelectric Couple

b. Configuration of Development Element

Figure 1. Thermoelectric Element

of the thermoelectric elements used in this converter. The elements were made of PbTe, alternately doped with sodium metal (p-type) and lead iodide (n-type). An element is defined as an assembly, consisting of a thermoelectric material with its electrical contacts. One end of the element is connected to a NaK-filled

tube which serves as a heat source, and the other end to an aluminum radiator which acts as a heat sink. The impressed temperature difference generates a thermal emf in the element.

A. PROPERTIES AND PROPOSED USE OF LEAD TELLURIDE

Lead telluride (PbTe) is a semiconductor material with properties which make it particularly attractive for use in a thermoelectric element. It has a relatively low thermal conductivity, which gives a high temperature differential between the hot and cold junctions. Its electrical resistivity can be low enough to permit high current flows with low potential. PbTe can be doped readily to form negative (n) and positive (p) materials. By arranging positive and negative elements in couples, and connecting the couples in series, the potential voltages can be increased to a useful value.

On the other hand, there are certain properties of PbTe which are undesirable, especially from the standpoint of fabrication into useful shapes. It has very low tensile and compressive strengths, and a very high thermal expansion coefficient. These properties, together with its low thermal conductivity, make the material very susceptible to rupture from mechanical and thermal shock. Some properties of PbTe are shown in Table I.

TABLE I
PHYSICAL PROPERTIES OF LEAD TELLURIDE

Density (g/cc)	8.25	
Coefficient of thermal expansion (/°C)	18×10^{-6}	
Compressive strength (psi)	10,000	
Tensile strength (psi)	1,000	
Youngs modulus (psi)	2×10^6	
	n	p
Thermal conductivity (w/cm-°C)	0.024	0.019
Electrical resistivity (75°F) ($\mu\Omega$ /in.)	112	192

B. SELECTION OF HOT PRESSING AS BONDING METHOD

In order to utilize the PbTe thermoelectric elements in the vacuum-design converter, it is necessary to bond contacts to the ends of the elements. These

contacts must have low electrical resistance (compared with PbTe), high thermal conductivity, and must not react with PbTe in such a manner as to destroy the thermoelectric properties of the element. Since little was known of the products of reactions between PbTe and other metals, a decision was made to try to effect a mechanical bond or "joint" between the PbTe and the metal contact. Investigators^{*} at Minnesota Mining and Manufacturing Company recommended iron as a material which did not react with PbTe and had other desirable properties for a contact. It was recognized that there was a considerable difference between the thermal expansion coefficients of PbTe and Fe, and it probably would be necessary to have rough mating surfaces between the PbTe element and the contacts. Therefore, a waffle pattern was pressed onto the iron contacts, to provide the rough surfaces.

Originally, three approaches were taken to attach the contacts to the elements: brazing, unidirectional heating, and hot pressing. No success was achieved with brazing. The braze alloys reacted deleteriously with the PbTe. Unidirectional heating was done by placing the iron contact in a cylindrical recess in the end of an iron rod. The PbTe pellet was clamped on a holder, about 1 in. away from the contact. The iron rod and contact were heated by an induction coil, and the pellet then pressed against the hot contact. The entire operation was carried out in an argon atmosphere. Some limited success was had with this method. However, several incidental problems were encountered, and it became obvious that considerable refinement of the process would be necessary before its potential could be realized. The best initial results were obtained with hot pressing. Because of the limited time available, and the comparatively advanced state of the art of hot pressing, the decision was made to pursue this approach. After considerable experimentation, a reference hot pressing process was established.

C. LIMITATIONS OF HOT PRESSING AS A PRODUCTION METHOD

Hot pressing is generally a slow and expensive process, because it is not adaptable to continuous operation. In addition, it sometimes is necessary to make the dies from an expensive material which is difficult to fabricate. Special precautions are necessary, to protect the dies and other components from

^{*}Personal Communication

atmospheric attack at high temperatures. For these reasons, as a rule, hot pressing is used as a fabrication method only if no other acceptable method can be found.

In this instance, however, most of these objections were eliminated by the fact that this was a low-temperature, low-pressure operation, and multiple pressings could be made. The small size (0.500 in. in diameter by 0.200 in. long) of the PbTe elements made multilayer and multicavity pressing feasible. Although the pressing had to be done in an inert atmosphere, the low temperature (1550°F) at which the operation was carried out eliminated many of the containment problems usually encountered in controlled-atmosphere hot pressing.

II. DESCRIPTION OF APPARATUS

A PRESSES

The presses used are 30-ton, motor driven, hydraulic presses with 5-in diameter, down-acting, spring-return cylinders. Each press is equipped with a nitrogen accumulator which permits pressure to be maintained throughout the pressing cycle. Figure 2 shows the bank of four hot presses.

B FURNACES

Furnaces used to heat the hot pressing setups are Nichrome-wound muffle tube furnaces, 3 in ID by 12 in. long (See Figure 3).

C DIES

Pressing dies are machined from National Carbon Company, grade ATJ graphite. Three-cavity die bodies are slightly less than 2-1/2 in in diameter. Cavities are 0.512 in in diameter, and the punches are 0.509 in. in diameter. Figure 4 shows a three-cavity die and punches.

D RETORTS

Pressing retorts are made of 2-1/2-in ID by 0.200-in wall Inconel pipe with a Type 304 stainless steel flange and cover. The cover is water cooled by copper cooling coils soldered to the top surface. The hot pressing ram is sealed in the cover by an O ring. Figure 5 shows a cutaway drawing of the hot pressing assembly.

E CONTROLLERS

Controllers are program controller-recorders with timers and interrupters. Power is supplied to the furnaces by three 2-kva saturable core reactors.

Figure 2. Hot Presses

7580-1840

Figure 3. Hot Pressing
Furnace

7580-1842

Figure 4. Hot Pressing Die and Punches

Figure 5. Hot Pressing Assembly

NAA-SR-7036

III. RESEARCH AND DEVELOPMENT ON PbTe, USING HOT PRESSING

A. EVOLUTION OF REFERENCE PROCESS

The preliminary experimentation, on which the reference process is based, was carried out by N. H. Katz, and is described in Reference 1. Thus, it will be only summarized here. Verification of the results obtained by Katz, and experimentation on powder metallurgy fabrication of PbTe elements with Fe powder contacts, were conducted by B. N. Briggs, and are described in Reference 2.

Hot press runs were made, using graphite dies at several pressures to 5000 psi, temperatures to 1600°F, and times to 2 hr. Several combinations of parameters gave equally good low cap-to-cap electrical resistances of the bonded elements after hot pressing. However, when the elements were subjected to thermal cycling, such as would be encountered in subsequent processing and during the operation of the thermoelectric device, it became evident that the optimum hot pressing parameters were 5000 psi, 1550°F, and 30 min (Figure 6 shows the increase in electrical resistance during thermal cycling). Hot

Figure 6. Cap-to-Cap Resistance vs Temperature for Elements at 4000 psi

pressing of elements, using these parameters, was established as the reference process; and the production of elements was begun. In the meantime, further developmental work was carried out, to verify the original data on hot pressing and to find an alternate method of bonding. Verification of the optimum hot pressing parameters was made, using reference iron contacts. Two other approaches to contacting of elements were investigated: (1) powder metallurgy fabrication of elements, using Fe and PbTe powders, and (2) deposition of Fe contacts on the ends of PbTe pellets by electroplating.

Some promising results were obtained with the powder metallurgy method, but it was not possible to sinter the Fe-PbTe bodies to a sufficiently high temperature to densify the Fe contacts. Erratic results in cap-to-cap resistances were obtained, and the Fe powder contacts were very porous and friable.

Electroplating of Fe contacts gave an element having a much lower cap-to-cap resistance than could be obtained by any other method of contacting. However, the contact was porous, and had a tendency to spall off during thermal cycling. A serious problem was revealed, during attempts to braze the elements into modules. The porous Fe contact absorbed the braze alloy, which then reacted with the PbTe. Further experimentation reduced these difficulties, but it was not possible to eliminate them. Some representative data on electroplated elements are shown in Table II.

The work done by Katz¹ was performed, using single-cavity dies, and some experimentation was necessary to effect the transition to three-cavity dies. Since it was necessary to use the same retorts and furnaces, the three-cavity die walls were considerably thinner than were those of the single-cavity dies. Because of this, it was necessary to more rigidly control the upper pressure limit on the rams, in order to prevent rupturing of the dies. The heights of the three 4-layer stacks had to be the same, within 0.005 in., to prevent unequal loading of the stacks. However, it was not necessary to alter the hot pressing parameters, in order to produce satisfactory elements.

B. IRON-TO-IRON "SANDPAPER" CONTACTS

The use of "sandpaper" contacts (made by sintering Fe granules onto Fe sheet) was attempted, on the basis of expected increased "interlocking" with the PbTe. Results were very promising; in that, while elements with these contacts

TABLE II
CAP-TO-CAP RESISTANCE OF IRON-ELECTROPLATED
PbTe PELLETS

Pellet Number	Resistance ($\mu \Omega$)	
	Before Plating	After Plating
	n Elements	
63-1	70	62
2	121	79
3	79	88
4	43	81
5	50	58
6	62	99
	p Elements	
80-5	59	124
6	128	135
7	158	105
8	146	143
9	45	151
10	67	127

had a cap-to-cap resistance no lower than the reference elements, degradation* during subsequent processing was greatly reduced. However, it was impossible to braze these elements into modules, because the braze alloy would not wet the contacts. This was attributed to the high carbon content of the iron granules (cast iron, 3% C) which diffused into the iron sheet. This difficulty was overcome by first decarburizing the contacts, through heating in wet hydrogen to 1650°F for 1 hr, and then reducing the surface oxidation, by heating in dry hydrogen to 1800°F for 1 hr. One very limited experiment has indicated that it is possible to reduce the temperature for hot pressing, and still have good resistance to thermal cycling degradation. In this instance, the cap-to-cap resistances of two p-elements, after hot pressing at 1100°F and 5000 psi for 30 min, were 210 and 240 $\mu \Omega$ (compared with two reference elements, pressed simultaneously, having cap-to-cap resistances of 230 and 296 $\mu \Omega$). After thermal

*Degradation is defined as the loss in thermoelectric output, under reference test condition. In general, it is synonymous with increased electrical resistance.

cycling, the elements with sandpaper contacts had cap-to-cap resistances of 297 and 300 $\mu\Omega$, while the reference elements showed 590 and 764 $\mu\Omega$ resistances. A 22-element module, made with sandpaper elements, showed a total resistance of only 5387 $\mu\Omega$. The resistances of the elements in this module, at the various stages of fabrication, are shown in Table III.

TABLE III
RESISTANCE OF "SANDPAPER" END CAPS ASSEMBLED INTO
A 22-ELEMENT MODULE*

Element Number	Cap-to-Cap Resistance ($\mu\Omega$)		
	Hot Pressed	Encapsulated	Assembled
n Elements			
A-1	157	157	140
2	119	173	148
3	111	163	146
4	156	158	150
B-1	165	189	145
2	132	173	143
3	170	195	146
4	164	202	150
C-1	179	192	148
2	148	170	150
3	173	167	141
p Elements			
A 1	373	373	300
2	388	376	320
3	357	395	320
4	361	397	326
B-1	351	370	320
2	347	378	342
3	379	390	327
4	414	404	338
C-1	396	357	309
3	403	387	307

*Total resistance of the module 5387 $\mu\Omega$

C. ALUMINUM CONTACTS IMPREGNATED WITH IRON GRANULES

Aluminum is attractive as an end cap material for PbTe thermoelectric elements for the following reasons:

- 1) Close thermal expansion match with PbTe
- 2) Excellent thermal and electrical conductivity
- 3) Low density
- 4) Ease of bonding, in assembling the thermoelectric module

The compatibility of aluminum with PbTe thermoelectric pellets, under assembly and use conditions, is not yet accurately known. Indications are that the presence of the aluminum oxide film seems to result in a high contact resistance. To overcome this difficulty, iron granules (50 to 30 mesh in size) have been imbedded, by cold pressing at 3000 psi, in the aluminum discs to pierce the oxide film and give a better mechanical bond. Hot pressing these end-caps with PbTe pellets at 3000 to 4000 psi and 1150°F for 5 to 10 min resulted in p elements of low resistance, as shown in Table IV. Metallographic investigation of a hot pressed element revealed some reaction between the aluminum and iron, but only a mechanical bond between the aluminum and PbTe.

TABLE IV
RESISTANCE OF p ELEMENTS WITH IRON-IMPREGNATED
ALUMINUM END CAPS

Element Number	Resistance ($\mu \Omega$)	
	Before Hot Pressing	After Hot Pressing
472-1	99	220
2	119	210
3	131	232
4	123	255

Small changes in the original procedure (e.g., smaller size of the iron grains, or attaching the iron powder to the aluminum with Krylon instead of imbedding it) resulted in poor bonding or high resistance.

The experimental work conducted so far shows that elements with aluminum contacts can be exposed to temperatures greater than 1100°F for a limited time only (not in excess of ~15 min). Otherwise their resistances increase considerably. Preliminary results of the heat cycling tests conducted show that Al end-cap elements, pressed at 4000 psi, are superior to those pressed at 3000 psi and to the reference elements (Table V).

TABLE V
ELECTRICAL RESISTANCE OF ELEMENTS, BEFORE AND
AFTER THERMAL CYCLING

Type of Element	Resistance ($\mu \Omega$) (Average of 7 Specimens)	
	Before Cycling	After 1 Cycle to 1100°F and 3 Cycles to 1000°F
Reference	245	460
Al End caps pressed at 3000 psi	274	565
Al End caps pressed at 4000 psi	204	312

Figure 7. Loading Pellets and Contacts in Die

7580-1846

Figure 8. Loading Die in Retort

Figure 9. Bolting Retort Cover

7580-1845

IV. PRODUCTION PROCEDURES

Production of bonded PbTe elements is carried out in the following manner. PbTe pellets, 0.500 in. in diameter by 0.170 in. long, are cleaned by washing in acetone and drying on lint-free tissue. The Fe contacts, 0.020 in. thick by 0.500 in. in diameter, are cleaned by degreasing in a trichlorethylene-filled ultrasonic cleaner. The pellets and contacts are loaded into the die cavity, as shown in Figure 7. Graphite spacers, 1/4 in. long, are used to separate the element assemblies from each other. Pressure is transmitted to the pellets by the longer of the two graphite plungers (Figure 7). After the die is loaded, it is placed in the pressing retort, and the cover is bolted into place (see Figures 8 and 9). The retort is evacuated to a pressure of about 0.1 mm Hg, and is then located in a Nichrome-wound resistance furnace on a hydraulic press. Figure 5 shows a section through the hot pressing setup. Vacuum is maintained on the retort during heating to 400°F, in order to outgas the element assemblies. At this point, the retort is filled with argon to just over 15 psia. This atmosphere is maintained throughout the remainder of the cycle. The temperature of the die is raised from 400 to 1550°F, in about 1-1/2 hr, by the automatic furnace controls. A pressure of 5000 psi is applied to the pellets when the temperature has reached 1550°F. After 30 min at 1550°F, the power is shut off by the controller, and the pressing assembly is allowed to cool to 1200°F in the furnace. At this point, the pressure is released, and the retort is removed from the furnace and placed in the cooling rack (Figure 10). When the assembly has cooled to room temperature, the die is removed from the retort and the elements are pressed out of the die (Figure 11). The bonded elements are cleaned with a dental abrasive grit blaster, and are sent to inspection.

The procedure described applies equally to single- or multiple-cavity dies; except that, with multiple-cavity dies, care must be taken to be sure that the loadings in each die are the same height, so that equal pressure is applied to each element. No difficulties are encountered in the use of multiple-cavity dies, as long as this precaution is taken.

Figure 10. Furnaces in Cooling Rack

7580-1851

Figure 11. Ejecting Elements From Die

V. ANALYSIS OF PRODUCTION

A production rate of 144 elements per day was attained; and, since this was primarily an engineering effort, a 100% inspection was made of the production. Each element was inspected for cap-to-cap resistance, length, and flatness. Originally, the following acceptability limits were imposed:

- a) Cap-to-cap resistance — less than $600 \mu \Omega$
- b) Length — 0.210 ± 0.010 in.
- c) Flatness — 0.002 in. TIR

The limit for cap-to-cap resistance was reduced first to $< 400 \mu \Omega$, and then to $< 300 \mu \Omega$. The limits on length and flatness were not changed. Figure 12 is a graph of the average cap-to-cap resistance, on a weekly basis, and the

Figure 12. 3-Sigma Control Limits for Hot-Pressed Elements (1 July to 8 December, 1961)

3-sigma (99.7%) limits, on a monthly basis. The significant improvement in quality is attributed to constant efforts to maintain better control of the process by frequent calibration of temperature controllers and pressure gauges, constant attention to the apparatus during operation, and continuous checks of the retorts to insure that no atmosphere leaks developed.

Since the three cavities in the die were concentric with the longitudinal axis of the die, no variation, from cavity to cavity, was noted. However, there did appear to be some variation, from top to bottom, within the 4-layer stack. An analysis of this variation was made, and it was found to be within the limits of reproducibility of the cap-to-cap resistance measuring device. Occasionally, a run would be made in which the three bottom elements would come out slightly shorter than the specified 0.200 in. This was believed to have been caused by a temperature gradient over the length of the die. The phenomenon was almost always present when the temperature controller began to override the 1550°F limit by 20 to 30°F. Calibration and resetting of the controller eliminated the difficulty.

No difficulty was encountered in maintaining the flatness tolerance, as long as the graphite spacers between elements in the stack were not allowed to become worn or chipped.

VI. PRODUCTION POTENTIAL OF HOT PRESSING

The fact that 4-layer, 3- and 6-cavity pressings were employed is not an indication that these are necessarily the limits for this process. Based on experience gained with the 6-cavity die, it appears likely that 5-layer, 12-cavity dies could be used with very little additional modification. The number of pressings would be determined by the size of the press and the length of the furnace. At some point, the diameter of the furnace becomes so large that heatup time is prohibitive and diametral gradients are intolerable, but 4-layer, 6-cavity pressings can be accomplished very nicely. Table VI shows the comparative number of parts per hour which could be produced with various die capacities.

TABLE VI
HOT PRESSING PRODUCTION RATES WITH MULTIPLE DIES

Die		Number of Pellet per Pressing	Duration of Pressing Cycle (hr)	Production Rate/Press	
Cavities	Layers			Hourly	Daily (24 hr)
1	4	4	2	2	48
3	4	12	2-1/2	4.8	115
6	4	24	3-1/2	6.85	164
12	5	60	5-1/2	10.9	262

VII. SUMMARY

A production method for bonding Fe contacts to PbTe thermoelectric elements was developed by adapting controlled-atmosphere hot pressing. This normally slow, expensive process was modified to a relatively high-volume process by the use of multilayer, multicavity pressing dies and automatic controls. Several thousand elements were produced by this process.

Alternative methods of contacting PbTe pellets investigated were: cold pressing and sintering of PbTe and Fe powders, electroplating of Fe on PbTe, brazing of Fe to PbTe, and unidirectional heating. Hot pressing was superior to any of these, from the standpoint of consistently yielding an element which would meet specifications. "Sandpaper" contacts, consisting of Fe on Fe or Fe on Al, appear to be somewhat better than the reference contact, Fe sheet with a stamped waffle pattern.

There appears to be no theoretical limitation to the number of elements which could be simultaneously produced by multiple hot pressing, although there are practical limitations imposed by heatup times and size of equipment.

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