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The binary constitution diagrams for the aluminium-silicon and aluminium-germanium systems at 56 kbar *(Research Note)*

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Abstract. Simple thermal analysis in the tetrahedral apparatus is applied to determine phase boundaries in constitution diagrams at very high pressures of the order of 56 kbar. This method has been applied to the Al-Si and Al-Ge systems, both of which have been extensively studied at ordinary pressures and both of which show well-known simple eutectic behaviour.

The samples used in this work were typically about 100 mg in total weight, in the form of cylinders of overall size 2.9 mm diameter × 3.0 mm length. The mole fractions were varied by combining either concentric cylinders or appropriately sized discs of the two materials. These samples were contained within a tantalum heater tube and end discs, as shown in figure 1a. The tube ran directly between opposite faces of a 23.8 mm edge pyrophyllite tetrahedron. A single thermocouple was carried in as shown through one edge of the tetrahedron. Thermocouples for this work must satisfy two conditions: (a) they must be of relatively small thermal mass; (b) they must remain continuous despite the large-scale deformation which occurs during compression of the tetrahedron. In practice we find that the Thermocoax type, in which Chromel and Alumel wires of 0.1 mm diameter are contained in a stainless steel sheath of 0.5 mm outside diameter, are very satisfactory. (Their use was first suggested to us by workers at the National Physical Laboratory, Teddington, UK.)

The thermocouple was mounted between tantalum discs as shown, to prevent reaction between it and the samples. To minimise reaction with the tantalum container, the samples were heated at pressure to a temperature below the liquidus and then quickly taken above it before quenching by disconnecting the heater current supply.

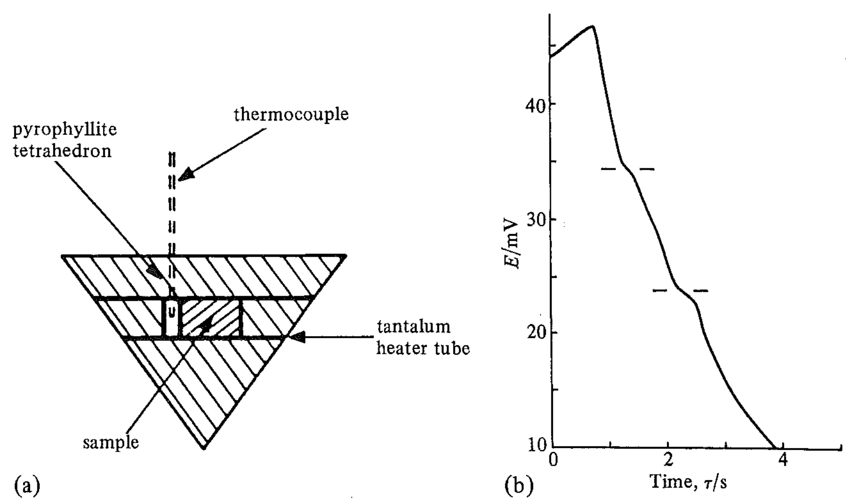

Figure 1. (a) Sample assembly. (b) Typical cooling curve.

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The sample was thus molten for a few seconds only. Subsequent metallographic examination showed that the sample was homogeneous throughout, and x-ray microprobe analysis confirmed that reaction with the container was negligible in most cases. In practice, reactions were found in some experiments towards the germanium-rich composites—these were easily recognised from anomalous cooling-curve behaviour, and were attributed to exposure to too long a time or too high a temperature in the molten phase.

The typical cooling curve observed in this work is shown in figure 1b. This was obtained by displaying the thermal emf, E, against a constant time base on a fast-acting x-y recorder. Note the two discontinuities, the first corresponding to the liquidus, and the second to solidification of the eutectic composition. Owing to the fast thermal response of the thermocouple, liquidus compositions close to the eutectic point were resolved without difficulty.

The two constitution diagrams obtained at 56 kbar by this method are shown in figure 2. The pressures in these experiments were inferred from the normal room-temperature calibrations of the apparatus; fixed-point calibration was made from the Bi(I–II) (25.4 kbar), Tl(II–III) (36.7 kbar), Ba(I–II) (55 kbar), and Bi(III–V) (77 kbar) transitions. Temperatures were corrected in line with the pressure corrections of Hanneman and Strong (1965).

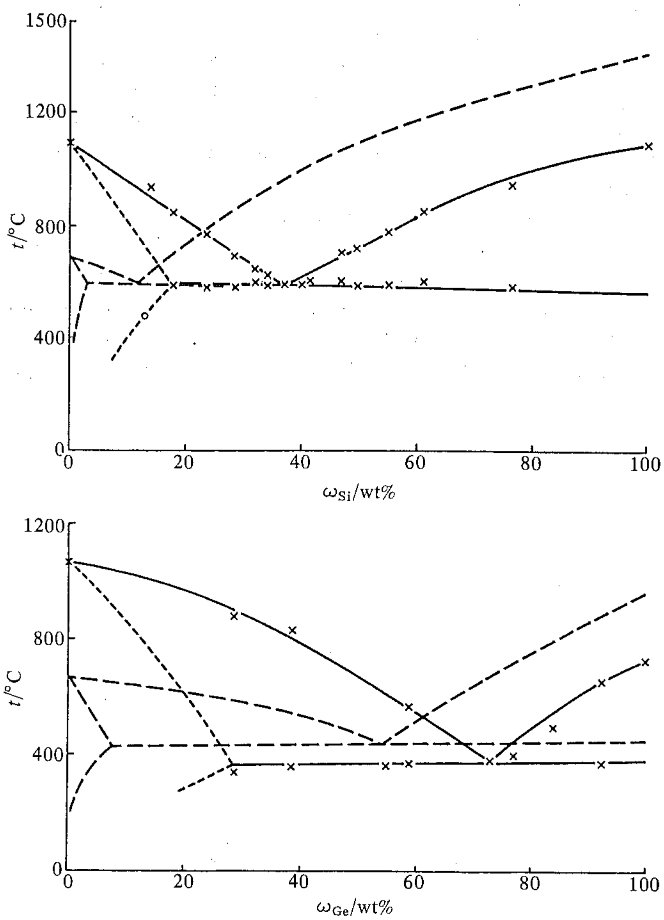

Figure 2. Constitution diagrams of the systems at 56 kbar. (a) Al–Si; (b) Al–Ge.

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System	Eutectic con	
	1 atm	5 e
Al–Si	11 Si	4
Al–Ge	46 Ge	2

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Figure 2 shows that quite large modifications of both systems have been effected by pressure. The magnitude of these changes produced in the liquidus is related to the large changes in the melting points, in opposite directions, of the two components in each case. The solid-solution range, particularly at the aluminium-rich composition, was markedly increased. It was difficult to measure the solid solubility by microprobe analysis owing to the 'coring' phenomena and to the relatively small size of the dendrites involved in our samples. Future experiments are planned to produce homogeneous phases in an attempt to obtain more definite values. These measurements indicate extreme values between 5 and 25 wt% silicon in aluminium, but the more usual value obtained is ~18 wt%. This contrasts with ~1.6 wt% silicon in aluminium at ordinary pressures. Similar effects are observed in one measurement only in the Al–Ge system, where the solid solubility of germanium in aluminium is also increased from 7.2 wt% to possibly as much as 27 wt% germanium.

Interesting effects are also observed in the variation of the position of the eutectic temperature and composition; these are summarised in table 1, where it will be noticed that agreement between the values for the eutectic composition derived (i) from extrapolation of the branches of the liquidus at 56 kbar, and (ii) from x-ray microprobe measurements, is excellent. It will be noted that although the aluminium content of the eutectic composition for both systems is considerably decreased at 56 kbar, the eutectic temperature for the Al–Si system is slightly increased, whereas that for the Al–Ge system is considerably decreased.

Table 1. Variation of the eutectic temperature, t_E, and composition with pressure for Al–Si and Al–Ge systems.

System	Eutectic composition (values in wt%)			t_E/°C	
	1 atm	56 kbar, liquidus extrapolation	56 kbar, x-ray microprobe	1 atm	56 kbar
Al–Si	11 Si	40 Si	41 Si	577	601
Al–Ge	46 Ge	28 Ge	26 Ge	425	359

Author's note. This article reports work carried out some years ago at STL, and now published in view of its relevance to the work of Kingon and Clark reported in this issue.

Reference

Hanneman R E, Strong H M, 1965 *J. Appl. Phys.* **36** 523