ESD-TR-67-38

UNCLASSIFIED

AD 647 295

DEVELOPMENT OF TECHNIQUES FOR PREPARING HOMOGENEOUS SINGLE CRYSTALS OF LEAD TELLURIDE, LEAD SELENIDE, AND LEAD SULFIDE

J.F. Miller, et al

Battelle Memorial Institute
Columbus, Ohio

15 April 1966

Processed for . . .

DEFENSE DOCUMENTATION CENTER
DEFENSE SUPPLY AGENCY

ESLL

CLEARINGHOUSE
FOR FEDERAL SCIENTIFIC AND TECHNICAL INFORMATION

U. S. DEPARTMENT OF COMMERCE / NATIONAL BUREAU OF STANDARDS / INSTITUTE FOR APPLIED TECHNOLOGY

UNCLASSIFIED

Copy 1 of 10 Copies

NINETEENTH QUARTERLY PROGRESS REPORT

on

DEVELOPMENT OF TECHNIQUES FOR PREPARING HOMOGENEOUS SINGLE CRYSTALS OF LEAD TELLURIDE, LEAD SELENIDE, AND LEAD SULFIDE

to

MASSACHUSETTS INSTITUTE
OF TECHNOLOGY

April 15, 1966

by

J. F. Miller, J. W. Moody, and R. C. Himes

Subcontract No. 212 of Prime
Contract No. AF 19(628)-5167

Period Covered
January 15 to April 15, 1966

RECEIVED
JUN 13 1966
DISTRIBUTION

BATTELLE MEMORIAL INSTITUTE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

DISTRIBUTION LIST

Dr. S. E. Blum
Thomas J. Watson Research Center
International Business Machines Corporation
P. O. Box 218
Yorktown Heights, New York

Dr. T. S. Burkhalter
Central Research Laboratories
Texas Instruments Incorporated
P. O. Box 5474
Dallas 9, Texas

Mr. R. T. Leudeman
Daystrum, Inc.
Weston Instruments Division
614 Frelinghuysen Avenue
Newark 12, New Jersey

Elizabeth H. Weeks, Librarian
Research Division
Library
Raytheon Company
28 Seyon Street
Waltham 54, Massachusetts

Dr. L. R. Weisberg
David Sarnoff Research Center
Radio Corporation of America
RCA Laboratories
Princeton, New Jersey

Mr. F. Clarke Abbott
Department of Chemical Engineering
University of Delaware
Newark, Delaware

Mr. K. F. Cuff
Department 52-40
Lockheed Missiles and Space Company
Sunnyvale, California

Elizabeth W. Channel, Librarian
Harry Diamond Laboratories
Washington 25, D. C.

Battelle Memorial Institute · COLUMBUS LABORATORIES

505 KING AVENUE COLUMBUS, OHIO 43201 · AREA CODE 614, TELEPHONE 299-3151 · CABLE ADDRESS: BATMIN

June 9, 1966

Massachusetts Institute of Technology
Lincoln Laboratory
Lexington, Massachusetts 02173

Attention Mr. R. S. Clarke
Distribution Office

Gentlemen:

Enclosed are ten copies of the Nineteenth Quarterly Progress Report on the project "Development of Techniques for Preparing Homogeneous Single Crystals of Lead Telluride, Lead Selenide, and Lead Sulfide", Subcontract No. 212 of Prime Contract No. AF 19(628)-5167. This report covers the period from January 15 to April 15, 1966.

The remaining copies are being distributed to addressees on the approved distribution in accord with the agreement.

Yours very truly,

J. F. Miller

J. F. Miller
Associate Chief
Physical Chemistry Division

JFM:so
Enc. (10)

TABLE OF CONTENTS

Page

INTRODUCTION. 1

SUMMARY 1

EXPERIMENTAL DETAILS AND DISCUSSION. 2

Platinum-Doped PbTe. 2
Low-Temperature Electrical Measurements on Selected PbTe Crystals . 4
Growth of PbTe Crystals From Solution in Pb. 4
PbTe-SnTe Alloy 6
Melt Growth 6
Vapor Growth 6
Heat Treatment 7

FUTURE WORK PLANS 7

LIST OF TABLES

Table 1. Data on Platinum-Doped PbTe. 3

Table 2. Data on Selected PbTe Crystals 5

Table 3. Data on Heat-Treated Specimens of PbTe-SnTe Alloy and SnTe. . . 7

BLANK PAGE

DEVELOPMENT OF TECHNIQUES FOR PREPARING HOMOGENEOUS SINGLE CRYSTALS OF LEAD TELLURIDE, LEAD SELENIDE, AND LEAD SULFIDE

by

J. F. Miller, J. W. Moody, and R. C. Himes

INTRODUCTION

This Nineteenth Quarterly Progress Report on the project covers the period from January 15 to April 15, 1966.

SUMMARY

In this quarterly period, experimental work in progress on the doping of PbTe was concluded and the major thrust of the experimental work was shifted to the preparation of PbTe-SnTe alloy crystals. The results of studies on platinum-doped PbTe indicate that platinum is a donor in PbTe which apparently reacts with lead vacancies to give material with low free-carrier concentrations. Carrier mobilities are high, indicating that the densities of scattering centers in the resulting material are low.

Electron mobilities in excess of 10^6 $cm^2/v\text{-}sec$ at 4.2°K were observed for several PbTe crystals. All of the high-mobility crystals were prepared from PbTe which had been purified by vacuum sublimation or zone melting. It has been shown previously that the purification reduces the concentrations of a number of impurities, and hence it is concluded that the impurities may form scattering centers which affect mobilities in the low-temperature range. The mobility for one crystal at 4.2°K was observed to be 10^7 $cm^2/v\text{-}sec$. Additional, more comprehensive study of this exceptional material is in progress.

Initial experimentation on the growth of PbTe crystals from solution in Pb has yielded only small crystals. However, the compound phase appeared to be continuous and development of the method for ultimate use for the growth of large alloy crystals is planned.

Experimental work on the preparation of SnTe and PbTe-SnTe alloy crystals has included growth from the melt, the heat treatment of melt-grown crystals, and the vapor growth of crystals. The first two procedures have, to date, yielded only high-carrier-concentration, p-type material. Small, high-mobility n-type crystals of alloy, containing about 20 mole percent SnTe, have been prepared by vapor growth, however.

Plans call for continuation of the alloy-crystal preparation, with emphasis in the immediate future on the vapor growth of other compositions.

Accepted for the Air Force
Franklin C. Hudson
Chief, Lincoln Laboratory Office

EXPERIMENTAL DETAILS AND DISCUSSION

Platinum-Doped PbTe

Additional studies of platinum-doped PbTe which were carried out in this quarterly period were concerned with the heat treatment and with room-temperature aging of the crystals. Data on the preparation, treatment, and electrical properties of a series of crystals from a directionally crystallized platinum-doped PbTe ingot are given in Table 1. The single-crystal ingot was Bridgman-grown at 0.05 in./hr from a melt containing about 50.012 atomic percent tellurium, 49.998 atomic percent lead, plus 0.1 atomic percent platinum (Seventeenth Quarterly Progress Report, October 15, 1965). In the table, the series is arranged in progression from the first-to-freeze to the last-to-freeze specimen. In agreement with the tentative initial conclusion regarding platinum in PbTe (Eighteenth Quarterly Progress Report, January 15, 1966), the data indicate that, within experimental error, the concentration of platinum increases with progression along a directionally frozen ingot. As-grown crystals from the first portion of the ingot, which contain the lower concentrations of platinum (85-1 and 85-4), are p-type at room temperature, appear to be compensated, and show the double crossover of the Hall coefficient. In the remaining as-grown n-type specimens, carrier concentrations at 77°K generally increase through the series with increasing platinum concentration, and corresponding resistivities show a general decreasing trend. The carrier concentrations are low, however, and mobilities are high for the carrier concentrations. Platinum is indicated to be a donor in PbTe, which can be utilized to prepare low-carrier-density, high-electron-mobility crystals. Properties of the crystals suggest that platinum, which may react with lead vacancies, is not a normal donor.

Head treatment in lead vapor at 750 and 850°C is seen to increase free-electron concentration appreciably. The carrier concentrations (at 77°K) for the more heavily doped crystals near the tail of the ingot (i.e., 85-5 and 85-3) are near those expected in undoped PbTe heat treated under the same conditions. On the other hand, carrier concentrations remain low in less heavily doped crystals such as 21321-85-2 and 18761-98 (described in Seventeenth Quarterly Progress Report, p 2). Carrier concentrations in the latter crystals were increased by the heat treatments, but not to "normal" levels. One possibility is a slow, and hence incomplete, approach to the equilibrium state for this type of PbTe.

Room-temperature "aging" of platinum-doped PbTe has been found to produce changes in the electrical properties. Recheck of properties of platinum-doped crystals prepared several years ago (Seventeenth Quarterly Progress Report, April, 1963) showed that the carrier mobility ($R\sigma$) values had decreased by factors of 2 to 10, primarily owing to increase of resistivity. In the crystals currently being studied, effects on resistivity and carrier mobility vary, but carrier concentration appears to be increased by the aging. Similar aging phenomena also have been observed previously in undoped PbTe (Fifth Quarterly Progress Report, October 15, 1962) and were attributed to the precipitation of a phase which exerts varied effects on carrier concentration and mobility, depending on the concentration of the phase and its identity and nature.

TABLE 1. DATA ON PLATINUM-DOPED PbTe

				Observed Electrical Properties				
Specimen	Description	Concentration of Platinum[(a)], percent by weight	Date Measured	Temperature, °K	Resistivity (ρ), ohm-cm	Hall Coefficient (R), cm^3/coulomb	Ro, cm^2/v-sec	Carrier Concentration (n), cm^{-3}
21321-85-1	As grown	0.16 ± 0.03	Feb., 1966	300	8.51×10^{-2}	+6.76	79	9.3×10^{17}
				81	3.97×10^{-1}	-561	1,400	1.1×10^{16}
				4.2	9.37×10^{-2}	-679	7,250	9.3×10^{15}
21321-85-4	As grown	0.19 ± 0.04	Jan., 1965	300	5.12×10^{-2}	+7.2	140	8.8×10^{17}
				77	2.2×10^{-1}	-664	3,000	9.5×10^{15}
21321-85-2	As grown	0.28 ± 0.06	Oct., 1965	300	6.03×10^{-2}	-76.4	1,270	8.2×10^{16}
				77	2.52×10^{-3}	-96.4	38,200	6.5×10^{16}
	After heat treatment at 850°C for 48 hr in vapor of Pb-rich Pb-Te stock		Jan., 1966	300	3.60×10^{-2}	-7.2	200	8.8×10^{17}
				77	5.66×10^{-3}	-13.7	2,400	4.6×10^{17}
	Above specimen remeasured		May, 1966	300	3.92×10^{-2}	-5.6	140	1.1×10^{18}
				77	1.06×10^{-3}	-10.5	9,900	6.0×10^{17}
21321-85-2a	As grown (from same portion of ingot as -2)		Mar., 1966	300	1.83×10^{-1}	-136	890	4.6×10^{16}
				77	1.24×10^{-2}	-244	19,700	2.6×10^{16}
				4.2	2.50×10^{-4}	--	976,000[(b)]	--
21321-85-5	As grown	0.30 ± 0.06	Jan., 1966	300	5.30×10^{-2}	-62	1,170	1.0×10^{17}
				77	2.75×10^{-3}	-84.5	30,700	7.4×10^{16}
	After heat treatment at 750°C for 48 hr in vapor of Pb-rich Pb-Te stock		Jan., 1966	300	3.22×10^{-3}	-5.05	1,570	1.2×10^{18}
				77	1.55×10^{-4}	-4.81	31,000	1.3×10^{18}
	Above specimen remeasured		May, 1966	300	3.20×10^{-3}	-3.9	1,200	1.6×10^{18}
				77	1.47×10^{-4}	-3.76	25,600	1.7×10^{18}
21321-85-3	As grown	0.20 ± 0.04	Jan., 1966	300	5.17×10^{-2}	-65.3	1,260	9.6×10^{16}
				77	3.15×10^{-3}	-77.9	24,700	8.1×10^{16}
	After heat treatment at 850°C for 48 hr in vapor of Pb-rich Pb-Te stock		Jan., 1966	300	3.93×10^{-3}	-4.8	1,200	1.3×10^{18}
				77	1.70×10^{-4}	-5.2	30,500	1.2×10^{18}
	Above specimen remeasured		May, 1966	300	3.90×10^{-3}	-4.1	1,050	1.5×10^{18}
				77	2.42×10^{-4}	-4.9	20,200	1.1×10^{18}

(a) By semiquantitative emission spectrographic analysis. Values appear generally too high. Indication as do distribution profile should be valid, however.
(b) Calculated utilizing 77°K Hall coefficient.

Low-Temperature Electrical Measurements on Selected PbTe Crystals

The results of electrical-property measurements on selected PbTe specimens, which are given in Table 2, indicate that the mercury-doped material (Specimen 19733-9A) has no unusual properties. In previous investigation of the heat treatment of the mercury-doped PbTe (Eighteenth Quarterly Progress Report, January 15, 1966), results likewise indicated that the mercury-doped material was similar to undoped PbTe.

The remainder of the specimen for which data are presented in Table 2 are of PbTe which has been zone refined or sublimed. The helium-temperature electron mobility ($R\sigma$) for the zone-refined PbTe specimen (18761-93) is higher than those observed for PbTe previously prepared on this program. The vacuum-sublimed crystals are the only single crystals prepared in this work for which $R\sigma$ values greater than 1×10^6 cm^2/v-sec have been observed. The results are not uniformly good, however; it can be noted that $R\sigma$ values for some specimens that have been sublimed (21321-28 and -66) are low. This may be attributable to (1) the pickup of copper from the fused-silica ampoules, or (2) erratic shifts in composition (i. e., slight shifts with respect to stoichiometry) resulting from inadequate control of the sublimation process. Such shifts in composition could result, for example, from slight variations in temperatures and temperature gradients – and hence in vapor composition and deposition rate. From spectrographic analysis of Specimen 21321-66, it is estimated that 0.2 ppm of copper is present in this material. On the other hand, it has been shown that the concentrations of a number of both metallic and nonmetallic impurities in the PbTe are reduced by vacuum sublimation.* In view of the generally high helium-temperature mobilities (i. e., $R\sigma$ values) it is suggested that the purification processes reduce the concentration of ionized-impurity scattering centers.

The extremely high $R\sigma$ value for Crystal 19733-87 is believed to be the highest reported for PbTe since it exceeds values reported by Kanai, et al.** The value, which was obtained from measurement of both resistivity and Hall effect at 4.2°K, falls very near the $T^{-5/2}$ curve of $R\sigma$ versus temperature for PbTe. The Hall specimen on which these measurements were made is large (~2.0 x 0.63 x 0.45 cm) and additional sizable (centimeter dimensions) portions of the crystal are available. More comprehensive study of the crystal, including electrical-property measurements, X-ray diffraction, and metallographic examination, is in progress. Precision X-ray lattice-constant determinations on specimens from the first to deposit and the last to deposit of the vapor-grown crystal gave values of 6.4605 and 6.4614 ± 0.0005 A, respectively. Although the values rigorously must be considered the same (i. e., within experimental error), the slight difference may not be entirely insignificant. The electrical-property studies are in progress, and it is anticipated that results of studies of this unusual crystal can be presented in the next progress report.

Growth of PbTe Crystals From Solution in Pb

The direct growth of n-type, Pb-saturated crystals from Pb-Te melts should occur at low temperature (e. g., in the range just above 330°C) in melts containing low

---

*Twelfth, Thirteenth, and Sixteenth Quarterly Progress Reports, September 9 and December 31, 1964, and July 15, 1965, respectively.

**Kanai, Y., Nii, R., and Watanabe, N., J. Appl. Phys., 325, 2146 (1961).

TABLE 2. DATA ON SELECTED PbTe CRYSTALS

			Observed Electrical Properties				
Specimen	Description	Date	Temperature, °K	Resistivity (ρ), ohm-cm	Hall Coefficient (R), cm^3/coulomb	Rσ, cm^2/v-sec	Carrier Concentration (n), cm^{-3}
19733-9A	Hg-doped (0.35 at.%); heat treated in vapor of Pb-rich PbTe at 855°C - 2 hr, 400°C - 72 hr, 300°C - 240 hr	Mar., 1966	300	3.80×10^{-2}	-41.6	1,100	1.5×10^{17}
			81	2.06×10^{-3}	-47.6	23,100	1.3×10^{17}
			4.2	7.22×10^{-5}	-52.3	724,000	1.2×10^{17}
18761-93	Zone refined; heat treated in vapor of Pb-rich PbTe at 855°C for 48 hr, then in vacuum 400°C - 6 days, 300°C - 10 days, 200°C - 21 days	--	300	3.42×10^{-2}	-43.6	1,280	1.4×10^{17}
			77	1.61×10^{-3}	-51.5	32,000	1.2×10^{17}
			4.2	5.84×10^{-5}	--	880,000(a)	--
19733-21	Heat treated in vapor of Pb-rich Pb-Te stock at 835°C - 5 days, 700°C - 2 days, 600°C - 2 days, 500°C - 4 days, 400°C - 6 days, 300°C - 10 days	--	300	5.43×10^{-3}	-7.38	1,360	8.5×10^{17}
			77	2.54×10^{-4}	-7.83	30,800	8.0×10^{17}
			4.2	5.41×10^{-6}	--	1,400,000(a)	--
19733-76-1A	Sublimed twice in vacuum at 715°C; crystal then grown by sublimation in sealed ampoule at ~700°C	Mar., 1966	300	2.62×10^{-3}	-3.89	1,480	1.6×10^{18}
			81	1.38×10^{-4}	-4.10	29,700	1.5×10^{18}
			4.2	2.12×10^{-6}	-3.96	1,870,000	1.6×10^{18}
21321-28	Sublimed twice in vacuum at 800°C; crystal then grown by sublimation in sealed ampoule at ~780°C	Oct., 1964	300	6.30×10^{-3}	-9.96	1,600	6.3×10^{17}
			77	3.25×10^{-4}	-10.6	33,000	5.9×10^{17}
		Mar., 1966	300	6.59×10^{-3}	-3.60	550	1.7×10^{18}
			81	3.86×10^{-4}	-5.32	13,800	1.2×10^{18}
			4.2	3.22×10^{-5}	-3.90	121,000	1.6×10^{18}
21321-52A	Sublimed 3 times in vacuum at 800°C, then heat treated in vapor of Pb-rich Pb-Te stock at 822°C for 48 hr	July, 1965	300	2.49×10^{-3}	-3.91	1,570	1.6×10^{18}
			77	1.18×10^{-4}	-3.25	27,500	1.9×10^{18}
		Mar., 1966	300	2.08×10^{-3}	-3.30	1,590	1.9×10^{18}
			81	1.04×10^{-4}	-2.94	28,300	2.1×10^{18}
			4.2	3.26×10^{-6}	-3.04	1,180,000	2.1×10^{18}
21321-66	Sublimed 4 times in vacuum at 800°C, then heat treated in vapor of Pb-rich Pb-Te stock at 822°C for 48 hr	July, 1965	300	2.30×10^{-3}	-2.91	1,260	2.2×10^{18}
			77	1.12×10^{-4}	-2.93	26,200	2.2×10^{18}
		Mar., 1966	300	2.54×10^{-3}	-3.21	1,260	2.0×10^{18}
			81	1.45×10^{-4}	-2.99	20,600	2.1×10^{18}
			4.2	1.13×10^{-5}	-3.13	277,000	2.0×10^{18}
19733-87	Sublimed twice in vacuum at 850°C; crystal then grown by sublimation in sealed ampoule at 850°C	--	300	4.73×10^{-3}	-5.37	1,140	1.2×10^{18}
			77	2.36×10^{-4}	-7.91	33,500	7.9×10^{17}
			4.2	2.18×10^{-7}	-2.11	10,000,000	3×10^{18}

(a) Calculated utilizing Hall coefficient at 77°K.

concentrations of Te. Since the temperatures involved are low, very low rates of crystal growth can be employed. Because of the low growth temperatures, properties of the crystals are likely to be different from those of crystals grown by high-temperature methods. Development of the method may be desirable, not only for the preparation of PbTe crystals, but for the growth of PbTe-SnTe alloy and other compounds as well.

In the first experiment on the growth of PbTe, the solution was made up to contain about 3 weight percent Te in Pb and was suspended in a temperature gradient (about 8°C/in.) in a tube furnace. After the solution was homogenized at 950°C, the median temperature was lowered to 650°C and then was decreased from 650°C to 325°C at a uniform average rate of about 0.4°C/hr over a 30-day period. A shell of PbTe was formed on the outer surface of the solution adjacent to the graphite container wall at the low-temperature end of the charge. However, crystallites in the shell were small and the shell was thin; hence, no specimen suitable for evaluation was obtained. In a second experiment, which is now in progress, the concentration of Te was increased to 10 weight percent, and the temperature is being decreased uniformly in the range 750 to 400°C.

PbTe-SnTe Alloy

Melt Growth

Initial experimental work on the PbTe-SnTe alloy has been concerned with crystal growth from the melt, with crystal growth by vapor deposition, and with the use of heat treatment for the adjustment of the composition of melt-grown crystals.

Crystals of SnTe and ingots of PbTe-SnTe alloy were grown from metal-rich melts by the Bridgman method at drop rates of 1.3, 3.2, 5.0 cm/hr. The 50-50 and the 33-66 ratios were selected as initial compositions of the PbTe-SnTe alloy melts. (These liquidus compositions correspond to solidus compositions near 90-10 and 77-23 PbTe-SnTe, respectively.*)

Large crystals of centimeter dimensions were present in the SnTe ingots. Crystals were much smaller in the alloy ingots, however, being of only millimeter cross-sectional dimensions.

All of the crystals grown from the melt, both of SnTe and of the alloy, were p-type with low electrical resistivity, as expected. These melt-grown crystals were employed in heat-treatment experiments.

Vapor Growth

A number of vapor-growth experiments were conducted utilizing as source material crushed, metal-rich, 80-20 PbTe-SnTe alloy that had been homogenized in the molten state and quenched. The crystal growth was conducted in sealed fused-silica ampoules at source temperatures in the range 660 to 800°C with temperature gradients of 1 to 2.5°C/cm and with total temperature differentials along the ampoules of about 10 to 20°C.

---

*Abrikosov, N. Kh., Dvul'dina, K. A., and Danilyan, T. A., Journal of Inorganic Chemistry, USSR, III, 214 (1958) - cover-to-cover translation.

In initial experiments, transport of the alloy was found to be too rapid at 795°C, with a 10°C temperature difference (~1°C/cm) in evacuated ampoules. In subsequent experiments, residual hydrogen atmospheres (to about 250 mm pressure at 300°K) were used and growth rates were lower.

In these growth experiments small (millimeter dimensions), well-formed crystals were obtained, but the deposits were of p-type or mixed p- and n-type crystals. However, the results of measurements on one of the small (0.12 x 0.71 x ~2.2 mm) n-type crystals grown at 750°C indicate that alloy crystals with interesting electrical properties can be prepared by the vapor-growth technique.

Specimen	Temperature	Resistivity (ρ), ohm-cm	Hall Coefficient, (R), cm^3/coulomb	Rσ, cm^2/v-sec	Carrier Concentration (n), cm^{-3}
23046-14	300	1.98×10^{-2}	-19.4	980	3.2×10^{17}
	77	1.07×10^{-3}	-35.9	33,600	1.8×10^{17}

The results of semiquantitative X-ray fluorescence analysis indicate that this crystal contains 28 mole percent SnTe. From the lattice constant, determined by X-ray diffraction, it was calculated that the crystal contained 17 mole percent SnTe, assuming Vegaard's law is applicable to the system. Although the agreement between the two methods is not good, the results indicate that the alloy crystals contain an appreciable concentration of SnTe. Standards are being prepared to provide for quantitative X-ray fluorescence analysis of the PbTe-SnTe alloy in the future.

Heat Treatment

A number of SnTe and PbTe-SnTe alloy crystals were heat treated in the vapor of Sn, Pb, Zn, Cd, or metal-rich PbTe-SnTe alloy at temperatures in the range 450 to 800°C for periods of 2 to 10 days. Following these "high-temperature" heat treatments, many of the specimens were then heat treated at 400 and 300°C for 3 to 7 days (at each temperature). It was expected that Zn, Cd, Sn, and/or Pb might diffuse into the crystals at the heat-treatment temperatures and fill metal-site vacancies.

All of the SnTe and PbTe-SnTe alloy crystals remained p-type. However, several of the alloy crystals were found to have measurable Hall coefficients following the heat treatment. As can be seen from the data in Table 3, although the Hall coefficient are still low, Rσ values range up to 6,000 cm^2/v-sec at 77°K, with maximum values for specimens heat treated at 650°C. In the series of SnTe specimens, the resistivity (at 77°K) appears to increase as heat-treatment temperature is decreased in the range 750 to 550°C. Both of these trends will be checked in future work.

FUTURE WORK PLANS

Plans for the next quarterly period call for:

(1) Some additional study of the high-mobility PbTe

TABLE 3. DATA ON HEAT-TREATED SPECIMENS OF PbTe-SnTe ALLOY AND SnTe

Specimen	Material	Vapor Producing Phase	Heat-Treatment Conditions	Temperature, °K	Observed Electrical Properties: Resistivity (ρ), ohm-cm	Observed Electrical Properties: Hall Coefficient (R), cm^3/coulomb	Observed Electrical Properties: R_σ, cm^2/v-sec
23046-39	Middle portion of 50-50 PbTe-SnTe ingot	Zn; 0.005X specimen wt	7 days at 750°C	77	2.0×10^{-4}	+0.28	1400
23046-36	Ditto	Ditto	7 days at 650°C	77	1.38×10^{-4}	+0.66	4800
23046-36	"	"	Additional 7 days each at 400°, and 300°C	77	2.0×10^{-4}	+1.2	6000
23046-33	"	"	7 days at 550°C	77	1.2×10^{-4}	+0.46	3800
23046-40	"	Cd; 0.01X specimen wt	7 days at 750°C	77	3.94×10^{-4}	+0.34	860
23046-37	"	Ditto	7 days at 650°C	77	2.71×10^{-4}	+0.76	2800
23046-52	First portion of 50-50 PbTe-SnTe ingot	Metal-rich 80-20 PbTe-SnTe stock; 4X specimen wt	2 days at 860°C, 3 days at 400°C	77	3.27×10^{-4}	+0.32	1000
23046-25	SnTe	Sn; 0.01X specimen wt	7 days at 750°C	77	(very low)	--	--
23046-26	SnTe		7 days at 650°C	77	2.62×10^{-5}	--	--
23046-27	SnTe		7 days at 550°C	77	5.7×10^{-6}	--	--

(2) Continuation of work on the vapor-growth of PbTe-SnTe alloy crystals, specifically aimed at obtaining larger crystals and crystals of several compositions.

(3) Continuation of the study of the heat treatment of SnTe and PbTe-SnTe alloy crystals

(4) Continued work on the development of the solution method for the growth of crystals.

The data upon which this report is based are recorded in Battelle Laboratory Record Book No. 23046, pp 4-70.

JFM/JWM/RCH:so

DOCUMENT CONTROL DATA – R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Battelle Memorial Institute under Subcontract No. 212 to Lincoln Laboratory, M.I.T.	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP None

3. REPORT TITLE
Development of Techniques for Preparing Homogeneous Single Crystals of Lead Telluride, Lead Selenide, and Lead Sulfide

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)
Progress Report – 16 January to 15 April 1966

5. AUTHOR(S) (Last name, first name, initial)
Miller, J. P. Moody, J. W. Himes, R. C.

6. REPORT DATE 15 April 1966	7a. TOTAL NO. OF PAGES 20	7b. NO. OF REFS 3
8a. CONTRACT OR GRANT NO. AF 19(628)-5167 b. PROJECT NO. 649L c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) 19th Quarterly Progress Report 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) ESD-TR-67-38	

10. AVAILABILITY/LIMITATION NOTICES
Distribution of this document is unlimited.

11. SUPPLEMENTARY NOTES None	12. SPONSORING MILITARY ACTIVITY Air Force Systems Command, USAF

13. ABSTRACT

Experimental work on the doping of PbTe was concluded and the major thrust of work was shifted to the preparation of PbTe-SnTe alloy crystals. The results of studies on platinum-doped PbTe indicate that platinum is a donor in PbTe which apparently reacts with lead vacancies to give material with low free-carrier concentrations. Carrier mobilities are high, indicating that the densities of scattering centers in the resulting material are low.

Initial experimentation on the growth of PbTe crystals from solution in Pb has yielded only small crystals. However, the compound phase appeared to be continuous and development of the method for ultimate use for the growth of large alloy crystals is planned.

Experimental work on the preparation of SnTe and PbTe-SnTe alloy crystals has included growth from the melt, the heat treatment of melt-grown crystals, and the vapor growth of crystals. The first two procedures have, to date, yielded only high-carrier-concentration, p-type material. Small, high-mobility n-type crystals of alloy, containing about 20 mole percent SnTe, have been prepared by vapor growth, however.

14. KEY WORDS

crystal growth single crystals lead telluride lead selenide lead sulfide