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Memorandum of Project MICHIGAN

PHASE DIAGRAM FOR THE BINARY SYSTEM
CdTe-In₂Te₃

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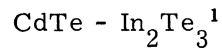
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Phase Diagram for the Binary System



ABSTRACT

The phase diagram for the binary system CdTe-In₂Te₃ has been obtained by correlating information from differential thermal analysis measurements, microscopic studies, diffusion couples, and X-ray powder patterns.

In establishing the terminal points for this diagram, the melting point for In₂Te₃ was found to be $667 \pm 1^\circ\text{C}$. For CdTe, a melting point of $1098 \pm 3^\circ\text{C}$ was indicated.

In going across the diagram, three peritectics are apparent. The first (β) is at 785°C and 50 mol % In₂Te₃; the second (γ) is at about 702°C and 74 mol % In₂Te₃; and the third (δ') is at 695°C and about 83 mol % In₂Te₃. There is a large retrograde solubility of CdIn₂Te₄ in CdTe. The δ -phase transforms to an ϵ -phase at 625°C and 100% In₂Te₃. The addition of CdTe stabilizes the δ -phase, and it finally disappears to $\gamma + \epsilon$ in a eutectoid reaction at about 360°C and 83 mol % In₂Te₃.

Some of the properties of the phases have been measured and are presented. There appear to be qualitatively significant monotonic relationships among the energy gap, the transition temperatures, and the compositions.

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Dr. David F. Edwards made the infrared measurements at Willow Run Laboratories. The authors would like to acknowledge the aid of many students who helped with the individual determinations.

INTRODUCTION

Since mixed crystals of binary, isomorphous, semiconductor compounds are formed from isoelectronic elements, the electrical properties appear to be monotonic functions between limits defined by the properties of the constituents. In some cases slight maxima or minima are observed, but these systems are exceptional.

Since new ternary phases are formed by combining nonisomorphous binaries with non-isoelectronic elements, this functional variation of the electrical properties is not known. An additional degree of freedom should be present by virtue of the presence of the third element. With this freedom, it should be possible ultimately to improve the performance of many semiconductor devices, including those that are used as sensors in military electronic systems. A valid experimental determination of this functional relationship requires the preparation of pure, homogeneous test samples as a first step. Such samples cannot be prepared intelligently and reproducibly without a knowledge of the phase diagram of the system from which the ternary compounds are formed.

This work was undertaken to ascertain the phase diagram of the system CdTe-In₂Te₃ which produced a typical chalcopyritelike ternary compound, CdIn₂Te₄. It was obtained by correlating information from differential thermal analysis measurements, microscopic studies, X-ray powder patterns, and diffusion couples. Preliminary measurements on the physical properties of the five phases found in the system are also reported. This system is of interest since all compounds formed by mixing CdTe and In₂Te₃ are semiconductors.

In establishing the terminal points for this diagram, the melting point for In₂Te₃ was found to be $667 \pm 1^\circ\text{C}$. For CdTe, a melting point of $1098 \pm 3^\circ\text{C}$ was indicated. In going across the diagram, three peritectics are apparent. At 785°C , the compound CdIn₂Te₄ is formed. This material has a tetragonal chalcopyritelike structure. At about 702°C and 74 mol % In₂Te₃, a second peritectic forms which has a structure somewhat similar to the first peritectic. At 695°C and about 83 mol % In₂Te₃, a third peritectic solidification creates a material which is isomorphous with In₂Te₃ in its zinc blende form. A large retrograde solubility of CdIn₂Te₄ in CdTe is apparent from the structures in the photomicrographs taken in the intermediate two-phase region. The δ -phase transforms to an ϵ -phase at 625°C and 100 % In₂Te₃. The addition of CdTe stabilizes the δ -phase and it finally disappears to $\gamma + \epsilon$ in a eutectoid reaction at about 83 % In₂Te₃ between 350°C and 375°C . Some of the properties of these new phases have been measured. There appear to be qualitatively significant monotonic relationships

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among the energy gap, the transition temperatures, and the compositions.

The experimental work can be conveniently classified into five categories: the preparation of the compounds; the determinations of the solid-liquid equilibria; the determination of the structural properties of the various components in the system; the determination of the solid-solid equilibria; and the determination of the semiconducting properties of the various components in the system.

2

SAMPLE PREPARATION

All compounds used in the study were made by direct fusion from commercially available high purity elements. Indium of 99.999+% purity was obtained from the Indium Corporation of America. Cadmium of 99.99+% purity and tellurium of 99.999+% purity were obtained from the American Smelting and Refining Company. Stoichiometric amounts of the elements were weighed into clear fused silica ampoules, evacuated to a pressure below 10^{-4} mm Hg, and sealed. The fusions were carried out over a period of several hours in such a way that the heat of reaction was evolved gradually, so as not to break the ampoules by any sudden increases in the vapor pressures. The content of each ampoule was homogenized by maintaining it above its liquidus temperature in an agitating furnace. After fusion each sample was removed from its original container, crushed, and transferred to a new fused silica tube containing a deep thermocouple well concentric with the tube axis. Each sample was annealed for at least 24 hours below the lowest transition temperature before the DTA (differential thermal analysis) measurements.

3

SOLID-LIQUID EQUILIBRIUM

The solid-liquid equilibrium lines have been determined primarily from DTA measurements. In the following section the experimental apparatus and procedures are described first, and the experimental results next. The available information from the literature will be reported and combined with our results to synthesize a phase diagram for the system $\text{CdTe-In}_2\text{Te}_3$. The liquidus line is examined thermodynamically.

3.1. DIFFERENTIAL THERMAL ANALYSIS APPARATUS AND PROCEDURES

A schematic diagram of the DTA apparatus is shown in Figure 1. The power input to the

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DTA furnace is controlled by means of an automatically programmed motor-driven autotransformer. The furnace is purged with dry nitrogen in order to prevent corrosion of the nickel sample holder, which is provided with three sample wells spaced at 120° intervals. Two specimens may be measured at one time, the third well being occupied by the reference material. Each sample temperature is measured by means of a chromel-alumel thermocouple, the output of which is measured on a Leeds and Northrup precision potentiometer. The output of the differential thermocouple (Figure 2) is amplified and recorded with a Leeds and Northrup Speedomax recorder. The thermocouples were calibrated against standards of indium (156°C), lead (327°C), and silver (961°C) and found to agree within 1°C.

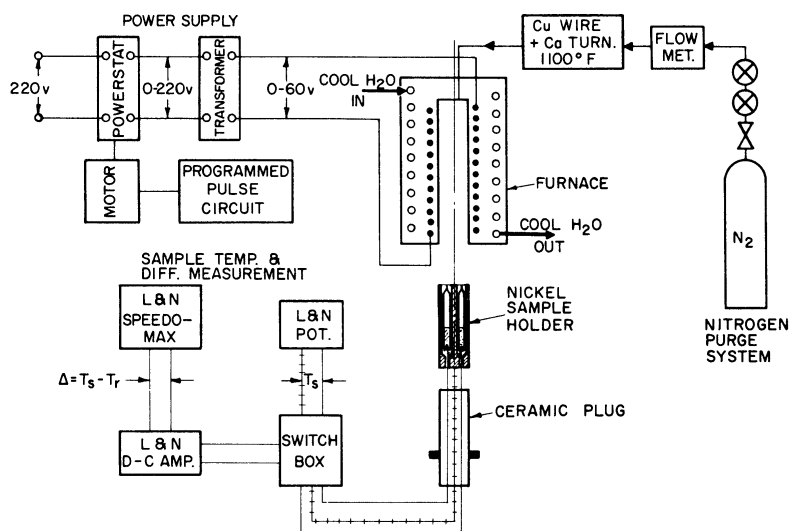


FIGURE 1. SCHEMATIC DIAGRAM OF DIFFERENTIAL THERMAL ANALYSIS EQUIPMENT

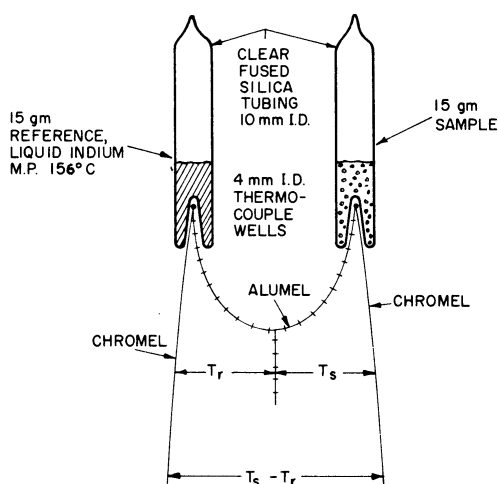


FIGURE 2. DIAGRAM OF DIFFERENTIAL THERMOCOUPLES AND SAMPLE TUBES

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The samples were heated and cooled inside the nickel block with a liquid indium standard, at a rate of $2.5^{\circ}\text{C}/\text{minute}$ from room temperature to a maximum temperature well above that of the highest transition, and back to room temperature. Although the DTA sample tubes sometimes cracked at low temperatures as the sample contracted around the thermocouple well, they were not wet significantly by the sample and no apparent oxidation occurred. A continuous record was taken as a function of time for both the sample temperature and the differential emf, and the data were replotted to show the differential emf as a function of sample temperature.

Each sample was subsequently reannealed and cooled before photomicrographs were taken and the X-ray determinations made. In some instances, X-ray samples were removed prior to the DTA runs.

3.2. EXPERIMENTAL RESULTS FROM DIFFERENTIAL THERMAL ANALYSIS

More than 50 samples covering the composition range from pure CdTe to pure In_2Te_3 were prepared in this system, and representative results from differential thermal analysis measurements are shown in Figure 3. Differential emf is plotted against temperature for several different compositions.

In the interpretation of the experimental DTA curves, the liquidus temperature on the heating curves was chosen as the point where the differential emf completed its last deviation. On the cooling cycle, considerable supercooling sometimes occurred, as indicated by a large initial differential emf which sometimes indicated an increase in the sample temperature. This effect was particularly noticeable in the high cadmium region, as is apparent in Figure 3. The liquidus temperature was taken as the point where the deviation in the differential emf was initiated, or attained its maximum value. The agreement between the two liquidus temperatures derived in this way was not always good. This ambiguity in interpreting the DTA results was particularly large over the range from 0 to 63 mol % In_2Te_3 . The peritectic transformations agreed well on both heating and cooling. The solidus lines were obtained from the heating curves by choosing the point where the first deviation in the differential emf was initiated. The data are tabulated in Table I.

In checking the terminal points for this diagram, the melting point for CdTe was found to be $1098 \pm 3^{\circ}\text{C}$ (Reference 1), which represents a considerable deviation from the widely reported value of 1045°C (Reference 2). The variation can be attributed to the differences in purity of the tellurium which was available to Kobayashi in 1910 (mp 437°C), and that which is available today (mp 454°C), as well as to differences in experimental techniques. Lawson,

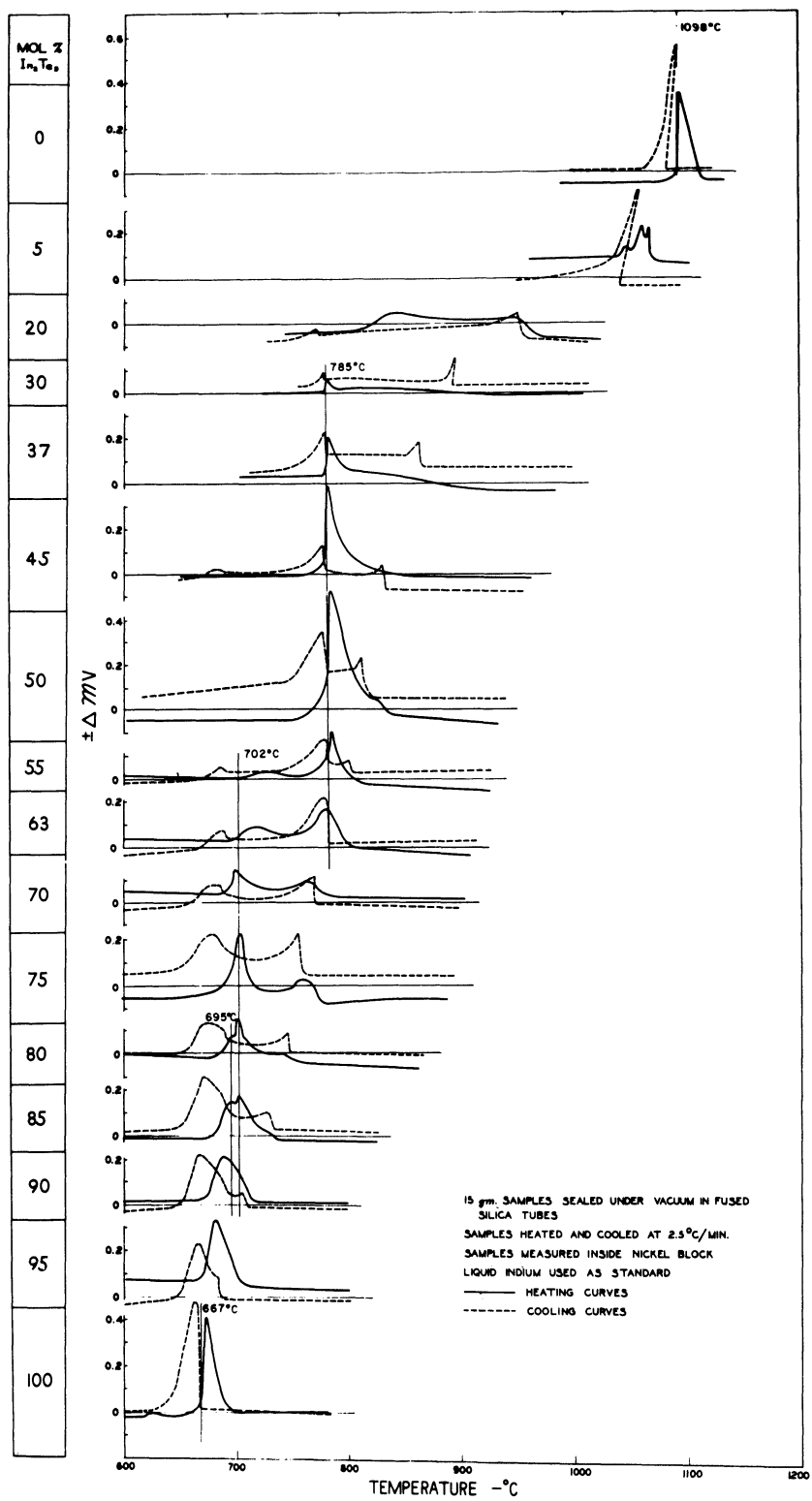


FIGURE 3. DIFFERENTIAL THERMAL ANALYSIS CURVES FOR CdTe-In₂Te₃ PHASE-DIAGRAM STUDY

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Nielsen, Putley, and Young (Reference 3) have recently reported the melting point of CdTe as 1106°C, and deNobel has reported a melting point of 1090°C (Reference 4).

TABLE I. SUMMARY OF DTA RESULTS FOR CdTe-In₂Te₃ SYSTEM

Sample Code No.	In ₂ Te ₃ (mol %)	Heating Liquidus (°C)	Cooling Liquidus (°C)	Solidus (°C)	Peritectic Temperature		
					1st (°C)	2nd (°C)	3rd (°C)
354	0.0	1098	1098	1098	-	-	-
333	5.0	1075	1065	1045	-	-	-
324	12.5	1030	-	955	-	-	-
332	20.0	975	960	840	-	-	-
319	25.0	955	930	805	-	-	-
346	30.0	930	900	-	784	-	-
355	33.3	925	885	-	784	-	-
345	37.0	910	870	-	785	-	-
309	40.0	885	865	-	785	-	-
344	45.0	860	840	-	784	-	-
307	50.0	840	825	-	783	-	-
352	55.0	820	805	-	784	725	-
308	60.0	805	790	-	785	720	-
349	63.0	805	783	-	-	715	-
310	66.7	800	775	-	-	715	-
350	70.0	785	775	-	-	700	-
371	75.0	770	765	-	-	704	696
367	77.5	760	755	-	-	702	696
351	80.0	755	750	-	-	702	695
368	82.5	745	740	-	-	700	696
356	85.0	740	735	-	-	704	695
366	87.5	725	725	692	-	702	-
357	90.0	715	711	690	-	702	-
369	92.5	710	705	684	-	-	695
348	95.0	702	690	680	-	-	-
370	97.5	690	685	-	-	-	-
303	100.0	667	667	667	-	-	-

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Other important transition points, at 785°C, 702°C, 695°C, and 667°C, can also be seen in Figure 3. Note also the transition in the pure In_2Te_3 at about 625°C. Hahn (Reference 5), Inuzuka and Sugaiki (Reference 6), and Woolley, Pamplin, and Holmes (Reference 7) have characterized a structure of In_2Te_3 which forms from the zinc blende structure after long annealing, which would indicate the presence of a transition somewhere below the melting point. Gasson, Holmes, Parrott, and Penn (Reference 8) have reported a transition at $615 \pm 5^\circ\text{C}$. Hahn et al. (Reference 9) have also investigated the crystal structures of the compounds formed in this system, and reported the existence of the CdIn_2Te_4 phase.

3.3. THE PHASE DIAGRAM

With this data, supplemented by diffusion couples, photomicrographs, and X-ray studies on quenched samples, it is possible to hypothesize the broad outline of the phase diagram shown in Figure 4. Both the heating and cooling liquidus points are plotted, as discussed above. This diagram shows that cadmium telluride ($\text{CdTe} = \alpha$) and indium telluride ($\text{In}_2\text{Te}_3 = \delta$) are congruently melting. In addition, there are three peritectic points, a eutectoid, and a

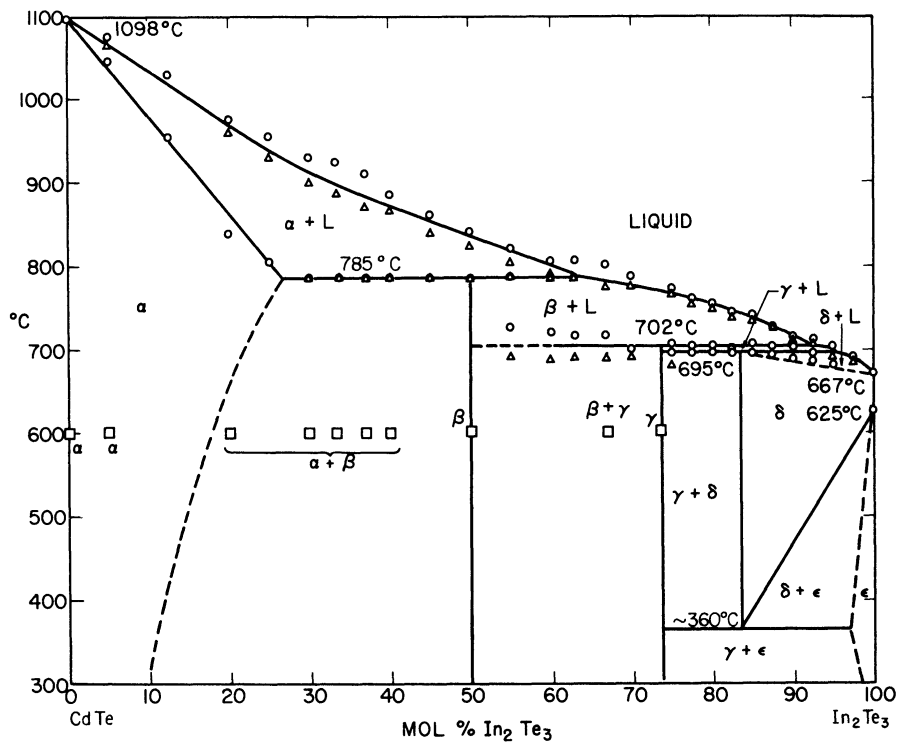


FIGURE 4. PHASE DIAGRAM FOR $\text{CdTe}-\text{In}_2\text{Te}_3$ BINARY SYSTEM. \circ From DTA on heating; \triangle from DTA on cooling; \square from photomicrographs.

transformation in the In_2Te_3 structure, from δ to ϵ . The peritectics occur at about 50 mol % In_2Te_3 ($\text{CdIn}_2\text{Te}_4 = \beta$) and 785°C , at about 74 mol % In_2Te_3 ($\text{CdIn}_6\text{Te}_{10}$ or $\text{Cd}_7\text{In}_{38}\text{Te}_{64} = \gamma$) and 702°C , and at about 83 mol % In_2Te_3 ($\text{CdIn}_{10}\text{Te}_{16} = \delta'$) and 695°C . No congruently melting compounds form along this particular line in the ternary system cadmium-indium-tellurium. The δ -phase is stabilized by adding CdTe , but it finally disappears to $\gamma + \epsilon$ in an eutectoid reaction at about 83 mol % In_2Te_3 between 350°C and 375°C .

3.4. THERMODYNAMIC CHARACTERIZATION OF THE LIQUIDUS LINE

The liquidus line comprises four separate and distinct segments: (a) from 0 to 63 mol % In_2Te_3 ; (b) from 63 to 93 mol % In_2Te_3 ; (c) from 93 to 95 mol % In_2Te_3 ; and (d) from 95 to 100 mol % In_2Te_3 . Prigogine and Defay (Reference 10) show that in general the liquidus curve is actually a composite of several separate relationships. In this particular case, four separate relationships would be required.

The regions (c) and (d) are too short to yield to an intelligible thermodynamic analysis with the data available. The large solid solubility in region (a) makes thermodynamic analysis difficult since the solidus curve is not precisely defined. However, by assuming negligible solid solubility in the β phase, the region (b) can be approximated by an ideal solution through the Schroeder-vanLaar equation

$$\ln x_\beta = \frac{\Delta H}{R} \left(\frac{1}{T_f^*} - \frac{1}{T} \right) = 19,400 \left(\frac{1}{1070} - \frac{1}{T} \right) \quad (1)$$

where x_β = mol fraction β phase in δ phase = $2(1 - y)$

y = mol fraction In_2Te_3 = abscissa in Figure 4

ΔH = "apparent" latent heat of fusion of β phase = $38,550 \text{ cal/gm mol}$

R = gas constant = $1.987 \text{ cal/gm mol } ^\circ\text{K}$

T_f^* = "apparent" melting point of β phase = $1070^\circ\text{K} = 797^\circ\text{C}$

T = temperature, $^\circ\text{K}$, of liquidus line at composition x_β over region (b) defined above

CRYSTAL STRUCTURES

The differential thermal analysis runs gave erratic results below about 600°C and could not be relied on to clarify the solid-solid transformations which were found particularly at the high indium end of the diagram. X-ray powder-pattern studies, microscopic examinations, and diffusion couples were used.

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TABLE II. TABULATION OF X-RAY LINES FOR COMPLEX COMPOUNDS IN CdTe-In₂Te₃ SYSTEM[§]

N ²	#810 50% In ₂ Te ₃ (β)*			#371 75% In ₂ Te ₃ (γ)*			#303 100% In ₂ Te ₃ (ε)**		
	I	θ	sin ² θ	I	θ	sin ² θ	I	θ	sin ² θ
3	10	12.58	0.0474	10	12.62	0.0480	10	12.69	0.0482
3.25	2	13.05	0.0507				1	13.81	0.0565
4.0	1	14.43	0.0621	1	14.61	0.0638	1	14.59	0.0630
4.25	1	15.34	0.0700						
5.	2	16.29	0.0786				2	16.19	0.0778
5.25	3	16.70	0.0825	2	16.84	0.0838			
5.75	2	17.41	0.0895						
6.0	2	17.85	0.0939	1	17.96	0.0954	3	18.34	0.0991
6.25	1	18.64	0.1022						
7.25	3	19.63	0.1138	1	19.81	0.1147	4	18.85	0.1045
8	10	20.63	0.1241	10	20.88	0.1271	10	20.88	0.1280
9	1	21.99	0.1402				1	22.07	0.1411
9.25	2	22.32	0.1442				3	22.45	0.1460
9.75	2	23.90	0.1641						
11	9	24.43	0.1711	9	24.66	0.1741	10	24.61	0.1743
14	1	27.73	0.2165						
15.25	3	29.07	0.2366	2	29.35	0.2410	4	28.85	0.2328
16	4	29.86	0.2480	5	30.12	0.2521	3	30.61	0.2594
17	3	31.08	0.2666	1	31.28	0.2691	3	31.44	0.2721
18	1	31.86	0.2786				2	32.40	0.2871
19	8	32.86	0.2945	8	33.03	0.2972	6	33.27	0.3006
21	1	34.80	0.3257				2	34.06	0.3135
22	1	35.71	0.3406						
23	1	36.79	0.3586				1	36.30	0.3505
23.25							1	36.45	0.3529
24	8	37.56	0.3716	9	37.91	0.3778	7	37.96	0.3782
25	2	38.65	0.3891						
25.25				2	38.93	0.3948	1	38.98	0.3953
26	2	39.60	0.4018	1	39.64	0.4077			
27	5	40.21	0.4168	5	40.64	0.4231	4	40.57	0.4230
27.25							2	41.42	0.4376
29				3	42.46	0.4577	1	42.38	0.4542
29.25	4	42.20	0.4512				1	43.83	0.4793

*Heat treated at 500°C for 100 hours, water quenched.

**100% In₂Te₃ was slowly cooled to room temperature.

§All measurements made from Cu-Kα radiation through nickel filter.

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TABLE II. TABULATION OF X-RAY LINES FOR COMPLEX COMPOUNDS IN CdTe-In₂Te₃ SYSTEM (Continued)

N ²	#810 50% In ₂ Te ₃ (β)*			#371 75% In ₂ Te ₃ (γ)*			#303 100% In ₂ Te ₃ (ε)**		
	I	θ	sin ² θ	I	θ	sin ² θ	I	θ	sin ² θ
31	2	43.93	0.4813						
32	4	44.62	0.4943	5	45.13	0.5038	1	45.85	0.5145
34	1	46.32	0.5230						
35	5	47.26	0.5394	6	47.73	0.5482	4	47.78	0.5486
37							1	48.55	0.5618
37.25	1	49.23	0.5735						
38.25	1	49.84	0.5841						
39									
39.25	1	50.99	0.6038				1	51.90	0.6193
40				6	52.22	0.6272	3	52.37	0.6270
40.25	4	51.66	0.6152						
41.25	1	52.63	0.6315						
43	3	54.50	0.6628	4	54.17	0.6743	2	55.06	0.6719
45	3	56.31	0.6919				1	56.16	0.6894
45.25	2	58.45	0.7262				2	56.95	0.7029
47.25				2	59.24	0.7395			
48	1	59.22	0.7381	1	59.89	0.7502	3	59.64	0.7443
49.25	3	60.36	0.7555						
50.25	1	61.14	0.7670						
52	5	62.26	0.7834	3	62.71	0.7892			
53				1	64.79	0.8189			
53.25	1	64.58	0.8157						
54.25	1	65.66	0.8302						
55.25				1	67.95	0.8590			
55.5	2	66.99	0.8473						
56	6	67.80	0.8568	6	68.64	0.8710			
57	3	68.19	0.8622						
59	5	72.33	0.9081						
61	2	75.66	0.9387						
62	2	76.81	0.9479						

*Heat treated at 500°C for 100 hours, water quenched.

**100% In₂Te₃ was slowly cooled to room temperature.

§All measurements made from Cu-Kα radiation through nickel filter.

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In order to interpret the results of these studies, it was necessary first to characterize all the phases in the system which were known to exist as a result of the DTA measurements and the literature survey.

All phases were carefully analyzed by X-ray powder techniques. The diffraction patterns of the β , γ , and ϵ compounds all contain the zinc blende lines characteristic of the α and δ phases. However, the distribution of the additional weaker lines are distinctive, although it requires very careful measurements to separate them. The results of the powder-pattern analyses are shown in Table II. The $\sin \theta$ values were taken from the films, which were made in 57.3-mm-diameter cameras with Cu-K α radiation through a nickel filter to eliminate the Cu-K β radiation. The X-ray film was covered with an aluminum foil to reduce spurious radiation effects which arise from scattering. The N^2 values listed were calculated by assuming that the structures are tetragonal with $c/a = 2.00$; hence

$$N^2 = h^2 + k^2 + \frac{l^2}{4} \quad (2)$$

The tabulated values of $\sin^2 \theta$ were quite reproducible from sample to sample. However, more recent diagrams on a 114.6-mm-diameter camera indicated poorer reproducibility; hence we are not certain as to the accuracy of the lines reported in Table II, but their precision was good. More careful structure studies are in progress and will be reported later.

The structure studies in this system have been based solely on X-ray powder patterns and are quite difficult to interpret. It is almost impossible to distinguish the atom positions from X-ray intensity measurements alone. Since Cd, In, and Te have atomic numbers 48, 49, and 52, respectively, they have almost identical X-ray scattering factors. However, since all these structures seem to be derived from the basic zinc blende structure, we have adopted the assumption of Hahn et al, (Reference 9) that the tellurium atoms alone form a face-centered cubic lattice, with the metallic atoms disposed in the tetrahedral interstices. The results of the measurements on each phase are discussed below, and summarized in Table III.

4.1. THE CdTe (α) PHASE

The α -phase (CdTe) has a zinc blende structure with lattice constant $a = 6.488 \pm 0.002$ A. Zachariassen (Reference 11) reported that $a = 6.477$ kX.

4.2. THE CdIn₂Te₄ (β) PHASE

The first peritectic compound (CdIn₂Te₄) has been characterized by Hahn et al. (Reference 9) as a tetragonal, chalcopyritelike structure with space group S_4^2 or $I\bar{4}$ (Figure 5). Our X-ray

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TABLE III. PROPERTIES OF COMPOUNDS IN CdTe-In₂Te₃ SYSTEM

Phase	Composition	Transition Temperature (°C)	Structure	Lattice Constants (Å)
α	CdTe	1098 1106 (3)* 1090 (4)	T_d^2 $F\bar{4}3m$	$a = 6.488 \pm 0.002$ $a = 6.477 \text{ kX (11)}$
β	CdIn ₂ Te ₄	785	S_4^2 $I\bar{4}$ (9)	$a = 6.235 \pm 0.002$ $c = 12.47 \pm 0.002$ $a = 6.192$ (9) $c = 12.38$ (9)
γ	Cd ₇ In ₃₈ Te ₆₄	702		
δ'	CdIn ₁₀ Te ₁₆	695	T_d^2 $F\bar{4}3m$	$a = 6.16$ (9)
δ	In ₂ Te ₃	667	T_d^2 $F\bar{4}3m$	$a = 6.171 \pm 0.002$ $a = 6.146$ (12)
ϵ	In ₂ Te ₃	625	T_d^2 $F\bar{4}3m$ (6) Im $m2$ (7) $P4_2/mnm$ (7)	$a = 18.40 \pm 0.04$ (6)

*Numbers in parentheses refer to "References" section, at the end of the text.

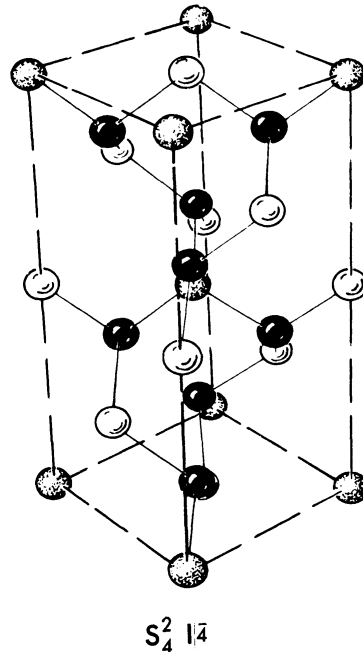


FIGURE 5. UNIT CELL STRUCTURE FOR CdIn₂Te₄. Black = tellurium atoms; white = indium atoms; gray = cadmium atoms.

results for the β -phase as tabulated in Table II agree with those reported by Hahn. We have accepted his structure designation without repeating the intensity calculations. We find a slight difference in lattice constants, with $a = 6.235 \pm 0.002$ A, and $c = 12.47 \pm 0.002$ A, whereas Hahn et al. (Reference 9) report that $a = 6.192$ A, and $c = 12.38$ A.

4.3. THE $\text{Cd}_7\text{In}_{38}\text{Te}_{64}$ (γ) PHASE

The second peritectic ($\text{Cd}_7\text{In}_{38}\text{Te}_{64}$), or γ -phase, also seems to be a chalcopyritelike structure very similar to the β -phase. However, this phase has only about half as many powder-pattern lines as the β -phase, as indicated in Table II. The distances between the diffracting planes in the β and γ phases also seem to be substantially identical, so that it is extremely difficult to distinguish them from each other on the X-ray powder pictures in the region from 50 mol % to 75 mol % In_2Te_3 . Hence it is not surprising that Hahn et al. (Reference 9) have not defined the existence of the γ -phase, since their survey relied only on X-ray analyses and no samples were run between 66.7 mol % In_2Te_3 and 80 mol % In_2Te_3 .

4.4. THE In_2Te_3 (δ) PHASE

The δ -phase extends from $\text{CdIn}_{10}\text{Te}_{16}$ to pure In_2Te_3 , and shows again the zinc blende structure, with some metal positions randomly vacant. At the pure In_2Te_3 side of the phase field we have found that $a = 6.171 \pm 0.002$ A whereas Hahn and Klinger (Reference 12) reported $a = 6.146$ A.

4.5. THE In_2Te_3 (ϵ) PHASE

The ϵ phase apparently forms from pure In_2Te_3 in the δ structure, by transformation in the solid state at about 625°C. Our line measurements are tabulated in Table II. Inuzuka and Sugaike (Reference 6) report the structure to be $\text{F}\bar{4}3\text{m}$ with $a = 18.40 \pm 0.04$ A.

Woolley and Pamplin (Reference 7), and Holmes (Reference 7) have also considered the structure of low-temperature modification of In_2Te_3 . Holmes suggests a tetragonal structure of $\text{P}4_2/\text{mcm}$ or $\text{P}4_2/\text{mnm}$, comprising basically nine cubic unit cells of a fluorite lattice with about two-thirds of the sites vacant. Woolley and Pamplin (Reference 7), on the other hand, suggest an orthorhombic structure, $\text{Imm}2$, derived from the zinc blende configuration with selected vacancies.

SOLID-SOLID EQUILIBRIA

With the individual phases characterized, several additional studies were then conducted to ascertain the solid-solid equilibria boundaries. These studies comprised one diffusion-couple experiment, X-ray powder-pattern studies, and photomicrograph studies.

5.1. DIFFUSION COUPLE

A diffusion couple was run at 650°C between CdIn_2Te_4 and In_2Te_3 , and only one intermediate constituent was found. It appeared that the intermediate phase was formed exclusively on the In_2Te_3 side of the interface, suggesting that the cadmium diffused through the structure at a much more rapid rate than the other constituents.

5.2. THE X-RAY POWDER-PATTERN STUDIES

Four separate series of X-ray powder-pattern pictures were taken. Two series were taken at 2.5% intervals over most of the range between 75 mol % and 100 mol % In_2Te_3 . Series I was quenched from 650°C and showed only γ and δ phases. The δ -phase was pure above 82.5 mol % In_2Te_3 . Series II was quenched from 500°C and showed that the pure δ -phase region extended only from 82.5 mol % to 92.5 mol % In_2Te_3 .

In order to ascertain the position of the eutectoid, Series III was run. Samples of 87.5 mol % In_2Te_3 were annealed at temperatures of 200°C, 250°C, 300°C, 350°C, 375°C, 400°C, 426°C, 505°C, and 650°C. It was observed from these measurements that, at 350°C and below, the X-ray lines are characteristic of a mixture containing both the γ and ϵ phases. At 375°C and above, the γ -phase lines are replaced by δ -phase lines, and we conclude that the eutectoid is between 350°C and 375°C. Series IV comprises miscellaneous X-ray determinations which were run to corroborate the conclusions from the microscopic and differential thermal analysis experiments. The results of the X-ray phase studies are summarized in Table IV.

5.3 PHOTOMICROGRAPH STUDIES

The retrograde solubility of CdIn_2Te_4 in CdTe was established by annealing samples in the α and $(\alpha + \beta)$ range at 600°C and observing a Widmanstatten structure in microscopic examinations. A typical photomicrograph taken at 33.3 mol % In_2Te_3 is shown in Figure 6. The pure β crystallites show up as a single phase material, whereas the α crystallites contain small platelets of precipitated β phase. The relative amount of the α phase observed in the photomicrographs decreased as the percentage of In_2Te_3 in the composition of the sample was increased towards the 50 mol % point. The solubility of In_2Te_3 in CdTe at the 785°C peritectic is more than 25 mol %.

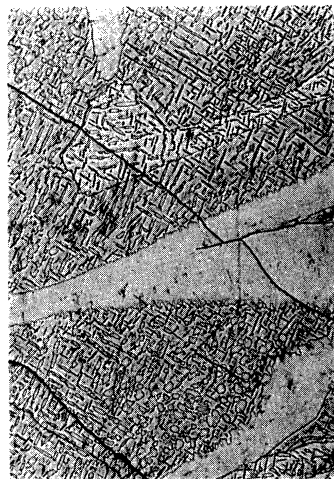


FIGURE 6. PHOTOMICROGRAPHS AT 33.3 mol % In_2Te_3 IN CdTe . Clear β -phase region (CdIn_2Te_4) and Widmanstatten structure in α -phase (CdTe). Magnification = 100X.

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TABLE IV. SUMMARY OF X-RAY STUDIES USED TO DEFINE PHASE BOUNDARIES

Sample No.	In ₂ Te ₃ (%)	Heat Treatment* (°C)	Phases Present
Series I			
371	75.0	650 AQ	γ
351	80.0	650 AQ	δ + γ
368	82.5	650 AQ	δ
356	85.0	650 AQ	δ
366	87.5	650 AQ	δ
357	90.0	650 AQ	δ
348	95.0	650 AQ	δ
370	97.5	650 AQ	δ
303	100.0	650 AQ	δ
Series II			
371	75.0	500 AQ	γ
351	80.0	500 AQ	γ + δ
368	82.5	500 AQ	δ
356	85.0	500 AQ	δ
815	87.5	500 AQ	δ
357	90.0	500 AQ	δ
369	92.5	500 AQ	δ
348	95.0	500 AQ	δ + ε
370	97.5	500 AQ	δ + ε
303	100.0	500 AQ	ε
Series III			
817	87.5	200 AQ	γ + ε
817	87.5	250 AQ	γ + ε
817	87.5	300 AQ	γ + ε
817	87.5	350 AQ	γ + ε
817	87.5	375 AQ	δ + ε
817	87.5	400 AQ	δ + ε
817	87.5	426 AQ	δ
815	87.5	505 AQ	δ
366	87.5	650 AQ	δ

*AQ = Air quenched; FC = furnace cooled at 2.5°C/minute;
WQ = water quenched.

WILLOW RUN LABORATORIES TECHNICAL MEMORANDUM

TABLE IV. SUMMARY OF X-RAY STUDIES USED TO DEFINE PHASE BOUNDARIES (Continued)

Sample No.	In ₂ Te ₃ (%)	Heat Treatment* (°C)	Phases Present
Series IV			
805	0.0	600 FC	α
333	5.0	600 FC	α
332	20.0	600 FC	$\alpha + \beta$
319	25.0	600 FC	$\alpha + \beta$
346	30.0	600 FC	$\alpha + \beta$
355	33.3	600 FC	$\alpha + \beta$
330	37.0	600 FC	$\alpha + \beta$
340	45.0	600 FC	$\beta + \alpha$
810	50.0	600 FC	β
810	50.0	650 AQ	β
810	50.0	660 WQ	β
308	60.0	600 FC	β
308	60.0	695 WQ	$\beta + \gamma$
349	63.0	600 FC	$\beta + \gamma$
809	63.0	770 WQ	$\beta + \gamma$
310	66.7	600 FC	$\beta + \gamma$
350	70.0	600 FC	$\beta + \gamma$
350	70.0	695 WQ	$\beta + \gamma$
318	75.0	600 FC	γ
318	75.0	695 WQ	γ
811	80.0	570 AQ	$\gamma + \delta$
811	80.0	684 AQ	δ
356	85.0	600 FC	δ
357	90.0	525 AQ	δ
357	90.0	600 FC	δ
357	90.0	684 AQ	δ
303	100.0	665 AQ	δ
808	100.0	665 AQ	δ
303	100.0	500 AQ	ϵ
808	100.0	500 AQ	ϵ

*AQ = Air quenched; FC = furnace cooled at 2.5°C/minute;
WQ = water quenched.

SEMICONDUCTOR PROPERTIES

All these compounds should be semiconductors according to the rules promulgated by Mooser and Pearson (Reference 13), as long as there are neither tellurium-to-tellurium bonds nor intermetallic bonds between indium and/or cadmium. These restrictions are fulfilled in the postulated structures.

6.1. PRELIMINARY MEASUREMENTS

A series of samples was prepared for infrared transmission measurements, and the preliminary results of optical energy gaps in electron volts, shown in Table V, were found. Since reasonably high transmissions of the order of 30% to 40% were obtained in these samples, the indication is that they were quite pure. The gap values were obtained by measuring the infrared transmission. The region of steepest slope on the cutoff edge of the short wavelength was projected to zero transmission.

TABLE V. ENERGY GAPS OF COMPOUNDS
IN CdTe-In₂Te₃ SYSTEM

Phase	Composition	Energy Gap		
		Authors' Results (ev)	Other Reported Results (ev)	(Reference)
α	CdTe	1.32	1.42	(14)
			1.51	(15)
			1.51	(4)
			1.55	(3)
β	CdIn ₂ Te ₄	1.077	0.9	(16)
γ	Cd ₇ In ₃₈ Te ₆₄	1.06	--	
δ	In ₂ Te ₃	--	2.4	(15)
			1.04	(8)
ϵ	In ₂ Te ₃	1.04	0.93	(15)
			1.12	(8)

In order to evaluate their significance, the results must first be compared with the measurements reported in the literature. The magnitude of the energy gap in CdTe at room temperature has been reported by Bube (Reference 14) to be 1.42 ev. Lawson, Nielsen, Putley and Young (Reference 3) report an infrared transmission edge of 0.8μ , or 1.55 ev. DeNobel (Reference 4) reports a gap of 1.51 ev using both infrared transmission and photo emf measurements. Appel and Lautz (Reference 15) measured differences in the activation energies of CdTe and In_2Te_3 by measuring resistivity vs. temperature from room temperature to their melting points. They report an average gap of 1.51 ev for CdTe. Busch, Mooser and Pearson (Reference 16) report a gap of 0.9 ev for CdIn_2Te_4 using resistivity vs. temperature measurements. Appel and Lautz (Reference 15) report that the low-temperature form of In_2Te_3 shows an average gap value of 0.93 ev, whereas the high-temperature average value is 2.4 ev. Of three samples measured, one never showed the high-temperature form; of the other two, one showed the transition at 250°C and the other at 600°C , which is in moderately good agreement with the transition at $620 \pm 5^\circ\text{C}$ reported in Section 4.5. Gasson, Holmes, Parrott, and Penn (Reference 8) have reported a gap of 1.04 ev for the δ phase and 1.12 ev for the ϵ phase of In_2Te_3 .

In view of the evidence, we feel that our preliminary gap values are qualitatively significant. There appear to be monotonic relationships among the energy gap, the transition temperatures, and the compositions of the compounds in this system. Although our energy-gap measurements are only approximate, and the agreement with the literature is poor in many instances, the internal consistency of the data is qualitatively significant.

6.2. MEASUREMENTS ON CdIn_2Te_4

More careful infrared transmission measurements were made on two samples from a particular ingot of CdIn_2Te_4 (cadmium indium telluride), which was felt to be better in quality than the other materials, and the results are shown in Figure 7. The transmission was measured at room temperature by an in-the-beam, out-of-the-beam technique on samples of thicknesses 0.121 mm and 0.533 mm at the indicated points. The energy gap was taken as the intersection of the sharp band edge with the abscissa, corresponding to $\Delta E_{300^\circ\text{K}} = 1.077$ ev. The refractive index is 3.08 in the region of the band edge and decreases to 2.83 at 2μ ; it then decreases continuously to 2.72 at 14.5μ . The index of refraction was calculated for the region between 7.5μ and 14.5μ from the interference fringes produced by multiple internal reflections. Over the region from 1.1μ to 8.0μ , it was calculated from essentially reflection measurements, with good agreement in the overlap region. The drop in transmission at long wavelengths can probably be attributed to free carrier absorption.

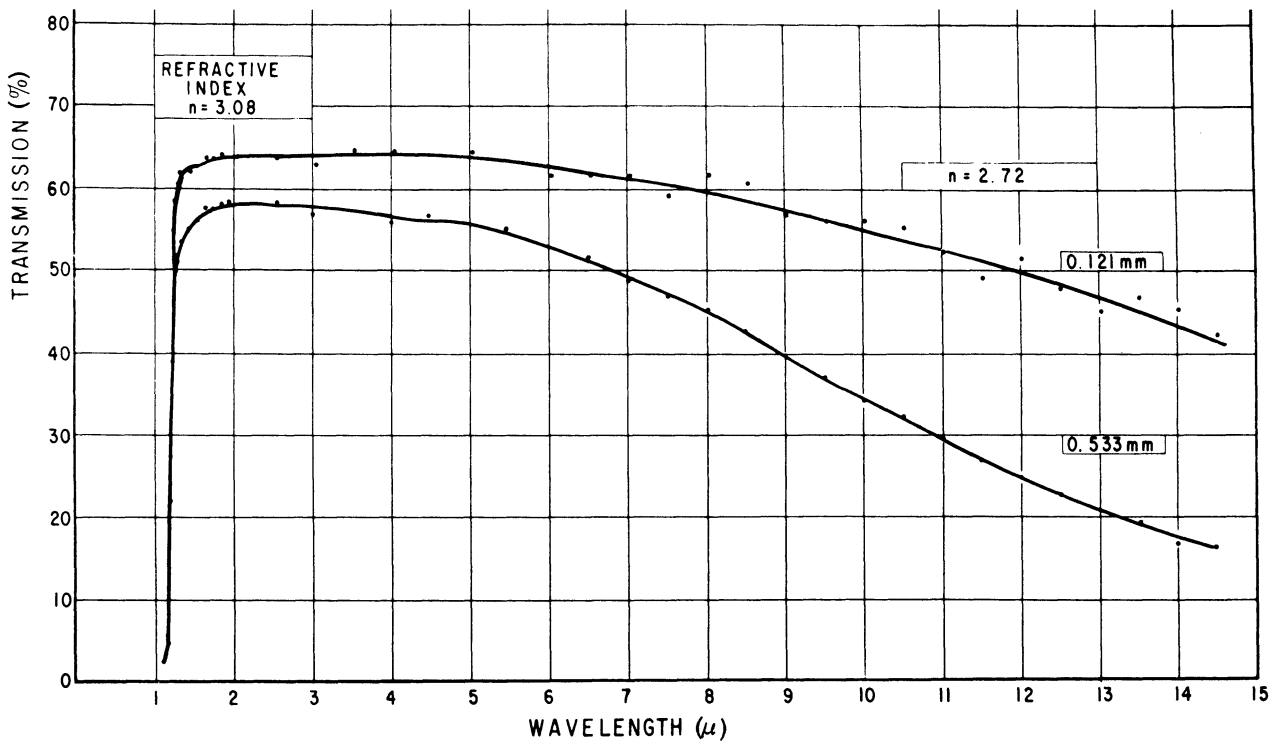


FIGURE 7. INFRARED TRANSMISSION FOR CdIn_2Te_4 . CdIn_2Te_4 , No. 1004;
 $\Delta E_{300^\circ\text{K}} = 1.077 \text{ eV}$; $\rho_{300^\circ\text{K}} = 2.9 \text{ cm}$.

7

CONCLUSION

It seems apparent that the problems which must be defined and solved before a large number of ternary compounds can be investigated intelligently are even greater than we thought a year ago. This is particularly true for the types of compounds with which we have been working. However, we feel that the potential flexibility in optimizing the semiconductor properties for many applications by using ternary compounds makes continued efforts in this direction desirable and necessary, and that ultimate results will be significant. A. M. G. D.

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Lars Thomassen and Donald Mason. Memorandum of Project
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(Memo no. 2900-80-R)

(Contract DA-36-039 SC-78801) Unclassified memorandum
The phase diagram for the binary system CdTe-In₂Te₃ has been
obtained by correlating information from differential thermal anal-
ysis measurements, microscopic studies, diffusion couples, and
X-ray powder patterns.

In establishing the terminal points for this diagram, the melting
point for In₂Te₃ was found to be $667 \pm 1^\circ\text{C}$. For CdTe, a melt-
ing point of $1098 \pm 3^\circ\text{C}$ was indicated.

In going across the diagram, three peritectics are apparent. The
first (β) is at 785°C and 50 mol % In₂Te₃; the second (γ) at about
 702°C and 74 mol % In₂Te₃; and the third (δ') is at 695°C and
about 83 mol % In₂Te₃. There is a large retrograde solubility of
CdIn₂Te₄ in CdTe. The δ -phase transforms to an ϵ -phase at
 625°C and 100% In₂Te₃. The addition of CdTe stabilizes the δ -phase
(over)

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