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FINAL REPORT
ADVANCED SELENIDE THERMOELECTRIC
DEVELOPMENT PROGRAM
TES-32075-107

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I. INTRODUCTION

The primary objective of this work was to demonstrate that copper silver selenide and TAGS could be segmented. The hot junction temperature was planned to be 725°C with the segmentation temperature at 400°C, both temperatures were selected to prevent excessive sublimation from the hot ends of the segments, respectively.

The program was planned as a cooperative effort between General Atomic Company and Teledyne Energy Systems. Accordingly, General Atomic synthesized the CuAgSe that was used to fabricate the test hardware that was ultimately delivered to General Atomic for testing. Both the CuAgSe and TAGS were hot pressed in an argon atmosphere then the segments were furnace-bonded to each other.

A secondary objective was to produce CuAgSe powder by rapid solidification.

II. SUMMARY OF WORK

Several copper silver selenide (CuAgSe (CAS)) ingots were synthesized from elemental materials for use in hot pressing studies. A glove box was modified to provide a high purity inert atmosphere in which to melt and hot press CAS and hot press TAGS. A hot-press furnace, dies, and a melting crucible were designed and fabricated. Hot-press parameters were established for CAS and TAGS. A bonding agent to join TAGS to CAS was developed and used. Seventy (70) CAS legs were hot pressed; forty-eight (48) TAGS legs were hot pressed; twenty-two (22) segmented legs were assembled. Thermal conductivity measurements were made on 2 CAS legs and 2 TAGS legs. Seebeck and Resistivity measurements were made on 3 CAS legs and 3 TAGS legs. Tensile tests were made on three CAS legs, three TAGS legs, and two segmented legs. Work was initiated on the rapid solidification of CAS material. Twelve (12) segmented legs with "top hat" contacts were fabricated and four (4) of these legs were submitted to GA for evaluation and test.

III. WORK ACCOMPLISHED

1. Material Preparation

A. CAS

Copper silver selenide was synthesized in-house according to the formula

$\text{Cu}_{1.97} \text{Ag}_{.03} \text{Se}_{1.0045}$. Five, twenty-gram batches of material were formulated and sealed in 15 mm OD graphite-coated Vycor tubes at 200 microns maximum pressure. All materials were 99.999+% purity. The copper and silver were in shot form while the selenium was approximately 1/8" fragments.

The sealed ampules were placed into a larger Vycor tube and the material was reacted at 1100°C for 2 hours in a rocking furnace, then furnace cooled to room temperature. The ingots had the visual appearance of being homogeneous and were a dark metallic grey in color.

B. TAGS

The TAGS material was synthesized per Teledyne Standard Process Specification PS 0500004 which describes the procedures for preparing, melting, and annealing this material.

2. Glove Box Preparation

Since all fabrication of CAS must be done in an inert atmosphere (argon), an existing glove box was modified for hot pressing, melting, and rapid quench of CAS material. This involved an overhaul checkout of the Dri-Train purifying system, installation of electrical, thermocouple, water, hydraulic, and argon (nitrogen) lines, installation of a powder blender, pressing frame, furnace, and pulleys for raising the furnace to the ceiling of the dry box. In addition, a new window with raised glove ports was fabricated and installed. Other installed items included a triple beam balance, mortar and pestle, small fan, high intensity lamp, and various size sieves.

The blender motor was mounted external to the box as was the furnace heater Variac, and hydraulic cylinder(s) pump. Other external equipment included pass-thru chamber vacuum pump, Dri-Train, Lockwood and McLorie Oxygen Analyzer, Panametrics Moisture Analyser, and Veeco thermocouple vacuum gauge. The glove box and ancillary equipment are shown in Figure 1.

3. Hot-Press Furnace and Die Design

CAS and TAGS were hot pressed in a resistance wound furnace placed on a press frame with dual ten-ton capacity hydraulic cylinders. Initially, the CAS was hot pressed in a graphite-coated molybdenum die with Mo die pins but this was changed to a POCO graphite body in a Mo strength member with POCO spacers and tungsten pins. TAGS was hot pressed in an ATJ graphite die with ATJ spacers and tungsten pins. These equipment items are shown in Figures 2, 3, 4 and 5.

4. Powder Studies

A. CAS

Mechanically cleaned CAS ingots were crushed in the mortar and pestle in the glove box and hand sieved through 230, 270 and 325 mesh sieves. Various mesh fractions were investigated in the course of process development, the final sieve fractions used to fabricate the hardware were 15 w/o -230 + 270, 25 w/o -270 +325 and 60 w/o -325. The sieve fractions were "Vee" blended 15 minutes to assure homogeneity. This material was used to establish the hot-press process.

A 100-gram ingot of CAS, received from General Atomic Company (GA) was used to fabricate all of the actual test specimens.

B. TAGS

The TAGS ingot was crushed in air in a mortar and pestle, then screened 15 minutes through 60, 120, 170, 230, and 325 mesh sieves. The sieve fractions used in this work were 40 w/o -60 + 120, 15 w/o -120 + 170, 15 w/o -170 + 230, and

15 w/o -230 + 325. These fractions were "Vee" blended for 15 minutes to assure homogeneity.

5. Hot Pressing Parameters

Using 3M generated CAS property data and desired generator configuration, the calculated dimensions for the CAS segment are 0.125 inch diameter by 0.140 inch long; using TAGS data, the dimensions for the TAGS segment are 0.125 inch diameter by 0.230 inch long.

A. CAS

GA's vacuum hot-press schedule was modified for hot pressing in argon. The modified process is as follows:

- (1) Place 0.20 grams of blended powder into die.
- (2) Preload pins to 100 psi.
- (3) Heat to 600°C, then press to 20TSI.
- (4) Hold at 20TSI and 600°C for 30 minutes.
- (5) After 30 minutes release pressure and heat to 800°C.
- (6) Hold at 800°C for 30 minutes.
- (7) After 30 minutes, shut off furnace and allow to cool to 500°C.
- (8) At 500°C, remove die from furnace and argon cool to 30°C.

B. TAGS

Various time, temperature and pressure combinations were investigated with the final process as follows:

- (1) Place 0.30 grams of blended powder into die.
- (2) Heat die to 400°C, then 15 TSI pressure.
- (3) After 15 minutes at pressure, cool while under pressure to 200°C.
- (4) At 200°C, release pressure, remove die from furnace, and argon cool to 30°C.

6. Diffusion Barrier and Segment Bonding Studies

Because of possible interdiffusion of TAGS and CAS, it was necessary to identify a diffusion barrier and bonding procedure with which to join the segments.

A. Diffusion Barrier Studies

Compatibility tests were run with TAGS in pressure contact with iron (Fe), nickel (Ni), TZM (molybdenum alloy), copper (Cu) and tungsten (W) at 350°C (heat losses from the test fixture prevented testing at the desired temperature, 500°C) for 72 hours in argon. The test results indicated no reaction with TZM or W and superficial reaction only with Fe, Ni, and Cu.

Since TAGS is bonded to iron cold caps in SNAP 19-type thermoelectric couples and the Fe/TAGS reaction at 350°C is so slight, 3-mil-thick iron was chosen as the diffusion barrier if it could be bonded to CAS. Subsequent hot-press studies showed that iron could be readily joined to CAS during the fabrication of that segment.

B. Segment Bonding Studies

In the SNAP 19-type thermoelectric couples, tin telluride (SnTe) with excess tin is used to join iron shoes to TAGS at the cold end. The TAGS-CAS interface will be operated at a nominal 400°C, therefore SnTe with elemental tin present would not provide a suitable bond. A TES proprietary bonding agent (441 bond) was selected because it had exhibited good "wetting" and good high temperature properties in tests from an earlier in-house program. This bond material contains nickel, therefore the 3 mil iron diffusion barrier was nickel plated on the mating surface to enhance the bonding action. Bonding studies with the 441 material showed it to be suitable for joining TAGS to iron.

C. Microprobe Analysis of CAS-TAGS Bond

A microprobe analysis across the CAS-TAGS interface as-bonded revealed that the iron was an effective barrier as no copper or selenium was found in the TAGS material.

7. Segmented Leg Assembly Details

A. CAS Leg to Hot Shoe

During the hot pressing operation, described earlier in Section 5A, a 3 mil nickel-plated (one surface) iron diffusion barrier is simultaneously bonded to the compacted powder with the nickel plate facing away from the CAS as shown in Figure 6, Step 1. Mo-46Re hot shoes, referred to as "top hats," fabricated by GA were prepared for bonding to the previously hot pressed CAS segments by spot welding two W-Re screens in the "top hat" depression to increase bond area and provide mechanical strength. The top hat was assembled into the hot pressing die with the bare end of the CAS segment contacting the W-Re screen. The assembly was hot pressed at 450°C and 18TSI for 15 minutes. After 15 minutes the die was cooled to 200°C while under pressure, then the pressure was released and the assembly removed from the furnace and argon cooled to 30°C, Figure 6, Step 2.

B. Bonding TAGS to CAS

After hot pressing TAGS segments as described in Section 5B, they are stress relieved at 465°C for 1 hour in argon, Step 3, Figure 6. A five-mil-thick disc of the tin telluride exothermic formulation was used to bond the cold contact to the TAGS segment while a seven-mil-thick disc of 441 exothermic formulation was used to bond the TAGS to the nickel plated surface of the iron barrier previously joined to the CAS segment, Figure 6, Step 4.

The various components were assembled into a Vycor-enclosed bonding fixture, Figures 7 and 8, and heated to 570°C in hydrogen. Upon reaching temperature, the fixture was withdrawn from the furnace and allowed to air cool to room temperature.

8. Production of CAS Powder by Rapid Solidification

A procedure for the rapid solidification of molten CAS material was developed. The hot-press furnace was used to fuse CAS material in a graphite crucible. The crucible has a hole in the bottom, the hole is stoppered with a graphite plunger. A sketch of the crucible is shown in Figure 9. The bottom of the furnace was suspended approximately 24 inches above the glove box floor. When the CAS was molten, the graphite plunger was extracted from the hole and the molten CAS dropped through the hole in the bottom of the crucible. The CAS material was heated to temperatures ranging from 2000° to 2160°F (1093° to 1182°C) with better flow at the higher temperature. The ten-mil hole in the crucible had to be enlarged to 47 mils to achieve flow. Ten-gram charges of material were melted and dropped into a stainless pan and into liquid nitrogen. In both cases the powder produced was spherical, approximately 1/32 to 1/16 inch in diameter. Time limitations did not permit sufficient process development to refine the rapid solidification procedure. Atomization of the molten CAS by a gas stream as it dropped from the crucible was intended but was not done.

9. Tensile and Shear Tests

A. Tensile Tests

Tensile tests were run on three CAS legs, three TAGS legs and two segmented legs, using a Hunter Spring Force Gage as shown in Figure 10. Because the legs were so small, it was not possible to mechanically grip the specimens for testing so Eastman 910 adhesive was used to secure the samples to machined anvils.

B. Shear Tests

Shear tests were run on three each of CAS and TAGS legs utilizing the shear fixture in the Hunter Spring Force Gage Shown in Figure 10.

10. Seebeck and Resistivity Measurements

Because of the small size of the legs, 0.125 inch diameter by 0.370 inch long, new fixturing had to be fabricated to run the test (Figure 11). The test setup is shown in Figure 12. Three each of the CAS and TAGS materials were tested and the results are shown in Figures 13 and 14.

11. Thermal Conductivity Measurements

The thermal conductivity apparatus was not designed for samples smaller than 3/8 inch in diameter so 1/2 inch diameter legs were fabricated for this test.

Two each of CAS and TAGS materials were tested and results are shown in Figures 13 and 14.

IV. TEST RESULTS

A total of thirty (30) CAS legs were hot pressed from TES-made material and forty (40) legs were pressed from GA furnished material.

Forty-eight (48) TAGS legs were hot pressed.

Fourteen (14) segmented legs were assembled without "top hat" shoes and twelve (12) segmented legs were assembled with "top hat" shoes and four of these were submitted to GA for testing.

A. Density Measurements

Density measurements were made on the hot pressed CAS and TAGS legs and are tabulated below:

CAS (GA material) Range-6.32 to 6.71 gm/cc - average of 28, 6.57 gm/cc

CAS (TES material) Range-6.29 to 6.6/gm/cc - average of 23, 6.42 gm/cc

This compares with a GA range of 6.5 to 6.8 gm/cc for vacuum hot pressed CAS.

TAGS Range-5.75 to 6.40 gm/cc - average of 43, 6.13 gm/cc

This compares with a reported 100% density of 6.44 gm/cc.

B. Room Temperature Resistivity

Room temperature resistivity measurements were made but because of the leg size it was difficult to obtain accurate readings as the voltage probes had to be hand held.

Average resistivity for 30 CAS legs was 7.09 milliohm-cm while for 32 TAGS legs it was 0.865 milliohm-cm. The 7.09 milliohm resistivity for CAS was 273% higher than the value of 1.9 milliohms which was extrapolated from the 3M data curve while the TAGS 0.865 milliohm-cm resistivity was 50% higher than the 0.575 milliohm-cm resistivity extrapolated from the TAGS curve.

C. Elevated Temperature Resistivity

Elevated temperature resistivity measurements were made on three each of CAS and TAGS legs between room temperature and 700°C for CAS and between room temperature

and 500°C for TAGS as shown in Figures 13 and 14.

The first CAS and TAGS elements tested had questionable readings and are not presented. The test apparatus was then modified to improve its accuracy. The measured resistivity of the two remaining CAS legs was lower than the 3M data and did not rise as rapidly with increase in temperature. These legs were tested with the three-mil iron cap bonded to one end (refer to Figure 6, Step 1) as this was how they were fabricated. This may have affected the test results.

On the other hand, the two hot pressed TAGS elements were higher in resistivity than the cast element resistivity data. The resistivity increased, then dropped with an increase in temperature. The increased resistivity is probably due to the fine grain structure of the hot pressed element.

D. Elevated Temperature Seebeck

Seebeck measurements were made on the same elements used to obtain resistivity measurements. Both CAS elements were lower in Seebeck than the 3M data and dropped off rapidly with increasing temperature. The size of the elements, 0.125 inch diameter by 0.140 inch long, made it very difficult to install them in the fixture and instrument. The material reacted with the thermocouples in the first test. In later tests a half-mil Mo foil was used as a barrier between the CAS and the thermocouple. On samples tested at temperatures above 500°C, small copper colored beads were observed on the element surface. These anomalies probably contributed to the discrepancy of the data compared to 3M data.

The hot pressed TAGS Seebeck data appears to match the cast data reasonably well.

E. In Gradient Test - Segmented Legs

In gradient tests were not made because it was not possible to instrument the

segment at the CAS-TAGS interface. The materials involved reacted with the thermocouples, causing improper temperature readings.

F. Tensile Tests

In initial attempts to fracture the element, the glue joint parted before the element fractured. Tensile tests on three CAS legs showed that the element strength ranged from 1092 to 1703 psi. The iron diffusion barrier parted from the leg at 924 psi on one sample. The other two legs did not have iron diffusion barriers.

Tensile tests on three TAGS legs were unsuccessful because the adhesive joint always broke first. Apparently this material does not bond well with this adhesive. The adhesive joints broke at pressures ranging from 532 to 1196 psi.

Attempts to run tensile tests on ten segmented legs resulted in breakage of eight legs during the test setup. This occurred because the legs were so small they required handling with tweezers. Five legs broke at the CAS-Fe interface while three legs broke at the Fe-441 bond. The remaining two legs broke at the CAS-Fe interface at tensile values of 81 and 83 psi, respectively.

G. Shear Tests

A shear test fixture, shown in Figure 10, was fabricated for testing the .125 diameter elements. Three TAGS elements were tested. They sheared at 3322, 3454 and 1606 psi respectively, for an average shear strength of 2794 psi. These elements were long enough to extend through the fixture jaws. Each was sheared in two places for an actual average shear value of 1397 psi.

The CAS elements were not long enough to extend through the fixture jaws, hence sheared in one place only. The values for three samples were 3339, 2401 and 2029 psi for an average shear strength of 2590 psi.

H. Thermal Conductivity Measurements

In order to do thermal conductivity measurements it was necessary to make two special elements 1/2 inch in diameter by 1/2 inch long. A resistance wound heater was placed between the two instrumented elements and the ΔT was measured across a measured distance in the element. Valid readings are dependent upon good contact between the elements and the heater, good insulation around the element exterior and good thermocouple contact.

The thermal conductivity values for both the CAS and TAGS were very scattered and much higher than the reference values. The GA hot pressed CAS material was about 3 mw/cm-°C higher at the elevated temperatures than the corresponding 3M values for cast material. When the test samples were examined, there were small beads on the surfaces and the surface was cracked and oxidized. The heater contact areas appeared non-uniform. These factors are probably associated with the high k values.

There were no anomalies in the appearance of the TAGS elements so the 5 mw/cm-°C difference cannot be explained on that basis. The higher k values for the hot pressed material versus the cast material was an unexpected result. It definitely appears that the 221°C point is in error.

I. Room Temperature CAS and TAGS Leg Resistances and Segmented Leg Bond Resistance

Room temperature resistances were measured on the segmented legs. The test method consisted of passing an AC current through the leg then hand probing the various bonds and segments with voltage probes to get a resistance reading. Hand probing led to variability in segment resistances as well as total resistances.

Resistances on the individual legs alone did not always agree with those values obtained when the leg was bonded to form a segmented leg. Table 1 presents a comparison between measured resistances vs calculated resistances assuming a resistivity of 1.9 milliohm-cm for CAS and .650 milliohm-cm for TAGS, the values extrapolated to 20°C from Figures 13 and 14.

The measured leg resistances for CAS varied from 5.2 to 60 milliohms whereas the calculated resistances should have been between 7 and 9.5. The ratio of measured to theoretical resistance ranged from 0.74 to 7.4, with the GA material averaging 3.5 and the Teledyne material 4.4.

It was noted that the measured resistances of the TAGS was much more consistent both in the individual leg and bonded segment condition. The CAS resistances varied widely in both conditions with the GA supplied material providing consistently lower resistances than the Teledyne material.

Table 2 shows the resistances measured along the segmented legs. Because of the hand-held-probe method used the totals do not always agree. The resistance of the CAS segments both increased and decreased from the individual leg resistance after bonding. It is not known if this was due to a change in properties or test setup/instrumentation.

The TAGS bonded element resistances were consistently lower than the individual leg resistance. The cold bond at the TAGS end ranged from .01 to .27 milliohms (ignoring one high value) while the intermediate bonds ranged from 0.3 to 3.5 milliohms.

The hot bond resistance ranged from 5 to 9.5 milliohms. This bond is the CAS interface with the double mesh W-Re screen spot tacked into a dished Mo-Re "top hat."

If the measured resistances of the CAS and TAGS is deducted from the total resistance, the remainder should be attributable to the bonds. When this was done 11 out of 22 samples had bond resistances in excess of 5% as shown in Table 3. But the measured resistances of the CAS and TAGS were probably low because the hand probes could not measure the entire length of the segment.

Using the measured bond resistances as a criterion, nine of the legs had bond resistances exceeding 5% and six of these legs were ones with "top hat" hot shoes. The measured bond resistances are probably high because the hand-held probe method included part of the elements on each side of the bond. These higher measured values would result in a higher percentage of the total resistance than is actually the case.

V. CONCLUSIONS

1. Hot pressed CAS and TAGS elements were fabricated successfully in an argon atmosphere.
2. CAS was hot pressed into dished shoes. Additional work is required to reduce the bond resistance at the shoe-element interface.
3. The hot pressed CAS had a wide range of resistances at room temperature.
4. The hot pressed TAGS room temperature resistance was fairly close to that calculated from cast TAGS data.
5. Elevated temperature resistivity of hot pressed TAGS was higher than cast material; Seebeck was similar to cast material.
6. Elevated temperature Seebeck and resistivity of hot pressed CAS was lower than 3M published data.
7. No useful thermal conductivity data was obtained on the apparatus used. Refinements are necessary in test procedures, equipment, and technique.
8. Iron appears to be an effective diffusion barrier between CAS and TAGS.
9. A bond material for joining iron to TAGS was identified. It appears to have adequate strength and low resistance. Further characterization of this bond is desirable.
10. The weakest joint in the segmented CAS-TAGS leg is the iron-CAS interface.
11. Shear and tensile strengths for CAS were 2590 psi and 1100 psi, respectively.
12. Shear and tensile strengths for TAGS were 1397 psi and greater than 1200 psi, respectively.

13. Resistances at the cold and intermediate bonds was less than 5% of the total resistance of segmented legs. The hot bond resistance exceeded 5% and needs further development.
14. Molten CAS was successfully solidified rapidly by dropping the material into liquid nitrogen. Further work is necessary to characterize this material.

TABLE 1
INDIVIDUAL LEG RESISTANCE VALUES

CAS Identification	Measured Resistance Milliohms	Theoretical Resistance Milliohms	Ratio Measured Theoretical	TAGS Identification	Measured Resistance Milliohms	Theoretical Resistance Milliohms	Ratio Measured Theoretical
7324-9	5.2	7.0	0.74	11	3.8	4.5	0.84
7324-10	7.9	7.5	1.1	5	3.8	3.8	1
7324-12	53	9.2	5.8	13	N/A	--	
7324-29	22.5	7.6	3.0	14	3.0	4.0	0.75
GA1-19	25	9.5	2.6	16	6.5	4.3	1.5
GA1-20	45	9.2	4.9	18	4.8	4.0	1.2
GA1-21	32	9.3	3.4	19	5.3	4.1	1.3
GA1-22	44	9.5	4.6	20	6.5	4.1	1.6
GA1-39	28	9.3	3.0	31	7.5	4.5	1.7
GA1-40	22	9.3	2.4	32	5.0	4.5	1.1
GA1-37	23	9.4	2.4	33	6.8	4.5	1.5
7324-14	56	7.8	7.2	28	5.0	4.2	1.2
7324-20	41	7.8	5.3	35	6.1	4.5	1.4
7324-24	33	7.6	4.3	36	6.6	4.5	1.5
7324-27	36	7.7	4.7	27	5.5	4.4	1.3
GA1-35	22	8.4	2.6	39	4.7	4.4	1.1
GA1-36	34	8.5	4.0	40	5.9	4.4	1.3
GA1-38	25	8.5	2.9	41	6.0	4.5	1.3
GA1-32	31	8.5	3.6	43	6.3	4.3	1.5
GA1-33	45	8.5	5.3	44	6.8	4.4	1.5
7324-22	60	8.1	7.4	45	6.5	4.4	1.5

TABLE 2
SEGMENTED LEG RESISTANCE VALUES

Cast No.	CAS Measured Resistance Milliohms	TAGS Measured Resistance Milliohms	Total Leg Measured Resistance Milliohms	Resistance Attributed to Bonds Milliohms	Measured Cold Bond Resistance Milliohms	Measured Intermediate Bond Resistance Milliohms	Measured Hot Bond Resistance Milliohms
1	27	3.7	38	7.3*	.42	5	N/A
2	5.1	4.1	9.8	.6	.03	.3	N/A
3	3.8	3.0	8.9	2.1	.22	1.7	N/A
4	51	3.3	55	.7	.05	1.0	N/A
5	12.5	3.3	20	4.2	.05	3.5	N/A
6	7	5.0	12.	0	.27	3.0	5.3
7	18.5	3.5	27.5	5.5	.14	3.2	5.7
8	21	3.6	23.5	-1.1	.25	1.0	7.0
9	17.5	4.5	28.5	6.5	.19	1.7	9.5
10	25	4.4	36	6.6	.05	.5	5.0
11	17	5	--	--	--	*	6
12	19.5	5	28	3.5	.02	2.5	5
13	85	4	90	1.0	01	1.0	N/A
14	170	5	175	0	01	1.5	N/A
15	45	4.7	54	4.3	05	0.4	N/A
16	150	3.8	155	1.2	.20	1.0	N/A
17	33	4.2	38	.8	.20	1.1	N/A
18	22	4.8	24	-2.8	.10	.35	N/A
19	30	5.4	39	3.6	.20	.43	N/A
20	35	4.5	42	2.5	.12	.50	N/A
21	33	4.5	37	-.5	.04	.77	N/A
22	85	4.9	88	-1.9	.02	.55	N/A

* Leg broke at CAS-Fe interface during resistance test.

TABLE 3
BOND RESISTANCES - SEGMENTED LEGS

<u>Cast No.</u>	<u>Measured Total Leg Resistance Milliohms</u>	<u>Resistance Attributed to Bonds Milliohms</u>	<u>% of Total Resistance</u>	<u>Total Measured Resistance Milliohms</u>	<u>% of Total Resistance</u>	
1	38	7.3	19.2	5.42	14.3*	
2	9.8	.6	6.1	.33	3.4	
3	8.9	2.1	23.6	1.92	21.6	
4	55	.7	1.3	1.05	1.9	
5	20	4.2	21	3.55	17.8	
6	12	0	0	8.57	71.4	
7	27.5	5.5	20	9.04	32.9	
8	23.5	-1.1	-4.7	8.25	35.1	
9	28.5	6.5	22.8	11.39	40.	
10	36.	6.6	18.3	5.55	15.4	
11	--	--	--	6.	--	
12	28	3.5	12.5	7.52	26.9	
13	90	1.0	1.1	1.01	1.1	
14	175	0	0	1.51	.86	
15	54	4.3	8.0	.45	.83	
16	155	1.2	.77	1.2	.77	
17	38	.8	2.1	1.21	3.2	
18	24	-2.8	-11.7	.45	1.9	
19	39	3.6	9.2	.63	1.6	
20	42	2.5	6.0	.62	1.5	
21	37	-.5	-1.4	.81	2.2	
22	88	-1.9	-2.2	.57	.65

 With Top Hat
 Hot Shoe

* Broke at CAS-Fe interface during resistance test.

FIGURE 1. GLOVE BOX

FIGURE 2. HOT PRESS FURNACE

FIGURE 3. HOT PRESS FURNACE

FIGURE 4

FIGURE 5. POWDER PRESSING DIES AND PINS

SEGMENTED LEG ASSEMBLY

STEP 1

STEP 2

HP @ 450°C, 18TSI, 15 Min.

STEP 3

HP @ 400°C, 15TSI, 15 Min.
Stress Relieve 465°C, 60 Min., Argon

STEP 4

Furnace Bond in H₂ at 1050°F, No Dwell at Temperature

FIGURE 6

FIGURE 7

FIGURE 8
SEGMENTED LEGS

FIGURE 9
GRAPHITE MELTING CRUCIBLE FOR CAS

0.125 Dia. x 0.370 Long

FIGURE 10
TENSILE AND SHEAR TESTER

COLLAR AND TEST FIXTURE FOR SEEBECK
OR RESISTIVITY MEASUREMENTS

FIGURE 11

TEST SET-UP FOR SEEBECK OR RESISTIVITY MEASUREMENTS

FIGURE 12

FIGURE 13
SEEBECK COEFFICIENT (α), RESISTIVITY (ρ) AND THERMAL CONDUCTIVITY (k) FOR CAS

FIGURE 14
SEEBECK COEFFICIENT (α), RESISTIVITY (ρ) AND THERMAL CONDUCTIVITY (k) FOR TAGS