SHORT COMMUNICATIONS

RECRYSTALLIZATION OF LEAD OXIDE FROM MOLTEN LEAD*

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During an investigation of the surface tension of liquid lead by means of a sessile drop technique, small transparent yellow crystals were observed to form on the solidified surface after heating at 750 °C in an argon atmosphere. X-Ray crystallographic analysis, using a 9 cm Guinier camera, confirmed the tetragonal structure and lattice dimensions of yellow lead oxide. It seemed probable that the oxide had been formed from small amounts of oxygen in the cylinder argon used in these experiments. A more systematic study of this crystallization process was subsequently carried out *in vacuo*, using weighed additions of A.R. lead oxide. This note describes the recrystallization process observed.

Pure lead specimens (99·999%; supplied by the Metallurgy Department, University of Melbourne) consisting of 3·5 by 6·5 mm cylindrical pellets, were placed on specially prepared high density uranium dioxide plaques ($UO_{2\cdot00}$), and a weighed amount of A.R. lead oxide placed on the pellet. The assembly was inserted into an induction heated tube furnace which was then evacuated and heated to 750 °C. Photographs of the profile of the sessile drop were taken at different temperatures and time intervals. After each run, the plaque and lead surface were examined microscopically (×300) and photomicrographs were taken when necessary.

The process of solution of lead oxide was accompanied by random motion of the particles over the surface of the lead drop, in much the same manner as small particles of camphor move on a water surface. This effect was observed to occur for lead sulphide, lead selenide, and lead telluride also, although in these cases formation of surface crystals did not occur, a result probably associated with greater solubility in the lead. Plate 1 shows three photographs (Figs. 1–3) of the solution process with lead oxide, the motion of the particles producing a blurred image in Figure 2, for which a 60 sec exposure was used. The lowest temperature at which appreciable solution was observed lies in the range 500 to 530 °C for each of the four solutes.

The rate of cooling has some influence on the formation of the crystals. Plate 1, Figure 4, is a photomicrograph of a rapidly cooled sample, and it will be noted that the lead oxide crystal is irregular in shape and appears to have been formed by growth from a crack in the initially formed lead " crust ". Plate 1, Figure 5, on the other hand, shows some of the more regular lead oxide crystals produced by the slower cooling obtained in a furnace of larger heat capacity. These crystals were coloured red (probably an interference colour).

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The approximate maximum solubility of lead oxide in lead at 750 °C was estimated to be 0·019 molal (0·42 wt.%), which was inferred from the surface tension *v.* concentration isotherm (Bradhurst and Buchanan 1959, unpublished data), which showed maximum depression at this concentration. The value obtained by Richardson and Webb* was somewhat smaller (saturation at an interpolated value of 0·012 molal).

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