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Study of Microscopic Process of Diffusion Welding by Electric-Resistance Measurement and Transmission Electron Microscope†

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Abstract

The measurement of electric resistance across a bonding interface and observation of microstructures for a bonding zone with a transmission electron microscope (TEM) have been applied to the investigation of microscopic process of aluminum diffusion welding. The variation of the resistance across the bonding interface has been measured while heating the bonding zone at a constant rate of 15°C/min. The resistance is much higher than the resistivity of matrix at room temperature and nearly equal to the resistivity of matrix at temperatures higher than 620°C. These phenomena indicate that the oxide film on the faying surface prevents the real metallic contact up to such high temperature as just below the melting point. The existence of voids and oxide film at the bonding interface during diffusion welding has been confirmed by the observation of the microstructure for bonding zone with a TEM.

1. Introduction

If two metal surfaces can be cleaned sufficiently well and pushed sufficiently close with each other, they will bond one to the other, even at room temperature[1),2)]. However, in practical metal surfaces there are factors such as oxide film and surface roughness which make such bonding difficult to achieve[1)]. With diffusion welding, it is necessary to overcome these difficulties of the oxide film and the surface roughness by heating the bonding zone and applying a pressure to the faying surface. The oxide film and surface roughness are considered as very important factors for the diffusion welding of metals. However, the thickness of the oxide film and height of the microasperity on the faying surface are so small that their behaviour during diffusion welding can not be observed with a conventional optical microscope or scanning electron microscope. The measurement of the electric resistance across the bonding interface and observation of microstructure for the bonding zone with a transmission electron microscope (TEM) are considered as very available for the investigation of microscopic process of diffusion welding in view of physical metallurgy. Therefore, in this experiment the measurement of electric resistance across the bonding interface and observation of microstructure for bonding zone with a TEM have been applied to the investigation of microscopic process of diffusion welding.

2. Experimental Details

2.1 Measurement of Electric Resistance

The base metal used in this experiment was commercially pure aluminum (1050). **Fig. 1 (a)** and **(b)** show the schematical diagram of the arrangement of specimen in diffusion welding and circuit for the measurement of the electric resistance used in this experiment. The specimen used in this experiment had a cubic shape whose edge length was 3~4 mm. The faying surface was finished by polishing with emery paper of 1500 grade. The specimens were placed between the pressing axes with their faying surfaces in contact. The specimens were contacted so that the grooves on the faying surface due to polishing with emery paper crossed at nearly right angles. The pressure was applied through the pressing axes of alumina as shown in Fig. 1 (a), and the bonding zone was heated with a radiant resistance heater of molybdenum foil. The diffusion welding was carried out in a vacuum environment of $10^{-4} \sim 10^{-5}$ mmHg, and the measurement of electric resistance was made during the diffusion welding.

As shown in Fig. 1 (b), a constant dirrect current I (supplied by the DC precission current supply) flowed

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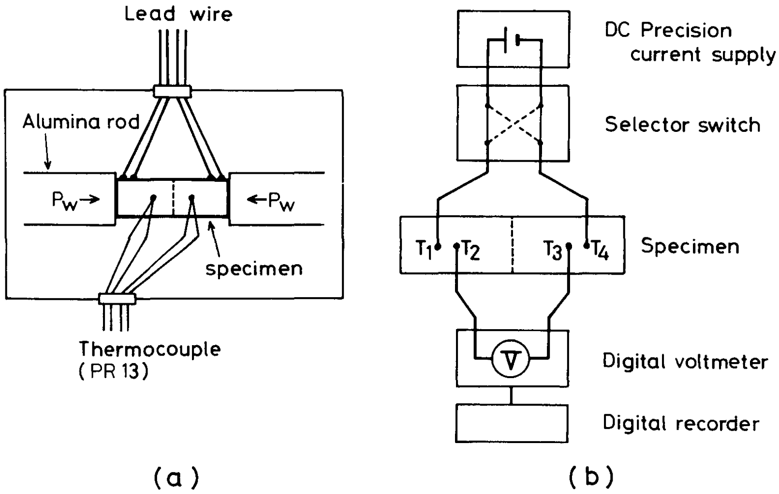

Fig. 1 Schematical diagram showing the arrangement of specimens in diffusion welding (a) and circuit for the measurement of electric resistance (b).

through the bonding zone between the terminal of T_1 and T_4. And the potential difference V between the terminal of T_2 and T_3 was measured with a digital voltmeter. Then, the resistance R between T_2 and T_3 can be evaluated by Ohm's law, as

$$R = V/I \text{ -------------- } (1).$$

In order to compensate the effect of thermoelectric power in the circuit on the measured value of the potential difference, the polarity of the current was reversed and the mean value of the potential difference for each polarity was adopted as V in (1). The current I was controlled to 0.01 mA and the potential difference was measured to an accuracy of 0.01 μV.

The resistance R of the bonding zone between T_2 and T_3 is described as

$$R = \frac{\rho_c}{S} - \frac{\rho_m}{S} \cdot l \text{ ----------- } (2)$$

which may be rearranged as

$$\rho_c = R \cdot S - \rho_m \cdot l \text{ --------- } (3)$$

where ρ_c, ρ_m, S and l are the contact electric resistance per unit faying surface, resistivity of the matrix, area of faying surface (equal to the sectional area of specimen), and distance between the terminal of T_2 and T_3 in Fig. 1 (b), respectively. In order to compare the resistance of the bonding zone with the resistivity of the matrix, ρ defined as

$$\rho = \rho_c + \rho_m$$

was calculated from the measured values of R, ρ_m, S, and l using equation (3).

2.2 Observation of Microstructure with a Transmission Electron Microscope (TEM)

In order to observe a microstructure of metal with a TEM, it is necessary to prepare a foil as thin as several thousand angstroms. The method of electropolishing is generally the most useful method of preparing thin foil from bulk metal. However, it is very difficult to prepare a thin foil from a bonding zone by means of electropolishing because chemical attack would change the profile of the bonding interface which contains many voids and inclusions such as oxide film. In this experiment, thin slices were prepared mechanically from the bonding zone by using an ultramicrotome with a diamond knife. The thin slices obtained were examined in Hitachi HU-12A electron microscope operating at 125 kV.

The specimen used for the observation with a TEM was welded by a similar method reported in a previous paper[3].

3. Results and Discussions

3.1 Electric Resistance of Bonding Interface

There is a possiblility of changing the bonding state due to the Joul heat at the bonding interface and breakdown of oxide film caused by the current I. **Fig. 2** shows the effect of the current on the measurement of electric resistance ρ for the bonding zone. As shown in Fig. 2, there is no remarkable change in the measured value of the resistance for the current density lower than 10 mA/mm^2. This indicates that the effect of the current on the bonding state is negligibly small for the current density lower than 10 mA/mm^2. On the basis of this result, 6.5 mA/mm^2 of current density was used in this experiment which corresponded to the current I of 100 mA.

Fig. 3 shows the variation of the resistance ρ for the

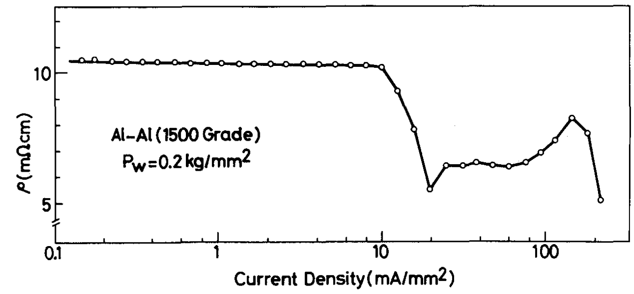

Fig. 2 Effect of current density on the resistance ρ of an aluminum bonding zone.

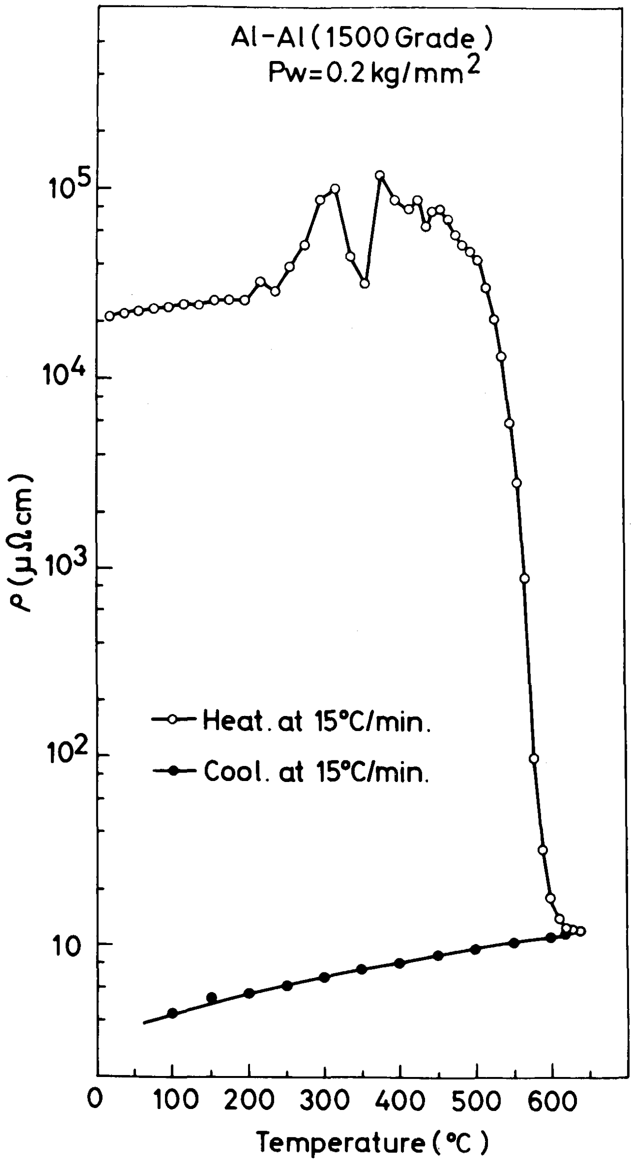

Fig. 3 Variation of the resistance ρ shown as a function of temperature while heating the bonding zone from room temperature to 640°C and then cooling to room temperature at the constant rate of 15°C/min.

aluminum bonding zone while heating the bonding zone from room temperature (R.T.) to 640°C and then cooling at the constant rate of 15°C/min. The welding pressure was 0.2 kg/mm^2. In Fig. 3, log ρ is plotted as a function of heating temperature. The resistance ρ during cooling is nearly equal to the resistivity ρ_m of the matrix. As shown in Fig. 3, the intial value of ρ (at room temperature) is much higher than the resistivity of matrix, and ρ during heating from R.T. to 200°C increases with the rise of temperature in the same manner as the resistivity ρ_m. In the temperature range from 200°C to 480°C, the value of ρ fluctuates largely in many cases as shown in Fig. 3. The amplitude of the fluctuation of the resistance in this temperature range is different in each specimen. In the case where the amplitude of the fluctuation is small, the resistance ρ increases in the same manner as ρ in the temperature range lower than 200°C. A large decrease in ρ with the rise of temperature was observed in the temperature range from 480°C to 630°C. This decrease of ρ was accompanied with the deformation of the base metals. At 630°C just below the melting point of aluminum, the value of ρ during heating was nearly equal to the resistivity of matrix.

Such a high initial value of ρ as in Fig. 3 indicates that the real metallic contact between the faying surfaces was achieved within only very narrow area. There have been several studies[1,3,4,5] which suggest that the oxide film of aluminum is very tenacious and prevents the real metallic contact between the faying surfaces. The high initial value of ρ for aluminum bonding zone may be explained by the existence of the tenacious oxide film on the faying surface. On the other hand, the value of ρ starts to decrease as the deformation of the base metal is recognized and is nearly equal to the resistivity of matrix at the temperature just below the melting point. These phenomena indicate clearly that the oxide film of aluminum is very stable and it is difficult to disrupt the oxide film by microscopic deformation in the bonding interface.

3.2 Observation of Microstructure with a Transmission Electron Microscope (TEM)

Photo. 1 shows an electron micrograph for the bonding interface between aluminum. As shown in Photo. 1, large lenticular voids are observed at the bonding interface. The ripples in the base metals are considered as due to deformation introduced during the cutting with a microtome. In the bonding interface between the lenticular voids, the contact between the faying surfaces is supposed to be achieved, as shown in Photo. 1.

Photo. 2 shows a micrograph of the bonding interface between the lenticular voids observed at a higher magnification. As shown in Photo. 2, there exists a much

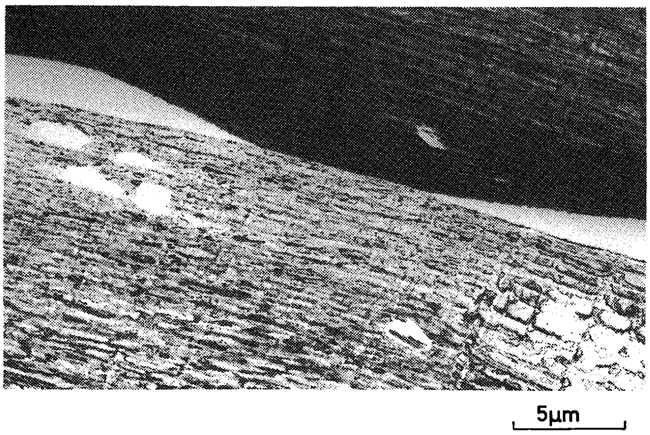

Photo. 1 Electron micrograph for a bonding zone of diffusion-welded joint of aluminum. The welding temperature, pressure, and time are 600°C, 0.05 kg/mm^2, and 60 min, respectively.

Photo. 2 Electron micrograph for a bonding zone of diffusion-welded joint of aluminum observed at a higher magnification than that shown in Photo. 1. The welding temperature, pressure, and time are 600°C, 0.05 kg/mm^2, and 60 min, respectively.

smaller void than those in Photo. 1 at the bonding interface. The void is margined by a layer where the intensity of transmitted electron is higher than that in the matrix. This layer may possibly be an oxide film of aluminum. Thus, the existence of voids with various size and oxide film at the bonding interface is confirmed by the observation with a TEM.

4. Conclusion

The measurement of electric resistance across the bonding interface and observation of bonding interface with a TEM were applied to the investigation on microscopic process of diffusion welding of aluminum. The main conclusions obtained in this investigation on the effect of oxide film and roughness on faying surface on the microscopic process of diffusion welding are as follows.

The variation of electric resistance across the bonding interface was examined while heating the bonding zone at a constant rate. The electric resistance across the bonding interface is much higher than the resistivity of the matrix, and nearly equal to the resistivity at the temperature just below the melting point of aluminum. These phenomena indicate that the tenacious oxide film of aluminum prevents the real metallic contact between the faying surfaces and it is very difficult to disrupt the oxide film by microscopic deformation in the bonding interface. It is confirmed by the observation of microstructure in the bonding interface with a TEM that the voids of various size and oxide film exist at the bonding interface.

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