JOURNAL OF MATERIALS SCIENCE 27 (1992) 1008–1010

# Temperature dependence of electrical resistivity and microstructure of Pb–Cd alloys

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The electrical resistivity of Pb–Cd alloys has been studied at various ageing temperatures, for various ageing times. The variations of microstructure with ageing temperature were investigated by scanning and transmission electron microscopy. In addition, the process of coarsening of Pb and Cd ($\alpha$ and $\beta$ phases) at different ageing temperatures was studied by the microanalysis technique.

## 1. Introduction

The electrical resistivity of a conducting solid can be experimentally determined without much difficulty and for many years it has been used as a research tool to investigate various microstructural and physical phenomena. Gibson and Delamore [1] concluded that at higher growth rates, the microstructure displayed a curious feature which at low magnification appeared to be a separate acicular phase embedded in the cellular substructure. When examined at higher magnifications this feature was seen to consist of isolated pairs of lamellae of the minor phase (Cd-rich phase), randomly distributed and oriented in the plane normal to the growth direction and extending over very large distances. The reasons for the transition from normal cellular growth to the extended lamellar structure are not clear. For ternary eutectics the growth morphologies are more complex than those of binary systems [2]. The ternary structure may consist of rod–lamellar combinations, non-parallel lamellae or more random configurations [3–5].

The aim of the present work is to investigate the effect of isothermal ageing stages in the region of transformation on the electrical resistivities of Pb–2 wt % Cd and Pb–17.4 wt % Cd alloys and correlate them with the microstructure variations.

## 2. Experimental procedure

Pb–2 wt % Cd and Pb–17.4 wt % Cd alloys were prepared from Pb (purity 99.999%) and Cd (purity 99.999%). Resistivity measurements were carried out on wire samples of diameter 0.5 mm and length 200 mm. The samples were annealed at 513 K for 1 h in a vacuum and were rapidly quenched in water at room temperature. These samples were aged at ageing temperatures ranging from 443 to 513 K. During measurement of the electrical resistivity, the samples were mounted on an isolating support. They were never taken away from this support. At each ageing temperature the samples were aged for different ageing times from 1 to 180 min. The electrical resistivity $\rho$ was measured by the four-point method. The resistivity measurements were performed at room temperature. The electrical resistivity $\rho_t$ at ageing temperature $T$ was calculated for each ageing time $t$.

Thin foils of Pb–17.4 wt % Cd (the eutectic composition) aged at temperatures of 453, 483 and 493 K were prepared by the standard thin-foil technique, and investigated by a Jeol 100 kV transmission electron microscope (TEM). Some samples were also investigated by using the scanning electron microscope.

## 3. Results

Fig. 1a shows the electrical resistivity of Pb–2 wt % Cd in the temperature range 443–513 K. From this figure it is clear that there is no change in the resistivity data and the microstructure is stable above 40 min, regardless of the working temperature. Fig. 1b shows the electrical resistivity of Pb–17.4 wt % Cd (the eutectic composition). The activation energy (Fig. 2) represents the formation and dissolution processes of precipitates in each stage for the two alloys. It was calculated from the slope of the linear relation of $\ln\rho_t$ versus $1000/T$ for different times. The values of the calculated activation energy range from 0.035 to 0.10 eV and from 0.05 to 0.075 eV for Pb–2 wt % Cd and Pb–17.4 wt % Cd alloys, respectively. Fig. 3 shows two scanning electron micrographs of the eutectic composition for specimen aged at 483 K for 15 min, at two different magnifications. Fig. 4a and b show the microstructure for specimens aged at 453 and 493 K, respectively, for 10 min. The microanalysis of the $\alpha$ phase (Pb-rich phase) of the eutectic composition was performed by a scanning electron microscope (Philips 505). This microscope was fitted with an energy-dispersive X-ray (EDAX) detector. Table I shows the weight percentage of both Pb and Cd at different isothermal ageing temperatures.


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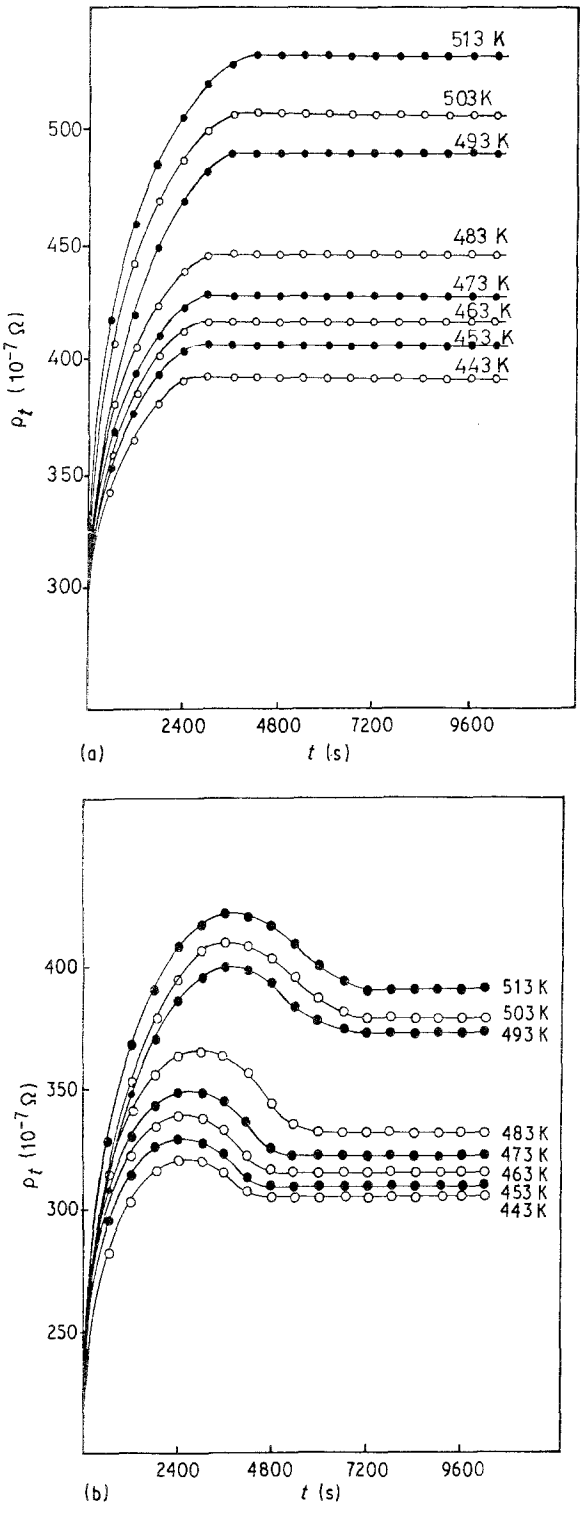


*Figure 1* Ageing temperature dependence of the resistivity change as a function of ageing time at constant ageing temperature: (a) Pb–2 wt % Cd, (b) Pb–17.4 wt % Cd.

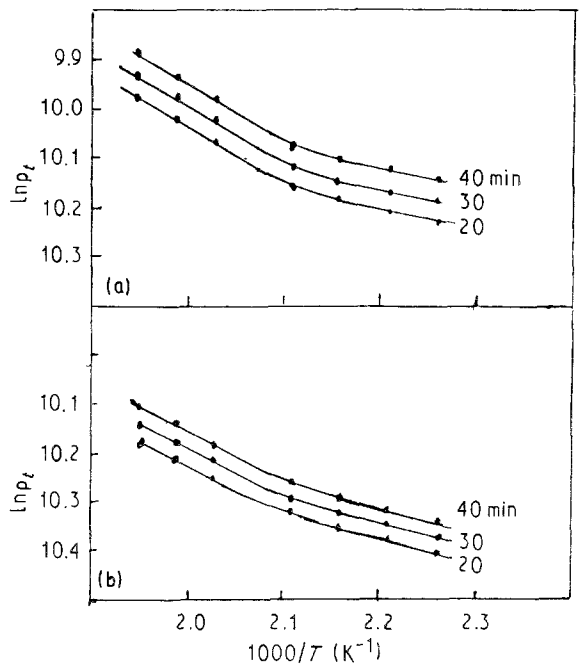


*Figure 2* Ln $\rho_t$ as a function of $1000/T$ at different constant ageing times for (a) Pb–2 wt % Cd, (b) Pb–17.4 wt % Cd.

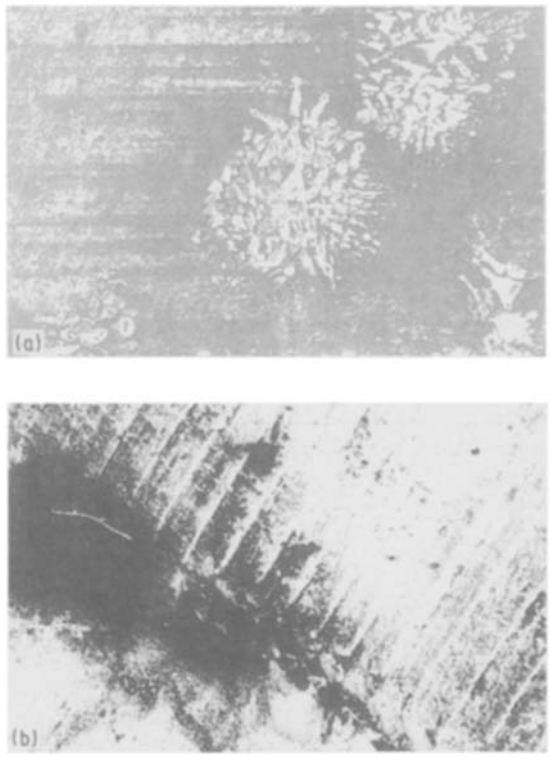


*Figure 3* Microstructures of Pb–17.4 wt % Cd specimen aged at 483 K for 15 min: (a) showing spherical nodules with branched Cd rods (750 ×), (b) showing solid–liquid interface with lamellar substructure (350 ×).

## 4. Discussion

The present study concerned mainly the electrical properties of two Pb–Cd binary alloys, namely, Pb–2 wt % Cd and Pb–17.4 wt % Cd (the eutectic composition). These alloys are widely used in industry. However, the changes of the electrical resistivity of both alloys were found to pass through maximum values depending on the ageing temperature and ageing time (see Fig. 1a and b). In the low- and high-temperature ageing stages (below and above 483 K which is the transition temperature [6, 7]), these peak values of resistivity increased as the ageing temperature was raised. This observation may be related to the relative increase of the number of precipitation particles acting as scattering centres for the conduction electrons, which occurs on increasing the ageing temperature [8]. The quenched vacancies form loops

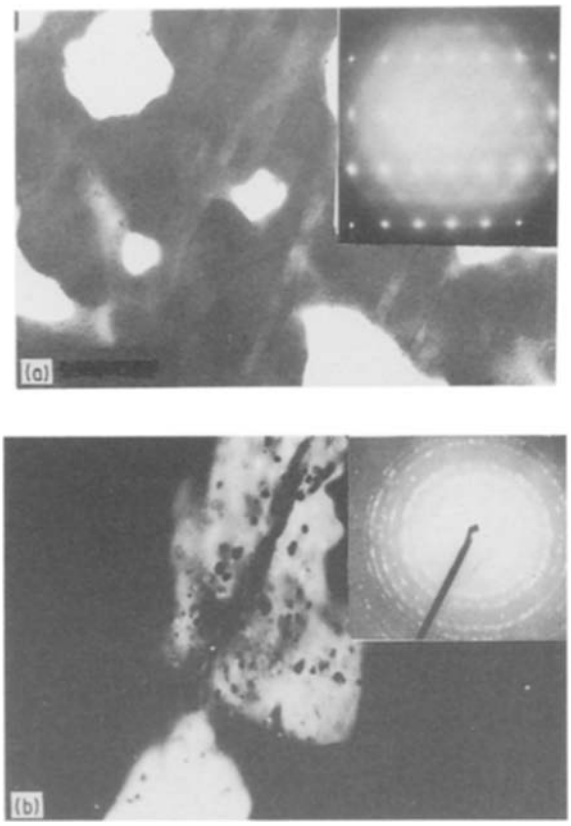

*Figure 4* (a) Electron micrograph of Pb–17.4 wt % Cd specimen aged at 453 K for 10 min showing fine lamellar structure of Cd-rich phase (80 000 ×) with a single-phase diffraction pattern (zone axis [1 0 $\bar{1}$ 2]), (b) Electron micrograph of Pb–17.4 wt % Cd specimen aged at 493 K for 10 min showing the dissolution of the lamellar structure shown in (a) (67 000 ×). Ring diffraction pattern was obtained from the fine precipitates (zone axis [1 1 0]).

TABLE I Cd and Pb concentrations in the α phase of eutectic composition samples aged under different conditions

| Ageing conditions | Cd (wt %) | Pb (wt %) |
|---|---|---|
| Quenched from 513 K, aged at 453 K for 10 min | 43.86 | 56.14 |
| Quenched from 513 K, aged at 483 K for 15 min | 36.39 | 63.61 |
| Quenched from 513 K, aged at 493 K for 10 min | 14.04 | 85.96 |

which are stabilized by cadmium atoms. The number of stabilizing loops can be increased by raising the ageing temperature because the diffusion rate of cadmium increases. These facts may explain the increase of the annealed value of resistivity with increasing ageing temperature.

The activation energies of formation and dissolution of precipitates of the test alloys (see Fig. 2) may be due to the binding energy between a vacancy and cadmium atom.

The sequence of change of microstructure of Pb–14.7 wt % Cd alloy with the change of ageing temperature has been investigated. Fig. 3a shows that spherical nodules are formed in the alloy aged at 483 K for 15 min. The diameter of these nodules can be taken as a function of its growth rate in terms of both ageing temperature and time. The cross-sectional area of the nodule also shows that the scattering centres are relatively big in area, but of course their number is rather small to explain the change of resistivity at this temperature. However, the formation and dissolution of precipitates are presented in Fig. 3b to show that high-purity eutectics solidify with an essentially planar solid–liquid interface. It is also clear from this figure that the interlamellar spacings can also be taken as a function of ageing temperature and vice versa. The extended lamellar substructure of the eutectic specimen aged at 453 K for 10 min (see Fig. 4a) is very fine in its interlamellar spacing, where it has been resolved at high magnification (×80 000). This observation indicates that the interlamellar spacing is very coarse on the specimen surface and gradually decreases into the core structure until it becomes very fine. The diffraction pattern confirms the formation under these ageing conditions of a Cd-rich phase as a single phase. Moreover, Fig. 4b shows the dissolution process with the ring diffraction pattern of a Pb-rich phase.

In addition, Table I shows the microanalysis of the α phase of Pb–17.4 wt % Cd samples for different ageing conditions. The microanalysis data illustrate the processes of formation and dissolution of the Cd-rich phase.

## Acknowledgement

One of the authors (AZM) is grateful to Dr N. Ridley of the Material Science Centre, UMIST, Manchester, England, for his kind invitation and permission to use the Department facilities.

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*Received 19 October 1990*
*and accepted 25 March 1991*