Density and Compressibility of Liquid Li-Pb Alloys

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Dedicated to Professor H. J. Seemann on the occasion of his 75th birthday

The density and the velocity of sound of liquid Li-Pb alloys have been measured in the composition ranges 0—17, 30—100 at % Pb and 0—10, 40—100 at % Pb, respectively. The temperature dependence of these properties has been determined for each sample in a temperature range of at least 130 K. The excess volume is negative and has a minimum at about 20 at% Pb, where the volume per atom at 800 °C is about 19% smaller than the volume of an ideal solution. The absolute value of the excess volume rises with the temperature, as does the absolute value of the deviation of the adiabatic compressibility from the mean compressibility.

Introduction

The properties of liquid Li-Pb alloys deviate strongly from the ideal behavior. Thus, very strong negative values of the thermodynamic properties of mixing have been observed for that system [1]. The excess stability function [2] probably exhibits a peak with an amplitude of several hundred kcal near the composition $x_{\text{Pb}} = 0.2$ [3]. At this composition the electrical resistivity of the liquid Li-Pb alloys has a sharp maximum and the thermoelectric power undergoes a change of sign [4]. From the X-ray [5] and the neutron diffraction patterns [3] it has been deduced that the atoms in the melt prefer unlike atoms as nearest neighbors. This short-range order is accompanied by a reduction of the distance between unlike atoms as compared with the mean distance of the pure components. In this paper it will be shown that also the volume per atom, the compressibility, and the corresponding temperature coefficients show strong deviations from a linear dependence with respect to composition.

The density and the compressibility data have been measured primarily in order to be able to evaluate quantitatively the above mentioned neutron scattering data. But it will be shown below that the composition dependence of the volume per atom leads to an additional confirmation of the ideas about the structure which have been deduced from the scattering experiments [3]. The density and sound velocity data of the pure components quoted in the literature are summarized together with the values obtained in this work in table 1 and 3, respectively. The only available data for the liquid alloys were the density and its temperature coefficient for the alloy with 62 at % Pb [6], Figure 2.

Experimental Details and Results

The samples were prepared from 4 N Pb and 3 N 5 Li and investigated inside a VAC glove box in an atmosphere of continuously purified argon. The crucibles and the other parts of the apparatus which came into contact with the liquid alloys were made from AME iron and from Remanit which do not react with the melts.

The density ϱ was measured using the maximum bubble pressure technique [7]. The bubbling tube had 4 mm i. d. The emerging end of the tube was bevelled at 45° and polished. The bubble frequency was about $1/30\ \text{s}^{-1}$. The pressure was measured using a quartz spiral manometer with a continuous pressure vs. time registration. At each temperature the maximum bubble pressure was determined with the bubbling tube in an arbitrary lowest position and at three levels 10, 15 and 20 mm higher than the lowest one. Reproducible maximum bubble pressures were obtained only when the melt wetted the bubbling tube. Therefore, prior to a measurement the melts were heated to the highest attainable temperature and the density vs. temperature diagrams, Fig. 1, were then established for stepwise cooling of the samples. The values relative to liquid lead were difficult to reproduce because of bad wetting. Vibrations of the glove box were only of minor influence.

The variation of the molar volume at 600 °C and of the thermal expansion coefficient with composition are depicted in Figure 2. In the dotted region the temperature of the liquidus curve is higher than 600 °C. The points in this composition range were obtained by extrapolation. Between 17 and 30 at % Pb no measurements could be performed because of

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Table 1a. Literature data on the density of liquid lithium (arranged in chronological order).

Table 1b. Literature data on the density of liquid lead published since 1960 [d] (arranged in chronological order).

Authors	Year	Temperature range (°C)	Density (g/cm³) at m.p.	$-\mathrm{d}\varrho/\mathrm{d}T \times 10^4$	Method used
Been [a]	1950	262 – 997	0.509	0.83	MBP [c]
Tepper [a]	1965	347 – 1040	0.518	0.99	Dilatometer
Cooke [a]	—	332 – 1007	0.521	1.03	MBP [c]
Gol'tsova [25]	1966	300 – 1600	0.517 ± 0.002	1.01	Pycnometer
Shpil'rain et al. [26]	1967	400 – 1125	0.518 ± 0.001	1.02	Pycnometer
Achener [b]	1968	500 – 740	0.522	1.17	—
Novikov et al. [27]	1969	300 – 1000	0.518 ± 0.001	1.04	Pycnometer
Dillon [24]	1971	483 – 663	0.593	3.83	γ-attenuation
This paper	1975	248 – 650	0.514 ± 0.003	1.1 ± 0.1	MBP [c]
Strauss et al. [29]	1960	335 – 807	10.683 ± 0.01	12.53	Archimedes
Kirshenbaum et al. [28]	1961	357 – 1700	10.674 ± 0.01	13.17 ± 0.15	Archimedes
Hesson et al. [14]	1968	373 – 739	10.66 ± 0.03	12.0 ± 0.3	Dilatometer
Thresh et al. [30]	1968	340 – 530	10.660 ± 0.005	12.22	Pycnometer
Lucas [31]	1971	330 – 1550	10.665 ± 0.001	12.64	Archimedes
This paper	1975	363 – 556	10.66 ± 0.06	13.5 ± 2	MBP [c]

[a] Drawn from a graph of Davison's paper [23].
[b] Drawn from a graph of Dillon's paper [24].
[c] Maximum bubble pressure.
[d] A compilation of former data is given by Kirshenbaum [28].

Table 2. Density of liquid lithium lead alloys.

Composition (at % Pb)	Temperature range (°C)	Liquidus temperature (°C) (Hansen and Anderko [42])	Density (g/cm³) at the liquidus temperature	$-\mathrm{d}\varrho/\mathrm{d}T \times 10^4$
0	248 – 650	180	0.514 ± 0.003	1.1 ± 0.1
1	424 – 664	180 (365 ± 20)*	0.658 ± 0.003	1.1 ± 0.1
2	481 – 702	340 (420 ± 20)*	0.787 ± 0.003	1.4 ± 0.2
3	496 – 706	420 (445 ± 20)*	0.929 ± 0.004	1.4 ± 0.2
5	528 – 712	480	1.221 ± 0.005	2.1 ± 0.3
7	547 – 721	515	1.52 ± 0.01	2.1 ± 0.3
10	582 – 742	550	1.98 ± 0.01	2.8 ± 0.4
15	648 – 763	620	2.79 ± 0.01	5.1 ± 0.5
17	669 – 772	640	3.13 ± 0.02	6.6 ± 0.5
30	663 – 777	642	4.86 ± 0.02	7.2 ± 0.5
40	520 – 672	470	6.08 ± 0.02	7.0 ± 0.5
50	497 – 692	482	6.97 ± 0.03	6.4 ± 1
60	465 – 684	430	7.82 ± 0.03	7.6 ± 1
70	533 – 644	350	8.65 ± 0.03	7.8 ± 1
80	491 – 653	260	9.33 ± 0.04	7.0 ± 1
100	363 – 556	327	10.66 ± 0.06	13.5 ± 2

* Inflection point in the ϱ and u vs. T diagrams.

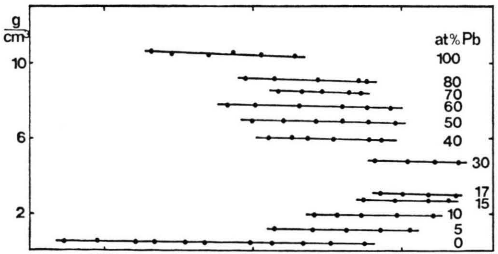

Fig. 1. Density vs. temperature diagrams of liquid Li-Pb alloys. The curves for $x_{\mathrm{Pb}} = 0.01$, 0.02, 0.03 and 0.07 have been omitted for the sake of clarity.

the high melting point of these alloys. Table 2 summarizes the data obtained with the different samples. For the alloys with high Li content an inflection point has been observed in the ϱ and u vs. T diagrams at temperatures well above the liquidus line published by Hansen and Anderko [42] indicating that the latter might be erroneous.

The velocity of sound (u) at 4 MHz has been measured interferometrically using the pulse-echo technique. This technique has first been described by Pochapsky [8] and it has been applied with much success in our laboratory by Seemann and coworkers who studied the compressibility of Al, Hg [9], Sn, Pb, Bi[10], Mg, Zn, Cd, Sb, Zn-Pb and of Sn-Pb[11]. In order to measure the velocity of sound of the Li-Pb alloys, the bubbling tube of the above described density apparatus was replaced by a steel rod which transmitted both, the primary pulse to the melt and the delayed echo pulse back to the quartz crystal, the bottom of the crucible serving as reflector. Here again, the measurements were carried out on cooling the samples in steps of about 40 K. At each temperature measurements were performed first with an arbitrary smallest distance between the steel rod and the bottom of the crucible and then with the steel rod in four higher levels, 10, 15, 20 and 25 mm above the lowest one. For each level the distance between transducer and reflector of a comparative water delay line was changed by a micrometric

Table 3 a. Literature data on the velocity of ultrasound in liquid lithium.

Authors	Year	Temperature range (°C)	Velocity at m.p. (m/s)	$-du/dT$ $\left(\frac{m}{s}/°C\right)$
Novikov et al.[41]	1969	227−827	4517±20	0.591
This paper	1975	221−528	4525±25	0.56±0.1

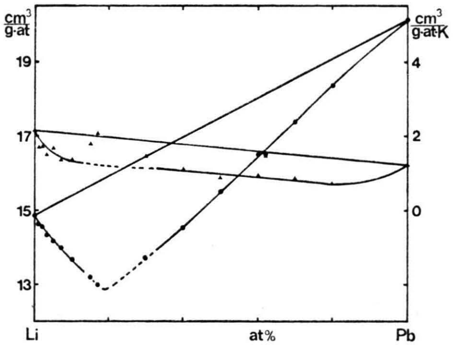

Fig. 2. Composition dependence of the volume per g atom at 600 °C (●——●) and its relative temperature derivative $(1/v_a)\,dv_a/dT$ (▲——▲). At 600 °C in the dotted region of the diagrams the alloys are not liquid. The values at 62 at % Pb are calculated from the data of Schneider et al.[6]: $v_a = 16.5$ cm³/g·at, $dv_a/dT = 1.5 \times 10^4\, v_a$.

Table 3 b. Literature data on the velocity of ultrasound in liquid lead.

Authors	Year	Temperature range (°C)	Velocity at m.p. (m/s)	$-du/dT$ $\left(\frac{m}{s}/°C\right)$
Jacob[32]	1939	340−450	1375	0.2 **
Kleppa[33]	1950	T_M−380	1790±15	0.5
Gordon[34]	1959	T_M−370	1776±4	0.277
Polotskii et al.[35]	1959	340	1838±6	—
Khodov[36]	1960	344	1831	0.3
Pronin et al.[37]	1963	T_M−930	1810	0.381
Plass[38]	1963	340	1764	—
Nagel[11]	1966	T_M−600	1816±4	0.273
Gitis et al.[39]	1966	T_M−1120	1820±2	0.31 (T_M−900) 0.4 (T_M−1120)
Speicher[10]	1968	T_M−360	1815±0.1	0.364
Konyuchenko[40]	1972	T_M−700	1823±5	0.325
This paper	1975	367−654	1820±5 *	0.28±0.03

* Value extrapolated with the temperature coefficient of 0.28±0.03.
** Drawn from a diagram.

Table 4. Velocity of ultrasound in liquid lithium-lead alloys.

Composition (at % Pb)	Temperature range (°C)	Liquidus temperature (°C) (Hansen and Anderko[42])	Velocity (m/s) at the liquidus temperature	$-du/dT$ $\left(\frac{m}{s}/°C\right)$
0	221−528	180	4525±25	0.56±0.1
1	421−590	180	3999±20	0.51±0.1
2	429−609	340	3556±14	0.41±0.05
3	463−724	420	3273±10	0.41±0.05
5	509−707	480	2883± 9	0.38±0.03
10	563−727	550	2329± 7	0.29±0.02
40	510−701	470	1820± 6	0.29±0.02
50	503−670	482	1770± 5	0.27±0.02
60	483−664	430	1750± 5	0.25±0.01
70	413−636	350	1770± 5	0.28±0.02
100	367−654	327	1820± 5	0.28±0.03

screw in order to superpose the echo signals from the two delay lines which were both displayed on an oscilloscope [12]. From corresponding distance variations of the two delay lines and from the known sound velocity of distilled water, the sound velocity u of the melt was calculated. The results are shown in Fig. 3 and summarized in Table 4. The adiabatic compressibility (χ_{ad}) obtained from $\chi_{\text{ad}} = (\varrho \cdot u^2)^{-1}$, and $d\chi/dT$ are shown in Figure 4.

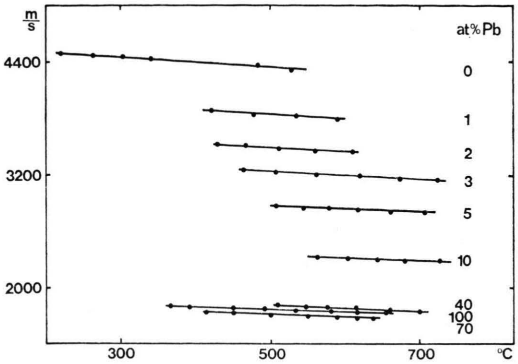

Fig. 3. Sound velocity vs. temperature diagrams of liquid Li-Pb alloys. The curves for $x_{\text{Pb}} = 0.5$ and 0.6 are omitted for the sake of clarity. They are between the curves for $x_{\text{Pb}} = 1$ and 0.7.

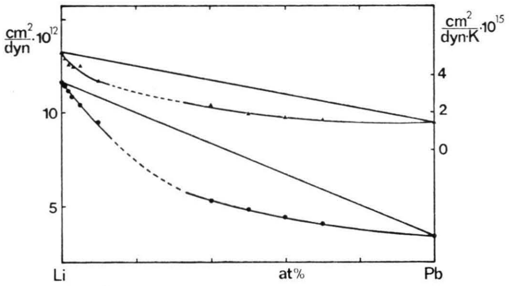

Fig. 4. Composition dependence of the adiabatic compressibility χ_{ad} at 600 °C (●——●) and its temperature derivative $d\chi_{\text{ad}}/dT$ (▲——▲). At 600 °C in the dotted region of the diagrams, the alloys are not liquid.

Discussion

The volumes per atom, v_{a}, which are depicted in Fig. 2 are smaller than the corresponding volumes v_{i} of an ideal solution. The excess volume $V^{\text{xs}} = v_{\text{a}} - v_{\text{i}}$ is thus negative and has a minimum at about 20 at % Pb. As was mentioned above at this composition also other properties of liquid Li-Pb alloys show anomalous behavior. The $v_{\text{a}}(x)$ curve interpolated between $x_{\text{Pb}} = 0.17$ and 0.30 implies a maximal value of $|V^{\text{xs}}/v_{\text{i}}|$ of about 18%. With rising temperature $|V^{\text{xs}}|$ increases. This can be seen from the concentration dependence of the thermal expansion coefficient in Figure 2. In order to diminish $|V^{\text{xs}}|$ with rising temperature, the expansion coefficients of the alloys should lie above the line connecting the points appropriate for the pure constituents, which is not the case. The very large volume contraction and its temperature dependence seem to be unusual [13], but the same behavior has been observed for liquid Na-Pb (17% contraction) [13], Na-Bi (17%) [13], Na-Hg (18%) [14] and Na-In (10 to 12%) [15]. And also for these alloys $|V^{\text{xs}}|$ does not decrease with rising temperature. According to Mc Alister [14] who studied the Na-Hg alloys, the abnormal temperature dependence of V^{xs} seems to be incompatible with tendency towards compound formation. For the Li-Pb alloys, however, the diffraction experiments indicate unambigously a preference for unlike nearest neighbors, and this tendency towards compound formation diminishes with rising temperature. Thus, the absolute value of V^{xs} increases in spite of a decrease of short-range order. There seems to be no direct relation between short-range order and excess volume. In fact, the volume contraction is probably not due to a structural rearrangement and a denser packing of the atoms but simply to a decrease of the Li-Pb interatomic distance [3]. The latter effect, evidently, is a consequence of a rearrangement of the electronic states, which on the other hand leads to special bonding effects and which is in that way also responsible for the short-range order, but via a different mechanism.

A comparison of the volume contractions of liquid and solid Li-Pb alloys leads to additional information about the constitution of the liquids and it shows that the behavior of the liquid Li-Pb alloys is far from exceptional; an analogous behavior is to be expected for any alloy of Li with a more electropositive metal! In the system Li-Pb five solid intermetallic phases exist which according to Zalkin and Ramsey [16] are characterized by three common properties: 1. They have almost the same over-all structure which is bcc as it is for solid lithium. 2. The appropriate number of Li atoms is substituted by Pb atoms in such a way that a direct contact between Pb atoms is almost prevented. There is thus a preference for unlike nearest neighbors. 3. The formation of the compounds leads to a substantial reduction in volume as compared with the sum of the volumes of the pure components, as is demonstrated in Figure 5. According to Nowotny and Dehlinger[17] items 2 and 3 are due to a transfer of charge from the Li atoms towards the Pb atoms which renders the bonding partially salt like and which diminishes the size of the Li atoms. Besides a shift due to the melting volume and to the higher temperature, the v_a vs. composition diagram of the liquid phase in Fig. 2 is almost identical to the curve connecting the points which represents the atomic volumes of the solid interatomic compounds in Figure 5. The cause for the anomalous behavior of the liquid and the solid alloys will be the same.

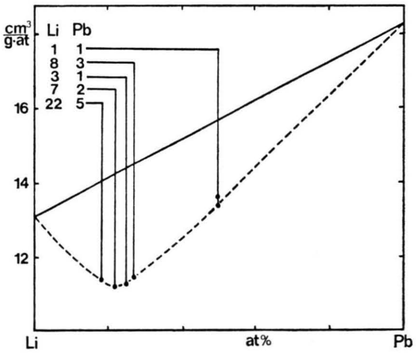

Fig. 5. Volume per g·atom of bcc Li, fcc Pb and of the five intermetallic compounds, Li_mPb_n, according to Zalkin and Ramsey[16].

From a study of v_a of many intermetallic compounds, Biltz and Weibke[18] deduced the empirical rule of "additive constant increments" which has recently been confirmed by Thümmel and Klemm[19]. According to this rule, a substantial volume contraction, the so called "chemical compression", occurs during the formation of binary intermetallic compounds from a group I A or II A metal with a more electropositive metal. And, starting from the pure electropositive metal, v_a varies linearily with composition in a range of at least 30 to 50 at %. Thus, in the case of Hg-Li $v_a = x_{Hg}\,\bar{v}_{Hg} + (1 - x_{Hg})\,\bar{v}_{Li}$ and the partial molar volumes $\bar{v}_{Hg}$ and $\bar{v}_{Li}$ have almost the same value for the four intermetallic compounds in this system. Therefore, $\bar{v}_{Hg}$ and $\bar{v}_{Li}$ are called "increments" of Hg and Li, respectively. It has been observed that the increment of the more noble metal is almost equal to the atomic volume of this metal, whereas the increment of the less noble metal is significantly smaller than the volume per atom of the pure metal. Thus, for Hg-Li $\bar{v}_{Hg} = 12.8$ and $\bar{v}_{Li} = 8.2$ cm^3 whereas the volumes of the pure constituents amount to 13.8 and 12.6 cm^3 for Hg and Li, respectively. The most astonishing property of the increments is that they are nearly independent of the type and especially of the valency of the partner. Thus, for compounds of Li the following values for $\bar{v}_{Li}$ have been observed: 9.0 (Ag), 9.1 (Zn), 9.6 (Cd), 8.2 (Hg), 8.5 (Ga) and 8.1 cm^3 (Tl)[18]. Biltz and Weibke assume that the increments correspond to the real volumes of the atoms in the compounds and that the Li atoms in the aforementioned compounds occupy only 65 to 75% of their original volume in pure Li.

For solid Li-Pb alloys a linear variation of v_a can hardly be demonstrated from the curve of Fig. 5 because of the lack of compounds with high lead content. But an extrapolation of the curve from pure lead through the points at 50, 27 and 25 at % Pb leads to a value of $\bar{v}_{Li}$ of about 8.5 to 9 which fits very well into the scheme of additive constant increments. For the liquids we observe from Fig. 2 an almost linear variation of v_a on both sides of the diagram which means that in these concentration ranges the partial molar volumes are almost constant. According to Biltz and Weibke[18] on the Pb side of the diagram the partial molar volumes have a real physical signification. The Li atoms are dissolved in Pb with a reduced size corresponding to a volume per atom of about 10.8 cm^3 compared with the 14.8 cm^3 of pure Li at this temperature. This volume contraction of 27% corresponds to a reduction of the most probable distance between nearest Li neighbors from 3 Å in pure liquid Li to about 2.8 Å in the alloys. The Pb atoms on the other hand retain their size, which is exactly what has been observed by X-ray diffraction[5]. At the Li side of the diagram the partial molar volumes have no immediate meaning. The strong reduction of the volume per atom if Pb is added to Li should be related to an increasing size reduction of the Li atoms. A linear variation of v_a with composition has also been observed for liquid Na-Pb alloys in the range from $x_{Na} = 0$ to at least 0.5[13]. The corresponding volume contraction of the sodium atoms amounts to 25% which is the same value as that for solid Na-Pb compounds[18]. For liquid Na-Bi[13] and Na-Hg[14], however, the rule of Biltz and Weibke seems not to hold.

The problem of calculating theoretically atomic dimensions in solid alloys has recently attracted the interest of many authors. According to Magnaterra and Mezzetti [20], starting from the free electron model, good agreement with experiments has been obtained for mono- and divalent components. However, results in agreement with experiments are very hardly obtained when one of the components has a higher number of electrons because some of the electrons remain preferably near their own ion to screen the strong ionic charge. From an extension of pseudopotential theory, v. d. Broek [21] calculated the volume changes of several binary systems, Li-Pb included. The curves fit very well to the experimental data, though smooth curves instead of two linear regions have been obtained. A theoretical confirmation of the rule of additive constant increments is still needed.

The compressibility vs. concentration diagram is shown in Figure 4. As in the preceding case the strong deviation from linearity rises with temperature. Actually, there seems to be no theory to calculate the compressibility in our case [22]. However, from the existing theories it may qualitatively be deduced that a rearrangement of the electronic states is responsible for the observed effects.

Acknowledgements

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