SEM and EDX of Microstructure of the Contact Layer in a Solidification Process

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INTRODUCTION

The microstructure of the contact layer that arises in the solidification process of tin on a cold copper plate is very interesting from a technical point of view. An understanding of the microstructure of the contact layer is very important and can allow indirect determination of the heat resistance of this layer. The contact layer exerts an influence on the solidification process, i.e. the thicknesses of the layers of tin and copper. Both the thickness of the frozen (solid) tin layer and solution tin layer in the contact layer are especially important in the technology of the production of printed-circuit electronics. Modelling of the properties of multicomponent Sn-Pb melts, especially flow [1-5], viscosity [6] and electroconductivity [7,8] is very important.

THEORETICAL MODEL

Figure 1 shows a theoretical model of the region where liquid tin flows beneath a cold copper plate and solidifies. This phenomenon is accompanied by the flow of instantaneous latent heat \( q \) through the contact layer (compos- site layer) which is bounded by a set of thin walls with different thermal conductivity.

The contact layer with variable thickness \( \delta_{\text{VX}} \) in the solidification process arises between both the cold copper plate (Cu) with a thickness \( H \) and the frozen or solid layer (Sn) with a variable thickness \( \delta_{\text{VS}} \). Both thicknesses \( \delta_{\text{VX}} and \delta_{\text{VS}} \) depend on time. The function \( T(t) \) describes the dependence of temperature distribution on time. The temperature \( T_{\text{C}} \) is the fusion point of tin.

In an earlier paper [1] a theoretical analysis was carried out. The following simplifying assumptions were made: all thermodynamic parameters are considered constant; the solidification front is sharp and planar; whole latent heat is absorbed by the copper plane; the frozen layer of the tin is very thick and does not absorb heat; the liquid tin is not overheated. The energy balance equates the instantaneous latent heat and the convective heat flow from the inside liquid tin to the frozen layer and to the cold copper. The solutions of the energy equation with the initial conditions \( \delta_{t} = 0 \) when \( t = 0 \) are in the dimensionless functions for both the copper temperature and the frozen layer thickness:

\[
\theta_{\text{C}}(t) = \frac{\delta_{\text{C}}(t)}{\lambda_{\text{C}} \cdot \text{Ste}} \quad \text{and} \quad \theta_{\text{F}} = \frac{T_{\text{F}} - T_{\text{C}}}{T_{\text{F}} - T_{0}},
\]

In the above equations we introduced the following set of dimensionless variables and parameters:

1. Dimensionless time, dimensionless frozen layer, dimensionless temperatures:
   \[ \tau = \frac{T_{\text{F}} - T_{0}}{\text{Fo} \cdot \text{Ste}}, \quad \delta = \frac{\delta_{\text{VX}}}{H}, \quad \theta_{\text{F}} = \frac{T_{\text{F}} - T_{\text{C}}}{T_{\text{F}} - T_{0}}, \]

2. The Fourier number, the Stefan number, the Biot number, and other dimensionless parameters:
   \[ F_{O} = \frac{\text{Fo} \cdot H^{2}}{\lambda_{\text{C}}}, \quad \text{Ste} = \frac{c_{\text{L}}(T_{\text{F}} - T_{\text{C}})}{L}, \quad \text{Biot} = \frac{\alpha_{\text{copper}} \cdot H}{\lambda_{\text{c}}}, \quad a = \frac{\alpha_{\text{c}}}{a_{\text{copper}}, \quad \lambda_{\text{c}} = \frac{\lambda_{\text{copper}}}{\lambda_{\text{c}}}}, \]

where: \( a_{\text{c}} and a_{\text{copper}} \) are the thermal diffusivity of copper and tin, \( \lambda_{\text{c}} \) and \( \lambda_{\text{copper}} \) are the thermal conductivity of copper and tin, \( T_{\text{C}} \) and \( T_{\text{F}} \) are the temperatures of copper and initial copper plate, \( c_{\text{L}} \) is the specific heat of tin, \( L \) is the latent heat of liquid tin fusion, and \( \alpha_{\text{copper}} \) is the liquid tin-cold copper contact layer heat transfer coefficient.

In Figure 2 we can see the dependence of the frozen layer thickness on time for the different Biot numbers. These results are the solution to the second equation. We can see

![Figure 1: Theoretical model of the contact layer.](Image)

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in Figure 2 that the influence the Biot number on the frozen layer thickness is very significant. It follows that the flow of heat by the contact layer indirectly depends on its structure, which is described by the macroscopic parameter of the heat transfer coefficient \( \alpha_{\text{layer}} \).

Thermal conductivity is attributed to both molecular interaction as in other phases, and to free electrons, which are present in metals. The free-electron mechanism of both heat and electrical conductivity are described by the Widemann-Franz-Lorenz equation \([7]\). As mentioned above, both conductivities depend on temperature, materials, and particularly on the concentration of the solution.

**MATERIALS AND METHODS**

Metallurgical tin Sn99, with the addition of 0.5% Pb and with 0.2% Sb, 0.04% As, 0.2% Bi, 0.05% Cu and 0.01% Fe, was melted in a reduction atmosphere. The chemical constitution of the alloy was corrected with glow LECO GDS 750.

Due to the furnace construction, there are special flow channels, and the liquid tin flows below the cold copper plate as in the theoretical model (Fig. 1). The copper plate was 5 mm thick, the flow times 10 s, and the melting temperature 600K.

Electron microscopy of the copper plate with the tin layer was performed in a Jeol JSM 5600 LV microscope.

**RESULTS**

Figure 3 shows scanning electron micrographs of the results of the tin-copper surface investigations.

The EDX results of the slag-copper surface scanning analysis (Fig. 4) shows the effects of lead precipitation on the tin area. The X-ray spectra acquired from the interfaces (Fig. 5) show the presence of Cu, Sn and Pb which is in good agreement with its initial chemical composition.

The results of the surface interfacial analysis are presented in the schematic in Figure 6.

**CONCLUSIONS**

It is generally known that heat conduction and electrical conductivity depend on the microstructures and distribution of elements in the tin solution \([7,9]\).

The authors have shown that the observed precipitates of poor lead and the tin containing grain area in the interface layer are important to the description of the thermal conductivity of the contact layer. The structure contact of the copper-lead-tin layer is described, which changes in a continuous way, but in the place with the lead inclusions must be uncontained (see Fig. 6).

The observations described here are extremely important because they explain the process of the formation of the products during dynamic solidification which is taking place on the tin-copper interfacial surface.