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Short communication

Phase equilibria in the quasibinary GaSb–Pb system

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Abstract

The phase relationships of the quasibinary GaSb–Pb section of the ternary Ga–Sb–Pb system were studied by means of the X-ray diffraction (XRD) and optical microscopy. Phase reactions were determined using differential thermal analysis (DTA). Based on this results phase diagram of the GaSb–Pb section was constructed, which was compared with literature data.

Keywords: Ga–Sb–Pb system; Phase diagram; Thermal analysis; X-ray diffraction; Optical microscopy

1. Introduction

The equilibrium phase diagrams are important tool in understanding the interfacial reactions since they indicate the equilibrium state that the materials would attain if they were left for a sufficient length of time at a given temperature. Because of that they present a starting point for any study of metal–semiconductor systems. In order to investigate the phase relationships in one of these systems the ternary Ga–Sb–Pb system, the quasibinary GaSb–Pb section was chosen for the experimental investigation in this work.

The equilibrium phase diagrams for the constitutive binary systems are well defined. Based on the numerous available literature data Ngai et al. assessed the Ga–Sb system [1]. The stoichiometric compound Ga–Sb is the only intermediate phase within the system that crystallizes in the cubic zinc blende structure and melts congruently at 711.7 °C. On the Sb-rich side eutectic reaction occurs at 589.3 °C; on the Ga side a degenerate eutectic reaction occurs at 29.7 °C.

The phase relationships in the Ga–Pb system were investigated first by Puschin et al. [2], who reported a monotectic transformation at 317 °C and 86.47 at.% Pb. No results concerning the compositions of the equilibrium phases of the miscibility gap were given in that study. Greenwood [3] determined the limits of the solid solubility while Predel [4] investigated the Ga–Pb system in the liquid state by differential thermal analysis and defined the contour of the miscibility gap. Ansara and Ajersch [5] assessed the Ga–Pb system. Mathon et al. [6] also assessed this system using a computerized optimization procedure.

Lead and antimony form a eutectic system with limited solid solubilities. On the Pb-rich side the liquidus has been well established by Blumenthal [7] and the solidus by Rhines and Pellini [8]. Obinata and Schmid [9] determined the solid solubility by X-ray diffraction.

Although there are many phase diagram data available for the constitutive binary systems, there seems to be only one published data for ternary the Ga–Sb–Pb alloys. Baranov et al. [10] gave complete calculated liquidus projection and phase diagram of the GaSb–Pb section.

In this work differential thermal analysis, X-ray diffraction and optical microscopy results were used for the phase diagram determination of the GaSb–Pb system.

2. Experimental

The samples were prepared from gallium (99.99%), antimony (99.99%) and lead (99.99%). Calculated amounts of the pure elements were weighted (±0.05 mg) into quartz glass ampoules that were subsequently sealed under vacuum (10^{-1} Pa). The samples were homogenized at

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1073 K for 15 h and cooled inside the furnace to the room temperature.

XRD was applied for the samples phase identification. The diffraction data were collected on an X-ray Siemens diffractometer with a power of 40 kV and 20 mA, employing filtered Cu Kα radiation.

Microstructure analysis of the samples was performed by optical microscopy, using a Reichert MeF2 microscope.

DTA measurements were carried out with the Derivatograph 1500 (MOM Budapest) apparatus under following conditions: air atmosphere, heating rate 10 °C/min, T_{max} = 1073 K. The total mass for each sample was approximately 2 g. The precision of the measurement in the investigated temperature interval was ±5 °C.

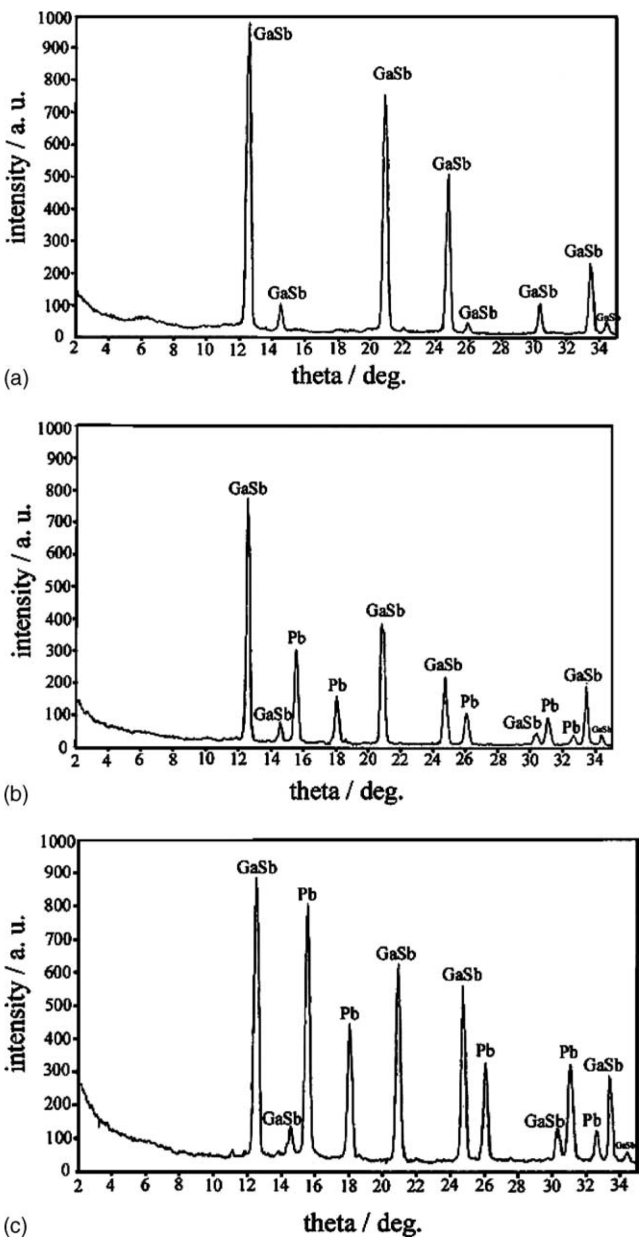


Fig. 1. X-ray diffraction powder patterns for (a) GaSb compound, (b) $Ga_{40}Sb_{40}Pb_{20}$ alloy, and (c) $Ga_{25}Sb_{25}Pb_{50}$ alloy.

Table 1
DTA results for the quasibinary GaSb–Pb alloys

Composition (at.% Pb)	Thermal effects (°C)	
	Eutectic reaction	Liquidus heating
0	–	712
10	312	674
20	311	646
30	311	621
40	312	603
50	311	575
60	312	551
70	312	533
80	312	499
90	311	415
95	312	350
98	312	–

3. Results and discussion

GaSb intermediate phase and alloys with 10, 20, 30, 40, 50, 60, 70, 80, 90, 95 and 98 at.% Pb were studied in this work. XRD characterization of the samples showed the existence of a two-phase structure in all investigated alloys at the room temperature. Using XRD, the phases were identified as gallium antimonide and elementary lead. Characteristic XRD powder patterns for selected samples are given in Fig. 1.

The results of the DTA heating measurements are listed in Table 1, together with the interpretation of the various thermal effects. In order to test reproducibility of the results every measurement run was repeated one more time. No significant temperature deviation was found between the first series and repeated series of DTA measurements.

It is found that the quasibinary eutectic reaction L ↔ GaSb + Pb occurs at 312 °C in this system. The eutectic concentration was found to be 98 at.% Pb. Graphical representation of the obtained phase diagram for the GaSb–Pb system, compared with literature data [10], is given in Fig. 2.

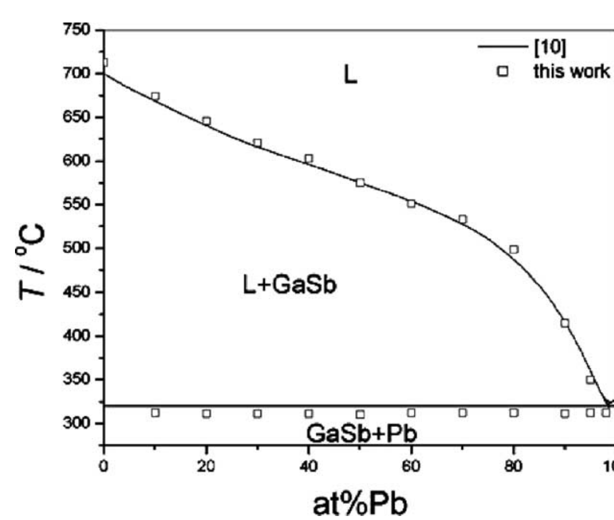
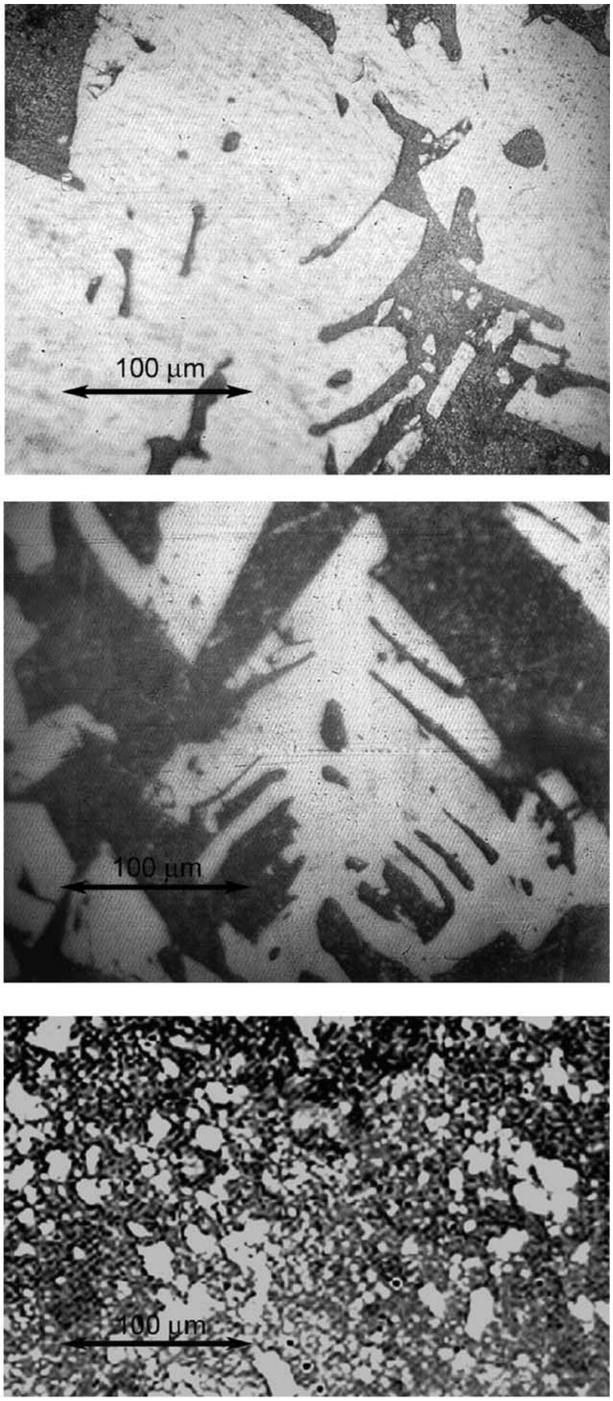


Fig. 2. Quasibinary GaSb–Pb section.



(a) (b) (c)

Fig. 3. Characteristic optical microphotographs (magnification 300×) for (a) $Ga_{30}Sb_{30}Pb_{40}$ alloy, (b) $Ga_{20}Sb_{20}Pb_{60}$ alloy, and (c) $Ga_{1}Sb_{1}Pb_{98}$ alloy (light regions: primary solidified GaSb crystals; dark regions: quasibinary eutectic GaSb + Pb).

It could be noticed that the phase diagram boundaries from DTA measurements in this work are in good agreement with existing literature data.

Characteristic microphotographs recorded by optical microscopy for the samples with 40, 60 and 98 at.% Pb are given in Fig. 3.

The quasibinary hypoeutectic alloys, presented in the widest part of the concentration range, solidify with the primary crystallization of the intermetallic compound GaSb (light regions) and the eutectic reaction/GaSb + Pb/(dark regions).

4. Conclusion

The GaSb–Pb system was studied by DTA, XRD and optical microscopy. Based on these results, the equilibrium phase diagram was constructed and quasibinary eutectic reaction at 312 °C was identified. Two-phase equilibria between GaSb and Pb at the room temperature were found in all investigated ternary alloys. The results show good agreement with existing literature data.

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