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Anomalous electrical resistive behaviour of Al–O–Pb sputtered alloys

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Application of the sputtering technique to $(\text{Al–O})_x\text{Pb}_{100-x}$ alloys containing an immiscible lead element has been found to result in the formation of a duplex material consisting of fcc lead particles dispersed in an amorphous Al_xO_y oxide matrix. The particle size and interparticle distance of the lead phase were about 10 to 50 nm and 10 to 40 nm, respectively. The Al–O–Pb alloys have been found to exhibit an extremely high electrical resistivity (ϱ), e.g., $4.06 \times 10^6\,\mu\Omega$ cm for $(\text{Al–O})_{87.5}\text{Pb}_{12.5}$ at 273 K, as well as a large positive temperature-dependent resistivity reaching 92% of ϱ_{273}. The peculiar resistivity behaviour was interpreted by assuming the mechanism that only the lead phase embedded in Al_xO_y matrix contributes to electrical conductivity and the mobility of lead electrons is greatly reduced in the intervening oxide region among lead phases. It has thus been demonstrated that the composite material exhibiting peculiar characteristics, which cannot be achieved in metallic composite materials, is obtained by simultaneously sputtering oxide and an insoluble metallic element.

1. Introduction

It is generally known [1–3] that electrical resistivities of metals and alloys are below about $300\,\mu\Omega$ cm in crystalline and noncrystalline states and the temperature dependence changes from positive to negative passing through zero in the vicinity of 150 to $170\,\mu\Omega$ cm. The existence of such general rule suggests that it is very difficult to obtain an alloy exhibiting a high electrical resistivity above $300\,\mu\Omega$ cm combined with a large positive temperature dependence. Most recently, by melt quenching Ge–(M = lead, tin or indium) alloys which are insoluble with each other in solid, the present authors [4] have succeeded in preparing the duplex phase alloys consisting of lead, tin or indium particles dispersed finely in a germanium matrix. Furthermore, the duplex alloys have been found [4] to exhibit large electrical resistivities of 620 to $9860\,\mu\Omega$ cm at 273 K and a large positive temperature dependence leading to a decrease of resistivity of 180 to $1160\,\mu\Omega$ cm at 10 K. The appearance of such anomalous electrical resistive behaviour has been thought [4] to be attributed to the achievement of a peculiar duplex structure consisting of a semiconducting germanium matrix and insoluble metallic particles.

Aluminium and M (M = lead, tin or indium) elements are also insoluble in solid [5], as are Ge–M alloys. Application of vapour quenching by the sputtering method to $(\text{Al}_2\text{O}_3)_{100-x}\text{Pb}_x$ is expected to result in a duplex phase material consisting of a lead phase embedded in an Al_xO_y matrix. Although the Al_xO_y itself is an insulator-type material with an electrical resistivity much higher than that [6] of germanium, the duplex material in an Al–O–Pb system may exhibit a metal-type resistant behaviour combined with a greater temperature-dependent resistivity. This paper intends to examine the vapour-quenched structure and the change in electrical resistivity as a function of temperature and applied field for $(\text{Al–O})_x\text{Pb}_{100-x}$ films prepared by the sputtering technique and to investigate whether or not the Al–O–Pb films exhibit peculiar electrical resistant and magnetoresistant behaviours which are not obtained for metals, alloys, semiconductors and oxides themselves prepared by a conventional solidification process.

2. Experimental procedure

$(\text{Al–O})_x\text{Pb}_{100-x}$ (x = 0, 36.1, 63.9, 87.5 and 92.8%) were sputtered on a water-cooled ceramic substrate into a form of rectangular film with a thickness of 20 to $30\,\mu$m using a radio frequency (r.f.) sputtering apparatus. The Al–O–Pb films for transmission electron microscopic observation were sputtered on a rock salt substrate into a thin foil with a thickness of about 100 nm. The target cathode consists of highly pure Al_2O_3 and pure lead (99.99 wt %) and the composition was controlled by changing the surface area ratio of the two raw materials. After evacuating the sputtering chamber mounted with the target material up to 2×10^{-5} Pa, Ar-gas of 4 Pa is fed through an automatic gas flow controller to make Ar-plasma in the chamber. The Ar-plasma is generated between substrate cathode and stainless steel anode. The anode current usually supplied is 60 mA and the anode voltage is 1 kV. Prior to sputtering the target the substrate is sputtered to clean its surface for 5 min by applying a negative bias against the substrate. The gap between the target and substrate is fixed at 20 mm.

The structure of the sputtered films was examined by X-ray diffractometry and TEM. Measurement of electrical resistivity as a function of temperature and

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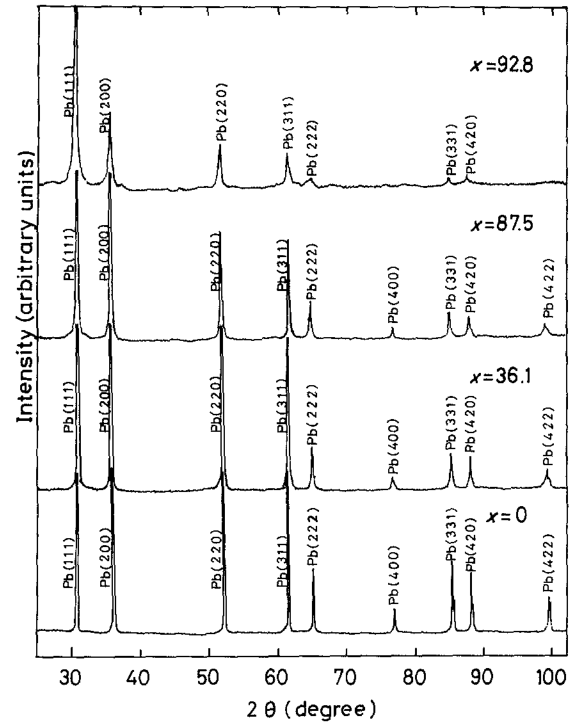

Figure 1 X-ray diffraction patterns of $(Al–O)_xPb_{100-x}$ (x = 0, 36.1, 87.5 and 92.8%) sputtered films.

magnetic field was done using a conventional four-probe technique. The magnetic field up to 8 T was applied transversely to the specimen surface and the excited current. The temperature was measured using a calibrated germanium thermometer at temperatures below 90 K and a calibrated diode thermometer in the higher temperature range with accuracy better than ±0.01 K and ±0.1 K below and above 90 K.

3. Results

3.1. Sputtered structure

Fig. 1 shows the X-ray diffraction patterns of sputtered $(Al–O)_xPb_{100-x}$ (x = 0, 32.9, 87.5 and 92.8%) films. All the patterns can be identified to be only fcc lead and no diffraction peaks corresponding to Al_xO_y oxide are seen. It is thus noticed that the diffraction peaks consist only of a lead phase even for the Al–O rich film only containing 7.2% lead. Although the intensity of lead diffraction peaks tends to decrease with decreasing lead content, there is no systematic variation in the peak position of lead phase with area ratio of lead to Al_2O_3.

In order to examine the reason why no diffraction peaks of Al_xO_y oxide in $(Al–O)_xPb_{100-x}$ films is detected, TEM observation was carried out for a deposited Al_xO_y thin film without lead. Fig. 2 shows a bright-field electron micrograph and a selected area diffraction pattern of sputtered Al_xO_y thin films. No distinct contrast revealing the existence of a crystalline phase is seen in the bright-field image and the diffraction pattern consists only of broad halo rings, indicating that the sputtered Al_xO_y film is composed of an amorphous phase. From this result, it appears reasonable to consider that the structure of $(Al–O)_xPb_{100-x}$ films prepared under the same sputtering conditions consists of amorphous Al_xO_y oxide and the fcc lead phase. The change in the lattice parameter of lead in $(Al–O)_xPb_{100-x}$ films with area ratio of lead to Al_2O_3 in the target is presented in Fig. 3. The lattice parameter remains constant (0.4951 nm) over the whole composition range and agrees with that (0.49502 nm) [7] of pure lead. It is therefore concluded that detectable dissolution of aluminium and/or oxygen into the lead phase does not occur even for the Al_xO_y-rich film containing 7.2 at % Pb, in accordance with the Al–Pb equilibrium phase diagram [5].

Fig. 4 shows the bright-field electron micrographs and selected area diffraction patterns of sputtered $(Al–O)_{87.5}Pb_{12.5}$ and $(Al–O)_{36.1}Pb_{63.9}$ films. The diffraction patterns revealed the coexistence of amorphous Al_xO_y and fcc lead phases for the former film and of fcc lead phase for the latter film, being consistent with the results derived from X-ray diffractometry. The average particle size and interparticle spacing of the lead phase are about 10 to 20 nm and 10 to 20 nm, respectively, for $(Al–O)_{87.5}Pb_{12.5}$ and about 10 to 50 nm and 10 to 40 nm, respectively, for $(Al–O)_{36.1}Pb_{63.9}$. The $(Al–O)_xPb_{100-x}$ films are concluded

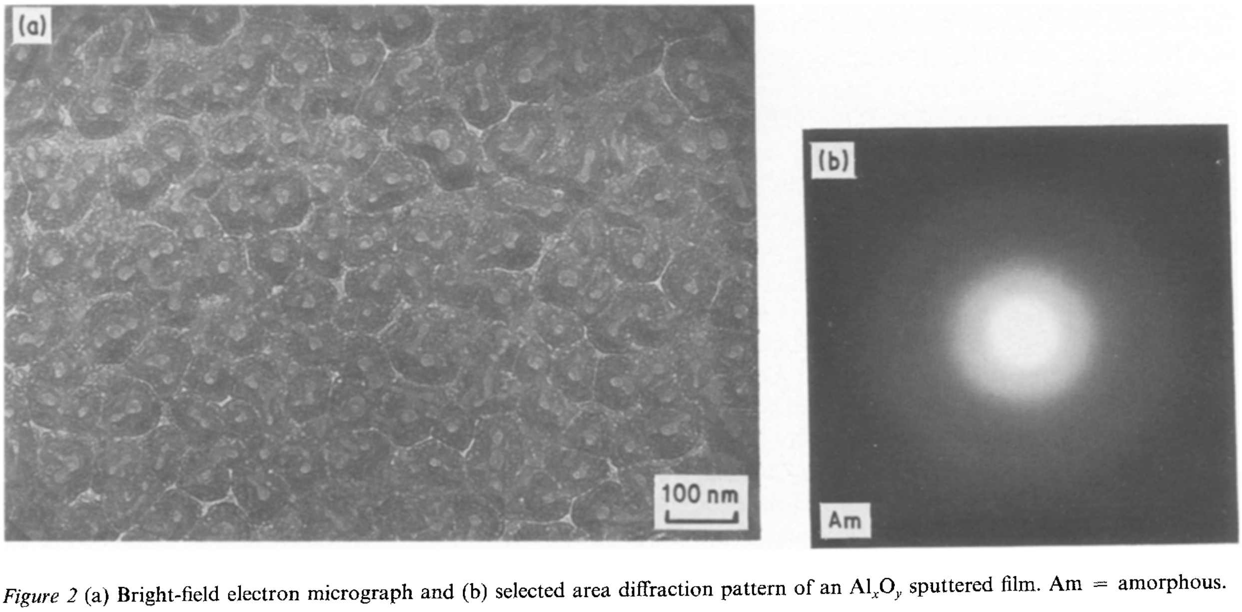

Figure 2 (a) Bright-field electron micrograph and (b) selected area diffraction pattern of an Al_xO_y sputtered film. Am = amorphous.

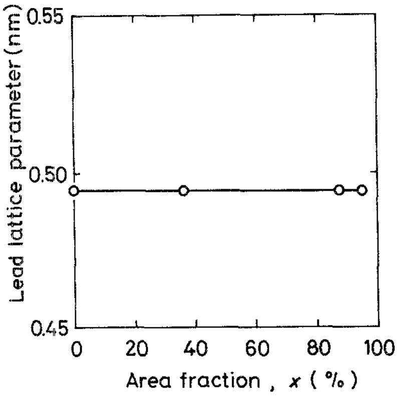

Figure 3 Change in the lattice parameter of lead in $(Al–O)_xPb_{100-x}$ sputtered films with area ratio of Al_2O_3 in sputtered target.

to have a duplex structure consisting of fine fcc lead particles embedded homogeneously in Al_xO_y amorphous oxide.

3.2. Electrical resistivity

Fig. 5 plots the electrical resistivities at 10 and 273 K of sputtered $(Al–O)_xPb_{100-x}$ films as a function of the area ratio of Al_2O_3 to lead in the target. The residual resistivity is 1.6 μΩ cm for pure lead, increases very significantly with increasing area fraction and reaches 1.26×10^7 μΩ cm for $(Al–O)_{92.8}Pb_{7.2}$. The sputtered Al_xO_y amorphous film is confirmed to be an insulator with an ultrahigh resistivity above 10^{15} μΩ cm. Fig. 6 shows the normalized electrical resistant curves (R/R_{273}) as a function of temperature for $(Al–O)_x$-Pb_{100-x} (x = 0, 36.1, 63.9, 87.5 and 92.8%) films. The electrical resistivity of the Al–O–Pb alloys containing more than 12.5% lead shows a large positive temperature dependence and increases significantly with rising temperature from 10 to 273 K, e.g., 3.15×10^5 to 4.06×10^6 μΩ cm for $(Al–O)_{87.5}Pb_{12.5}$, 126 to 2650 μΩ cm for $(Al–O)_{63.9}Pb_{36.1}$ and 5.6 to 165 μΩ cm for $(Al–O)_{36.1}Pb_{63.1}$, while that of $(Al–O)_{92.8}Pb_{7.2}$ shows a much smaller positive temperature dependence leading only to about 13% variation from 1.26×10^7 μΩ cm at 10 K to 1.46×10^7 μΩ cm at 273 K. With further increase in Al–O content, the temperature dependence of electrical resistivity appears to change from positive to negative. The resistivity, ϱ, as a function of temperature, T, varies in the approximate relation of T over the wide temperature range except below about 50 K. It is thus noticed that the sputtered $(Al–O)_{87.5}Pb_{12.5}$ film possesses an extremely high electrical resistivity combined with a large positive temperature-dependent resistivity. To the author's knowledge, this is the first evidence of such peculiar electrical resistant behaviour which has not been found in metallic, semiconducting and oxide materials with a single phase.

Additionally, Fig. 6a shows that all the Al–O–Pb alloys exhibit superconductivity in spite of high residual resistivities. As plotted in Fig. 6b, superconducting transition temperature T_c lies in the range from 7.24 to 7.74 K and remains almost unchanged

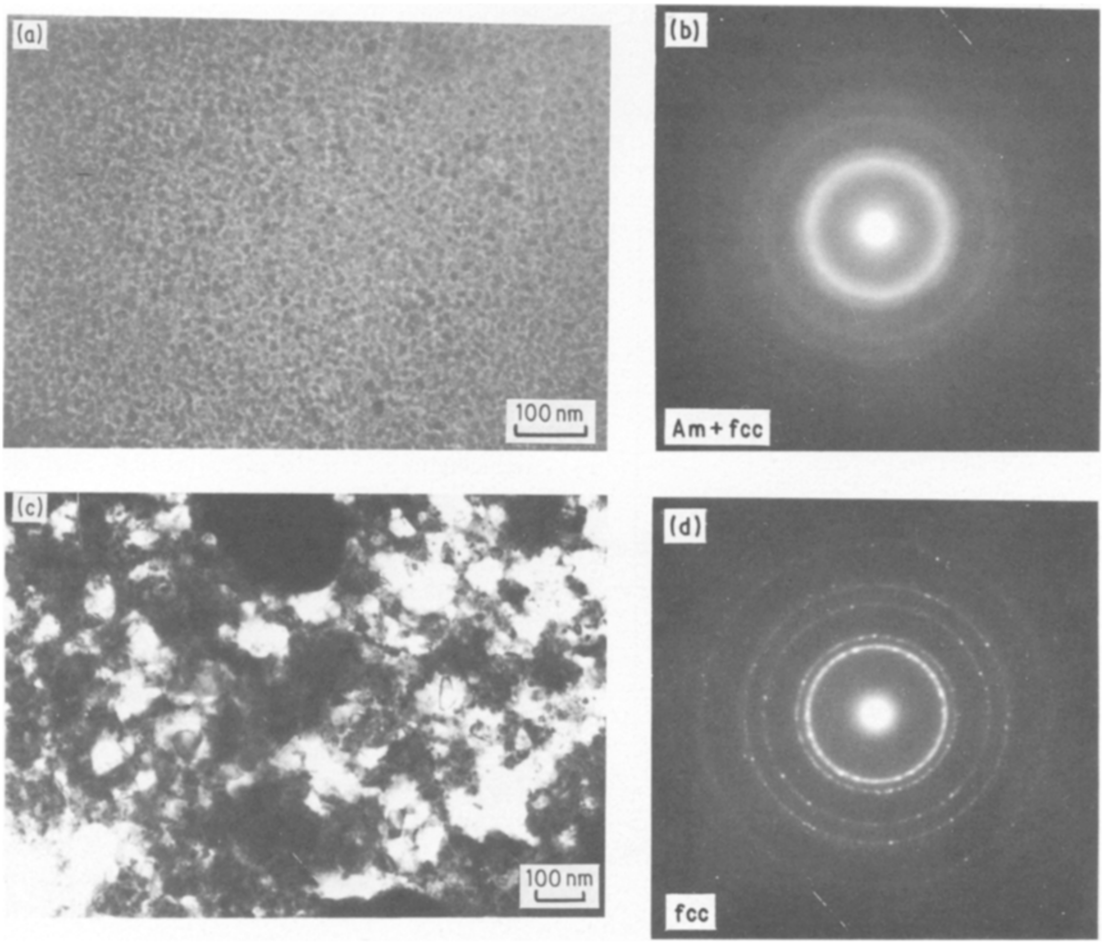

Figure 4 Bright-field electron micrographs and selected area diffraction patterns of (a, b) $(Al–O)_{87.5}Pb_{12.5}$ and (c, d) $(Al–O)_{36.1}Pb_{63.9}$ sputtered films.

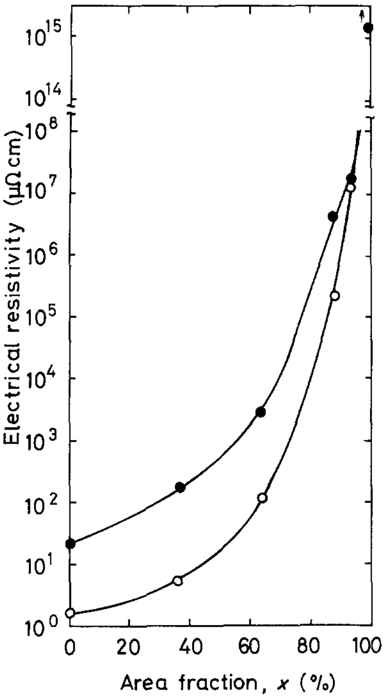

Figure 5 Change in residual electrical resistivity of $(Al\text{–}O)_xPb_{100-x}$ sputtered films with area ratio of Al_2O_3 in target. ●, 273; ○, 10 K.

over the whole concentration range. It is thus noticed that application of sputtering to Al_xO_y oxide containing immiscible lead metal results in a superconductor with high electrical resistivity which is the same level as that of semiconducting materials. The detailed results on the anomalous superconductivity of Al–O–Pb sputtered alloys will be presented elsewhere.

3.3. Magnetoresistivity

Fig. 7 shows the change in electrical resistivity at 10.1 K as a function of transversely applied magnetic field for sputtered $(Al\text{–}O)_xPb_{100-x}$ (x = 63.9, 88.2 and 92.8%) films. It can be seen that the transverse magnetoresistivities of the three alloys are positive and increase with increasing magnetic field in proportion to $H^{1/2}$, in accordance with the tendency [8] for a number of metals and alloys. Here it is particularly noticed that the magnetoresistive change at 7 T is as large as $2.62 \times 10^3\,\mu\Omega$ cm for $(Al\text{–}O)_{87.5}Pb_{12.5}$ and $4.64 \times 10^4\,\mu\Omega$ cm for $(Al\text{–}O)_{92.8}Pb_{7.2}$.

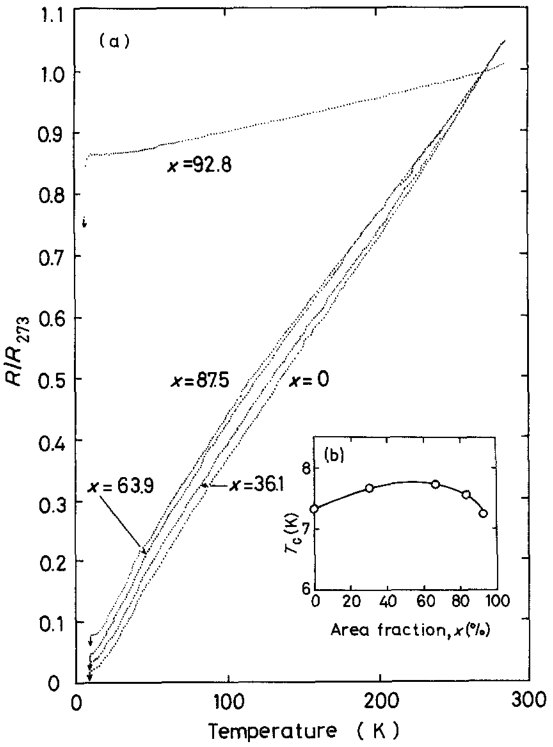

Figure 6 (a) Normalized electrical resistance R/R_{273} as a function of temperature and (b) change in superconducting transition temperature T_c with area ratio of Al_2O_3 in target for $(Al\text{–}O)_xPb_{100-x}$ (x = 0, 36.1, 63.9, 87.5 and 92.8%) sputtered films.

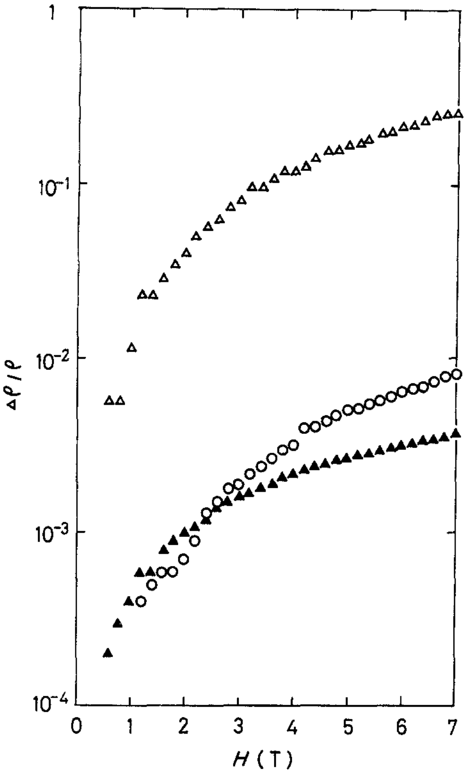

Figure 7 Magnetoresistivity at 10.1 K as a function of applied field for $(Al\text{–}O)_xPb_{100-x}$ (x = 63.9, 88.2 and 92.8%) sputtered films. x = △, 63.9; ○, 88.2; ▲, 92.8.

4. Discussion

It was shown in Section 3 that the sputtered $(Al\text{–}O)_{87.5}Pb_{12.5}$ film exhibits an extremely high electrical resistivity of $4.06 \times 10^6\,\mu\Omega$ cm at 273 K as well as a large positive temperature-dependent resistivity reducing to $3.15 \times 10^5\,\mu\Omega$ cm at 10 K. Such electrical resistive behaviour is a quite peculiar property which has not yet been reported for metallic and oxide materials. This peculiar electrical behaviour, i.e., the simultaneous achievement of high electrical resistivity and large positive temperature-dependent resistivity presumably results from the formation of the duplex structure consisting of an insoluble lead phase embedded finely in amorphous Al_xO_y oxide matrix, because no such resistive behaviour is seen for a single Al–O oxide phase without the insoluble elements. In this subsection the peculiar electrical resistive behaviour for the oxide-based duplex alloys containing a finely embedded lead phase will be discussed in relation to the theories of electrical conductivity of metallic and oxide materials.

The electrical resistivity of non-magnetic metals and alloys (ϱ_T) can be separated into temperature-

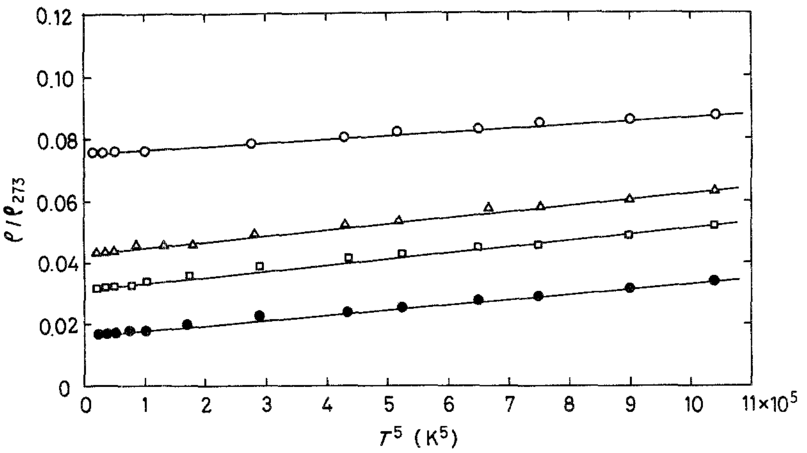

Figure 8 Normalized electrical resistance R/R_{273} as a function of fifth power of temperature (T^5) for $(\mathrm{Al{-}O})_x\mathrm{Pb}_{100-x}$ (x = 0, 36.1, 63.9 and 87.5%) sputtered films. x = ○, 87.5; △, 63.9; □, 36.1; ●, 0.

dependent (ϱ_{iT}) and temperature-independent (ϱ_0) contributions. The resulting equation is generally known as Matthiessen's rule [9];

$$\varrho_{\mathrm{T}} = \varrho_0 + \varrho_{\mathrm{iT}} \qquad (1)$$

The ϱ_0 corresponding to the residual resistivity arises from the scattering of conduction electrons by impurities and/or solute elements in solid solution or by other imperfections present in the lattice. The ϱ_0 value of the sputtered $(\mathrm{Al{-}O})_{87.5}\mathrm{Pb}_{12.5}$ film is as high as $3.15 \times 10^5\,\mu\Omega\,\mathrm{cm}$. From the result (Fig. 1) that the lattice parameter of lead phase is nearly the same as that of pure lead, it is unreasonable to consider that the high ϱ_0 arises from the impurity resistivity due to the very dilute solution of aluminium and/or oxygen into lead. Furthermore, the residual resistivity at 10 K of sputtered pure lead itself is as low as $1.6\,\mu\Omega\,\mathrm{cm}$ for lead as shown in Fig. 5. This indicates that the high ϱ_0 value of $(\mathrm{Al{-}O})_{87.5}\mathrm{Pb}_{12.5}$ film cannot be explained by the increase of the scattering of conduction electrons due to internal defects introduced by vapour-quenching.

On the other hand, ϱ_{iT} is expressed in the following form which is known as the Gruneisen–Bloch relation [10],

$$\varrho_{\mathrm{iT}} = C/M\theta_{\mathrm{R}}(T/\theta_{\mathrm{R}})^5 \int_0^{\theta_{\mathrm{R}}/T} x^5\,\mathrm{d}x/(\mathrm{e}^x - 1)(1 - \mathrm{e}^{-x}) \qquad (2)$$

where M is the atomic weight and C is a constant. θ_{R} is a temperature characteristic of the metal's lattice resistivity in the same way as the Debye characteristic temperature θ_{D} is characteristic of a solid's lattice specific heat. The ϱ_{iT} reduces to $CT/(4M\theta_{\mathrm{R}}^2)$ at higher temperatures ($T > 0.5\,\theta_{\mathrm{D}}$) and to $C'T^5/(M\theta_{\mathrm{R}}^2)$ at lower temperatures ($T < 0.25_{\mathrm{D}}$). As shown in Figs. 6 and 8, the ϱ_{iT} of the sputtered Al–O–Pb films varies in each expected relation of $\varrho_{\mathrm{iT}} \propto T^5$ at temperatures below about 20 K and $\varrho_{\mathrm{iT}} \propto T$ at temperatures above about 50 K. This indicates that the temperature-dependent manner of the present Al–O–Pb films is interpreted based on the general concept of electrical conductivity applicable for non-magnetic metals and alloys, even though the total magnitude of the temperature-dependent resistivity is much larger than that of conventional metals and alloys.

Since the sputtered Al–O–Pb films consist of two phases of amorphous $\mathrm{Al}_x\mathrm{O}_y$ and fcc lead, discussion of the reason for the appearance of the peculiar electrical resistive behaviour is given from the resistivity values of pure $\mathrm{Al}_x\mathrm{O}_y$ and lead phases by using the so-called mixing rule which can be applicable in the case of binary metallic mixtures. If one assumes that there is no correlation between the positions of the two phases and that both the phases are spherical and not needle or disc-shaped, Landauer [11] derived theoretically that the resistivity (ϱ_{m}) and conductivity (σ_{m}) of the mixture are expressed in the following equation

$$\begin{aligned} 1/\varrho_{\mathrm{m}} &= \sigma_{\mathrm{m}} = 1/4\,(\{3x_2 - 1\}\sigma_2 + \{3x_1 - 1\}\sigma_1 \\ &\quad + \{[(3x_2 - 1)\sigma_2 + (3x_1 - 1)\sigma_1]^2 \\ &\quad + 8\sigma_1\sigma_2\}^{1/2}) \end{aligned} \qquad (3)$$

Here x_1 and x_2 are the fractions of the total volume occupied by matrix ($\mathrm{Al}_x\mathrm{O}_y$) and the other second phase (lead), respectively, and σ_1 and σ_2 are the respective conductivities. By using the measured values (83 and $\simeq 10^9\,\mu\Omega\,\mathrm{cm}$) at 273 K for sputtered lead and $\mathrm{Al}_x\mathrm{O}_y$ films, the ϱ_{m} value at 273 K is estimated to be $165\,\mu\Omega\,\mathrm{cm}$ for $(\mathrm{Al{-}O})_{36.1}\mathrm{Pb}_{63.9}$, $2083\,\mu\Omega\,\mathrm{cm}$ for $(\mathrm{Al{-}O})_{63.9}\mathrm{Pb}_{36.1}$ and $2.44 \times 10^9\,\mu\Omega\,\mathrm{cm}$ for $(\mathrm{Al{-}O})_{87.5}$-$\mathrm{Pb}_{12.5}$. The estimated ϱ_{m} values of the Al–O–Pb alloys containing above 33.3% lead are nearly the same as the experimentally obtained ϱ values ($165\,\mu\Omega\,\mathrm{cm}$ and $2650\,\mu\Omega\,\mathrm{cm}$) of $(\mathrm{Al{-}O})_{36.1}\mathrm{Pb}_{63.9}$ and $(\mathrm{Al{-}O})_{63.9}\mathrm{Pb}_{36.1}$ films. However, the ϱ_{m} value evaluated for the Al–O rich film is significantly different from the measured ϱ value ($4.06 \times 10^6\,\mu\Omega\,\mathrm{cm}$). Furthermore, the temperature dependence of resistivity estimated from Equation 3 is negative for $(\mathrm{Al}_x\mathrm{O}_y)_{87.5}\mathrm{Pb}_{12.5}$ films, in contrast to experimental data for the Al–O–Pb films. Such a marked inconsistency for the Al–O rich film indicates clearly that the electrical resistivity higher than $10^4\,\mu\Omega\,\mathrm{cm}$ combined with large positive temperature-dependent resistivity cannot be interpreted only by the mixing rule of electrical resistance which is applicable to the alloys consisting of binary metallic mixtures. The inappropriateness for the highly-resistant material is thought to be due to the result that the metallic phase in the binary mixtures is completely surrounded by the amorphous oxide phase and direct contact between the metallic lead phase disappears.

The above discussion indicates that the temperature-dependent manner in the peculiar electrical resistive behaviour is interpreted by the Gruneisen–Bloch relation [10] which is applicable to conventional metals and alloys. Additionally, the large magnitude of the temperature-dependent resistivity agrees well with the theoretical values expected from the mixing rule for the Al–O–Pb films containing more than 33.3% lead which are thought to have a direct contact between lead phases. However, the combined property of the high electrical resistivities and the large temperature-dependent resistivity for the Al–O rich alloys is significantly different from the theoretically expected property. The discrepancy suggests the possibility that the other approach must be taken into consideration in order to interpret the peculiar electrical resistive behaviour. Although an established theory is not proposed, the combined achievement of the high electrical resistivity and positive temperature-dependent resistivity may be thought as due to the simultaneous satisfaction of a localization of conducting electrons only to the finely embedded metallic phases and a significant decrease in mobility of conducting lead electrons in the intervening Al_xO_y phase among lead particles. Additionally, the localized mechanism that the interface between the heterogeneous materials prevents significantly the percolation of conducting lead electrons into the Al_xO_y phase also appears to be important in understanding the present peculiar electrical resistant behaviour. Clearly, it is very important from technological and scientific points of view that fine dispersion of insoluble metallic elements in an oxide matrix results in extremely high electrical resistivities combined with large temperature-dependent resistivities which are not obtained for metallic and oxide materials, possibly because of the localization of conducting electrons into the metallic phases with a small volume fraction. An investigation in the practical use of these sputtered Al–O–Pb films as the sensing element in resistive thermometers and resistive magnetometers is in progress.

5. Summary

Al_xO_y-based duplex films exhibiting peculiar electrical resistive behaviour were prepared in $(Al–O)_xPb_{100-x}$ (x = 0, 36.1, 63.9, 87.5 and 92.8%) systems by the sputtering technique. The duplex films consisted of amorphous Al_xO_y matrices and a particulate fcc lead phase. The particle size and the interparticle distance were about 10 to 50 nm and about 10 to 40 nm, respectively, for the Al–O–Pb films.

The duplex films were found to exhibit extremely high electrical resistivities combined with large temperature-dependent variation of resistivities. The electrical resistivity at 273 K is $4.06 \times 10^6\,\mu\Omega$ cm for $(Al–O)_{87.5}Pb_{12.5}$ and decreases to $165\,\mu\Omega$ cm with increasing lead content to 63.9%. Additionally, the temperature-dependent resistivity variation is positive and the magnitude of the variation in the temperature range from 273 to 10 K reaches $3.74 \times 10^6\,\mu\Omega$ cm for $(Al–O)_{87.5}Pb_{12.5}$, $2520\,\mu\Omega$ cm for $(Al–O)_{63.9}Pb_{36.1}$ and $159\,\mu\Omega$ cm for $(Al–O)_{36.1}Pb_{63.9}$. The temperature dependence varies in proportion to T^5 at temperatures below about 20 K and T above about 50 K. The mechanism of such peculiar electrical resistive behaviour was discussed on the basis of the theory of electrical conductivity and the following theory was proposed: the contribution of the electrical conduction occurs only through the metallic lead phase embedded in Al_xO_y oxide matrix and the mobility of lead conducting electrons is remarkably hindered in the intervening oxide among the lead particles. It is thus very important from scientific and engineering points of view that the duplex films consisting of fine metallic particles embedded in the oxide matrix can be prepared by sputtering Al_2O_3 material containing a lead element, which is insoluble in aluminium in the equilibrium state, and that these duplex films exhibit peculiar and useful electrical resistive properties which cannot be achieved in metallic composite materials.

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