Journal of the Less-Common Metals, 32 (1973) 275-278

HIGH-PRESSURE SYNTHESIS OF LUTETIUM TRILEAD

KARL MILLER and H. TRACY HALL
Department of Chemical Engineering, Brigham Young University, Provo, Utah 84601 (U.S.A.)
(Received February 12, 1973)

SUMMARY

The compound $LuPb_3$ was synthesized at pressures of 67 to 88 kbar concurrent with temperatures of 700 to 1100°C. The $LuPb_3$ is face-centered cubic in structure ($AuCu_3$ type) with a cell parameter of 4.786 Å. The $LuPb_3$ could not be prepared at room pressure, but the previously indexed trilead compounds of Tb, Ho, Er, and Tm could be prepared by either room pressure or high pressure techniques. The rare earth-trilead compound series is now complete.

INTRODUCTION

The rare earth-trilead compounds have all been synthesized with the exception of $LuPb_3$[1,2,3,4,5] for which only negative results are listed[5,6]. The compound series is isostructural, with each compound exhibiting an $AuCu_3$ type face-centered cubic structure.

All the work cited above was carried out using standard vacuum techniques. For this study pressures up to 90 kilobars and temperatures to 1100°C were used by employing a tetrahedral anvil apparatus.

EXPERIMENTAL SECTION

The lutetiurn metal used for this study was 99.5% pure (ingot form) supplied by Research Chemicals, Phoenix, Ariz. The lead metal was 99.999% pure (ingot form) and was obtained from Alfa Inorganics, Beverly, Mass.

Experimental procedures, sample geometries and run techniques have been described previously[7]. Sample identification was accomplished using a Debye-Scherrer X-ray camera with nickel-filtered copper X-irradiation (λk_α = 1.5418 Å). $LuPb_3$ was indexed by using the $PrPb_3$ indexing given by Rossi[3]. The lattice parameter was calculated by the least-squares method.

RESULTS AND DISCUSSION

The trilead compounds of Tb, Ho, Er, and Tm were synthesized during this study using vacuum and high pressure techniques. The lattice parameters, existence of metallic lead in equilibrium with the compounds, and the decomposition of the compounds in the presence of air were independent of the method of synthesis used. The lattice parameters obtained and the presence of the metallic lead are in agreement with the work of Kuzma[5]. The decomposition rate in air was similar to that reported for the rare earth-tritin compounds[7].

The compound $LuPb_3$ could not be synthesized using vacuum techniques. This behavior has been previously reported in the literature[5,6]. However, using the tetrahedral anvil apparatus the compound could be synthesized when a minimum temperature of 750°C and a minimum pressure of 67 kbar were reached or exceeded (Fig. 1). Below the minimum pressure unidentified reaction products were observed. The unidentified reaction products gave complex and often non-reproducible X-ray patterns which could not be indexed.

The $LuPb_3$ material was indexed using the indexing given by Rossi ($AuCu_3$ type f.c.c. with space group *Fm3m*). The *d*-values, intensities, and Miller indices are listed in Table I. The cell parameter of $LuPb_3$, calculated by the method of least squares, is 4.786 ± 0.003 Å with an X-ray density of 12.062g/cm^3.

The $LuPb_3$ was always found to be associated with metallic lead, and decomposed in the air within one to two weeks.

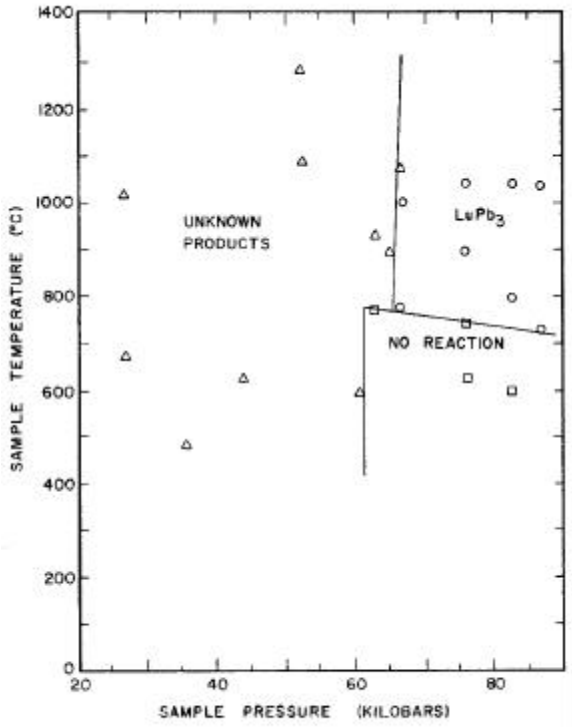

Fig. 1. Lu + 3Pb reaction product diagram.

Figure 2 illustrates the strong correlation between the cell parameters of the rare earth-trilead compounds and the ionic (3 +) radii of the rare-earth elements. The curve is smooth except for Eu and Yb, which differ from the other rare earths because of their potential divalency[8], and Y which is not a true rare-earth metal.

TABLE I

X-RAY DIFFRACTION DATA FOR $LuPb_3$

hkl	$I_{obs.}$	$I_{calc.}$	$d_{obs.}$	$d_{calc.}$
111	100	29	2.748	2.764
200	70	20	2.389	2.394
220	80	26	1.687	1.693
311	80	39	1.441	1.443
222	50	12	1.381	1.382
400	10	7	1.197	1.197
331	40	25	1.095	1.098
420	50	24	1.073	1.070
422	70	24	0.977	0.977
511, 333	70	35	0.922	0.921
440	*	18	*	0.846
531	*	100	*	0.809
442, 600	*	75	*	0.798

* Not observed.

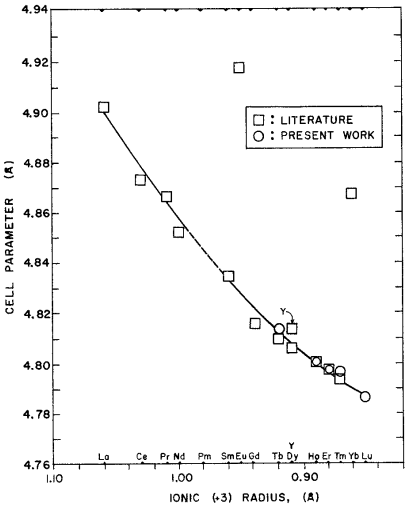

Fig. 2. Cell parameters of $R.E.Pb_3$ compounds as a function of the ionic (+3) radii of the rare-earth elements. The literature cell parameter values were obtained from the references listed in the Introduction section of this paper.

ACKNOWLEDGEMENTS

Assistance rendered by John Cannon, N. L. Eatough, James Hoen, M. D. Horton, L. Merrill, and Alan Webb is gratefully acknowledged. This research was sponsored by the U. S. Army Research Office (Durham) and the National Science Foundation.

REFERENCES

[1] A. Rossi, *Atti Accad. Lincei*, 17 (1933) 839.
[2] E. Zintl and E. Neumayr, *Z. Electrochem*., 39 (1933) 86.
[3] A. Rossi, *Gazz. Chim. Ital*., 64 (1934) 832.
[4] A. Iandelli, The Physical Chemistry of Metallic Solutions and Intermetallic Compounds, *Nat. Phys. Lab. Symp., 1958*, H. M. Stationery Office, London, 1959.
[5] Y. B. Kuzma, R. V. Skolozdra and V. Y. Markiv, *Dopovidi Akad. Nauk Ukr. S.S.S.R*., 8 (1964) 1070.
[6] O. D. McMasters and K. A. Gschneidner, *J. Less-Common Metals*, 19 (1969) 337.
[7] K. Miller and H. T. Hall, *Inorg. Chem*., 11 (1972) 1188.
[8] A. Palenzona, *J. Less-Common Metals*, 10 (1966) 290.