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DETERMINATION OF LIQUIDUS TEMPERATURES OF Hg-RICH Hg-Cd-Te ALLOYS BY DIFFERENTIAL THERMAL ANALYSIS

P. J. Meschter* and K. E. Owens**

McDonnell Douglas Corporation
St. Louis, MO 63166

T. Tung

Martin Marietta Laboratories
Baltimore, MD 21227

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Liquidus temperatures have been measured for eight $(Hg_{1-x}Cd_x)_yTe_{1-y}$ compositions with $0.099 \leq x \leq 0.401$ and $0.703 \leq y \leq 0.900$ by differential thermal analysis (DTA). The measured liquidus temperatures are in good agreement with those calculated from the thermodynamic model of Tung et al. The change in liquid composition caused by mercury vaporization into the free volume of the DTA ampule introduces an error in the liquidus temperature of less than 1.5°C.

Key words: liquidus, mercury cadmium telluride, thermal analysis.

Introduction and Experimental Procedure

Growth of high-quality HgCdTe crystals from the melt requires an accurate knowledge of the phase relationships and, in particular, the liquidus temperatures in the ternary Hg-Cd-Te system. The work reported here is an extension of previous studies of the Hg-Cd-Te liquidus by DTA (1,2) to a region of higher Hg mole fraction.

The DTA apparatus used in this study was similar to that previously described (1), except for the omission of the diatomaceous earth packing in the reaction tube. The data acquisition system was identical to that described in Ref. 2, except for the substitution of higher-sensitivity chromel-alumel thermocouples for Pt/Pt-10% Rh. Sources and purities of the Hg, Cd, and Te used here; cleaning of the Cd, Te, and ampule; and loading of the elements into the ampule have been described pre-

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**Senior Scientist, McDonnell Douglas Research Laboratories*

***Electronic Engineer, McDonnell Douglas Microelectronics Center* viously (1). Considerable care was taken in selecting quartz tubing for the ampules and in sealing the ampules to avoid weak areas or stress concentrations, which would allow the ampules to explode under the high Hg vapor pressures developed during an experiment. Only quartz tubing which was completely free of striations, air bubbles, and other visible defects survived the experiments. The sealed ends of the ampules were flame annealed until no sign of stress fringes was observed under polarized light.

After loading and sealing, each ampule was placed in a sealed Inconel cylinder in a tube furnace, heated at 2°C/min to a temperature 15-50°C above the estimated liquidus temperature, and held at that temperature for 16 h to ensure sample homogenization. The samples were then removed from the furnace and allowed to cool on the bench to room temperature. This procedure protected the DTA apparatus by allowing most explosions caused by imperfections in the ampule to occur during homogenization.

To perform an experimental run, the sample and reference ampules were heated at 2°C/min to a temperature 75-100°C below the estimated liquidus to pre-equilibrate the sample in the (liquid + solid) phase field. The melting temperature of the antimony reference, determined during heating, was always within 1°C of the accepted value of 630.85°C (3). The apparent melting temperature of a silver reference used in one run was within 1°C of the accepted value of 960.80°C (3). After pre-equilibration, the liquidus temperature was determined from the average of two heating runs at 2°C/min, separated by a 16-h re-equilibration.

Results and Discussion

Liquidus temperatures of eight $(Hg_{1-x}Cd_x)_yTe_{1-y}$ alloys with $0.099 \leq x \leq 0.401$ and $0.703 \leq y \leq 0.900$ are listed in Table I. Liquidus temperatures of samples at $x = 0.2$ and $y = 0.5$ and 0.6 were also measured, and agree with previously measured values (1,2) within experimental error. Uncertainties in these data are similar to those determined and discussed previously (2). As shown in Fig. 1, the maximum in the liquidus for any value of x occurs at a value of y well above 0.5, in agreement with calculations (4) and the results of Ref. 2.

Tung et al. (4,5) reviewed extensive phase diagram and thermodynamic data in the Hg-Cd-Te system, along with calculated values for these data obtained from a simultaneous, thermodynamically self-consistent fit to the experimental data. The thermodynamic description of the liquid phase was based on a complex associated-solution model. Consistency

Table I. Liquidus Temperatures and Hg Vapor Pressures for Hg-Rich $(Hg_{1-x}Cd_x)_yTe_{1-y}$ Alloys.

Original Composition x	y	Measured Liquidus (°C)	Calculated Liquidus (°C)	Vapor Pressure of Hg at Calculated Liquidus, (Pa /atm/)
0.200	0.500	793 ± 2	792	3.67×10^6 [36.2]
0.200	0.500	792 ± 3[a]	792	3.67×10^6 [36.2]
0.200	0.600	855 ± 4	863	8.18×10^6 [80.7]
0.200	0.600	849 ± 4[b]	863	8.18×10^6 [80.7]
0.099	0.751	858 ± 5	849	1.01×10^7 [99.6]
0.101	0.851	866 ± 4	856	1.21×10^7 [119.3]
0.201	0.701	915 ± 4	908	1.28×10^7 [126.7]
0.200	0.801	917 ± 3	921	1.55×10^7 [152.7]
0.299	0.703	956 ± 3	950	1.58×10^7 [156.1]
0.300	0.900	902 ± 3	907	1.26×10^7 [123.9]
0.399	0.751	983 ± 4	979	1.66×10^7 [164.2]
0.401	0.851	950 ± 3	951	1.31×10^7 [128.9]

[a] Reference 1
[b] Reference 2

between measured and calculated phase boundaries and vapor pressures was excellent over the whole composition range. Liquidus temperatures for our experimental compositions, calculated from Tung et al.'s model, are listed in Table I and shown as iso-concentration lines in Fig. 1. Agreement between measured and calculated values is within 5°C in five of eight cases. One measured data point at $x = 0.3$, $y = 0.8$ was discarded on the basis of a 22°C deviation from the calculated value.

The calculated Hg vapor pressures listed in the last column of Table I were used to determine the approximate change in the liquid composition at the liquidus caused by Hg vaporization into the free volume of the ampule. The calculated Hg pressure and the measured free volume were substituted into the ideal gas equation to determine the number of moles of Hg vaporized. The liquid composition was corrected for this Hg loss and the liquidus temperature was recalculated. The change in the liquidus temperature was always less than 1.5°C, well within experimental uncertainty. Losses of Cd and Te into the free volume were negligible relative to that of Hg.

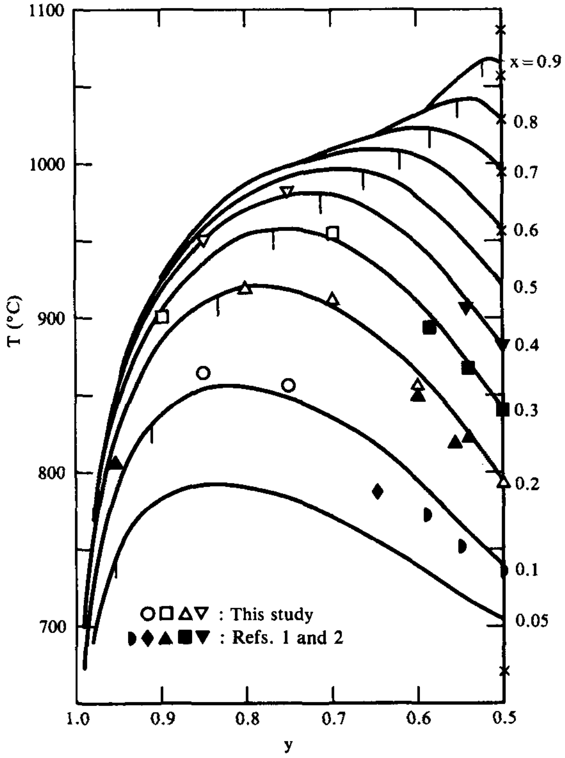

Fig. 1 Liquidus surface on the Hg-Cd rich side of the HgTe-CdTe pseudobinary section. The liquidus temperature in °C is plotted against the atom fraction of Hg + Cd for fixed values of x (shown on the right vertical axis). The composition of the liquid phase is written as $(Hg_{1-x}Cd_x)_yTe_{1-y}$. The single diamond point is for $x = 0.09$. The arrow below each calculated liquidus line (4) indicates where the atom fractions of Cd and Te are equal.

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