Thermodynamic Database on Microsolders and Copper-Based Alloy Systems

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Recent progress on the thermodynamic databases of calculated phase diagrams in microsolders and Cu-based alloys is presented. A thermodynamic tool, Alloy Database for Microsolders (ADAMIS), is based on comprehensive experimental and thermodynamic data accumulated with the calculation of phase diagrams (CALPHAD) method and contains eight elements, namely, Ag, Bi, Cu, In, Sb, Sn, Zn, and Pb. It can handle all combinations of these elements and all composition ranges. The elements of Al and Au have also been added to ADAMIS within a limited range of compositions. Furthermore, a database of Cu-based alloys, including binary (Cu-X), ternary (Cu-Fe-X, Cu-Ni-X, and Cu-Cr-X), and multicomponent (Cu-Ni-Cr-Sn-Zn-Fe-Si) systems, has also been developed. Typical examples of the calculation and application of these databases are presented. These databases are expected to be a powerful tool for the development of Pb-free solders and Cu substrate materials as well as for promoting the understanding of the interfacial phenomena between them in electronic packaging technology.

Key words: Pb-free solders, thermodynamic database, Cu-based alloys, calculation of phase diagram, phase-field method, interfacial reaction

INTRODUCTION

During the past decade, Pb-free solders, to replace conventional Pb-Sn alloys, have been designed and developed to meet the requirements arising from environmental and health issues concerning the toxicity of Pb.[1–3] Many investigations have indicated that Pb-free solders are likely to be multicomponent alloys because the melting temperatures of binary candidates are either too high or too low, and their mechanical properties are not adequate. In view of the necessity to develop Pb-free solders with high efficiency, and the transition time involved in replacing Pb-bearing alloys, a thermodynamic database of microsoldering alloys for reliable predictions of liquidus, solidus, phase fractions and constitutions, equilibrium and nonequilibrium solidification behavior, etc., in a multicomponent system is required because it is difficult to understand these factors from available references. In addition, a thermodynamic database of the Cu-based alloys is also important for the design of substrate materials. The calculation of phase diagrams (CALPHAD) is an effective method for alloy design[4] and has been widely used in development of new materials.[5] Recently, the present authors have developed thermodynamic databases for microsoldering materials[6–8] and Cu-based alloys[9] within the framework of the CALPHAD method. This database is useful for the design of Pb-free solders and Cu-based substrate materials as well as for understanding the interfacial reaction between them. In the present paper, the validity of these two thermodynamic databases for calculation, alloy design, and applications is demonstrated.

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THERMODYNAMIC DATABASE OF MICROSOLDERS

A thermodynamic tool of microsoldering materials named Alloy Database for Microsolders (ADAMIS) was developed by combining the thermodynamic

database of microsolders[6–8] with *Pandat*, (Madison, WI) a multicomponent, phase diagram-calculation software program.[10] In this database, the phase equilibria in any system that includes the elements Ag, Bi, In, Cu, Sn, Sb, Zn, and Pb can be calculated in the whole composition range. Recently, two elements, namely, Al and Au, were added to this database, and calculations are available for a limited composition range of these elements.

In the database, the thermodynamic parameters for describing the Gibbs energy of each phase are evaluated by optimizing the experimental data pertaining to the phase boundary compositions and thermodynamic properties, such as activity, heat of mixing, and enthalpy of formation. Regarding the present database, there are some important alloy systems for which there has been little or no experimental data. Thus, experimental work to determine phase equilibria, such as liquidus, solidus, isothermal and vertical sections, etc., has been carried out, and a better estimation of the thermodynamic parameters with good agreement between the calculated and the observed phase equilibria has been obtained.[11–21] The thermodynamic parameters were optimized and evaluated by Thermo-Calc (Stockholm, Sweden) software.[4]

ADAMIS is a user-friendly thermodynamic tool for the design of microsoldering materials. Beginners can easily manage it through the Windows interface. The calculated results are independent of the user's level of expertise because it has the ability to automatically find starting points and initial values for stable phase equilibria.

Figure 1 shows the main contents of ADAMIS, where much information, such as phase equilibria, projection of the liquidus surface, and simulation of solidification etc., can be obtained. In addition, the surface tension and viscosity in the liquid phase can also be calculated.

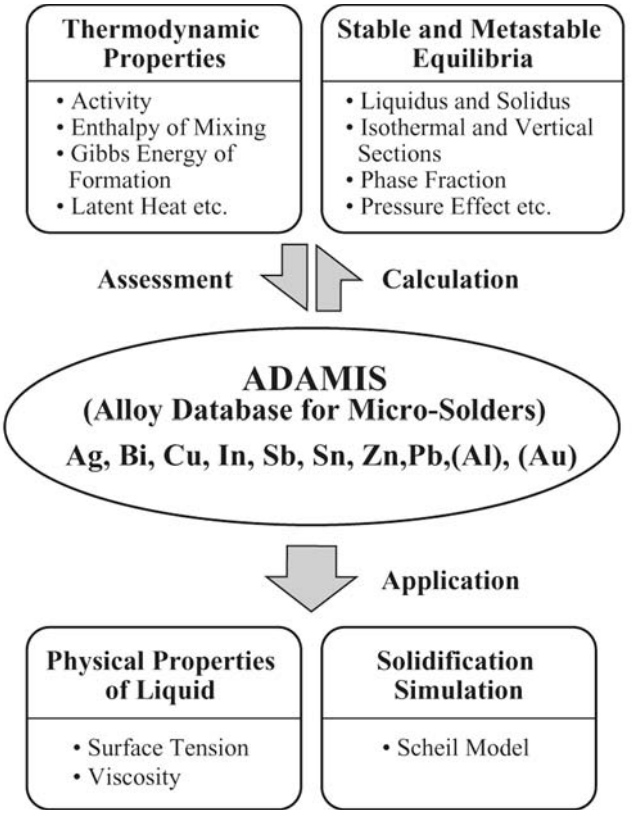

Fig. 1. An outline of ADAMIS.

THERMODYNAMIC DATABASE OF Cu-BASED ALLOYS

A thermodynamic database of Cu-based alloys containing Cu-X binary, Cu-Fe-X, Cu-Cr-X, and Cu-Ni-X ternary and multicomponent systems was also developed on the basis of the CALPHAD method. An outline of the assessed systems is shown in Table I. To obtain a good assessment for thermodynamic parameters, experimental investigations of phase equilibria, including not only first-order phase transformation but also order-disorder transition for some important systems with no or very little experimental data, were carried out on the basis of differential scanning calorimetry, energy dispersive x-ray spectroscopy, the diffusion couple method, etc.[22–29] For example, the ordering of the bcc phase has been determined in the Cu-Al,[26] Cu-Sn,[27] and Cu-Fe-Al systems.[28]

Thermodynamic parameters of the liquid phase were mainly estimated based on the experimental data of the phase equilibria and thermodynamic properties, such as activity, enthalpy of mixing, etc., and those for solid phases were assessed by fitting the experimental data of phase equilibria. Good agreement between the calculated results and experimental data was obtained for most systems. The present thermodynamic database of Cu-based alloys can provide information useful for the design of Cu alloys similar to those shown in Fig. 1. In addition, it is also important to facilitate understanding of the

Table I. Thermodynamic Database for Cu-Based Alloys

System	Elements (X)
Cu-X binary system	Ag, Al, Au, B, Be, Bi, C, Co. Cr, Fe, In, Li, Mg, Mo, Mn, Nb, Ni, P, Pb, Si, Sb, Sn, Ti, Tl, V, Y, Zn, Zr
Cu-Ni-X ternary system	Ag, Cr, B, Be, Fe, Mn, P, Sn, Si, Pb, Ti, Zn
Cu-Fe-X ternary system	Al, V, Nb, Cr, Mo, Mn, Ni, Co, Si, Sn, C, P, Zn
Cu-Cr-X ternary system	Fe, Ni, Nb, Ti, Sn, Zr, C, P, Si, Zn
Multicomponent system	Cu-Ni-Cr-Sn-Zn-Fe-Si

interfacial reaction between solders and Cu-based alloys by combining ADAMIS with this database of Cu-based alloys.

APPLICATION OF THERMODYNAMIC DATABASE

The thermodynamic databases of microsoldering materials and Cu-based alloys can be combined with other software programs, and various kinds of simulations can be conducted. Figure 2 shows an outline of the application of the present databases, which provides not only thermodynamic information, such as phase equilibria by using the Thermo-Calc and Pandat software, but also the information on the kinetics and evolution of microstructure. Some examples for simulations and applications are as follows.

Design of Microsoldering Materials

To develop suitable alternatives to Pb-Sn solders, the melting temperature is one of the most basic factors. Significant efforts have been made to design Sn-based solders because they have ranges of melting temperature similar to those of Pb-Sn solders. Figure 3 shows the liquidus projection of some Sn-based ternary systems in which the isothermal lines and reaction types are indicated. The solidification process of solders is also one of the important factors for the design of Pb-free solders. Although the Scheil model assumes that local equilibrium exists at the liquid/solid interface and diffusion is absent in the solid phase, such a calculation can still provide a predication close to reality. Solidification simulation of a promising candidate as an alternative to Pb-Sn solders, the Sn-2.0Ag-0.5Cu-7.5Bi (wt.%) alloy, was carried out. Figure 4a shows the variation of calculated mass fraction of the solid phases with temperature in this alloy under equilibrium and Scheil model solidification conditions. In both cases, the solidification starts with the primary crystals of η-Cu_6Sn_5, and the liquid phase disappears at 177.9°C under the equilibrium solidification condition. However, according to the Scheil model, Bi is concentrated in the liquid phase during solidification, which causes an extensive fall of the terminating temperature of solidification (139.9°C), corresponding to the eutectic reaction of the Sn-Bi binary system. Figure 4b shows the concentration of elements in the liquid phase in which Bi is concentrated in the liquid phase during solidification.

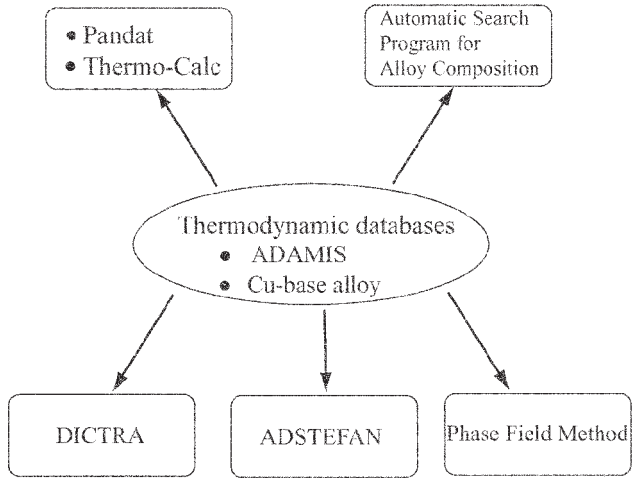

Fig. 2. The outline of the application of the thermodynamic database.

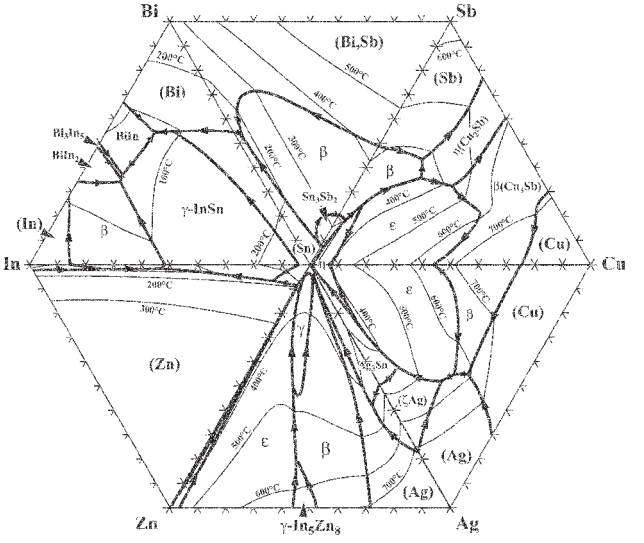

Fig. 3. The liquidus surfaces in the Sn-based ternary systems.

The surface tension and viscosity of the liquid phase are very relevant to the soldering process. They can be predicted from the Gibbs energy of liquid by combining the appropriate models.[30–34] Figure 5 shows the calculated results of the effect of alloying elements on surface tension and viscosity of the ternary-eutectic Sn-3.24Ag-0.57Cu (wt. %) alloy. It can be seen from Fig. 5a that the surface tension increases and decreases by the addition of Zn and Sb, respectively. The viscosity is slightly lower than that of the ternary-eutectic alloy because of the addition of elements Bi, In, Sb, and Zn (Fig. 5b).

Design of Cu-Based Alloys

On the basis of the thermodynamic database of Cu-based alloys, the phase equilibria, phase fraction, liquidus projection, etc., in Cu-X binary and Cu-Cr-, Cu-Fe-, and Cu-Ni-X ternary as well as Cu-Cr-Ni-Fe-Sn-Zn-Fe-Si systems can be calculated. High-strength Cu-based alloys with high electrical conductivity have been used for various electronic devices, such as connectors and springs. These Cu alloys are developed by making use of precipitation hardening of the fcc phase. Typical examples of phase equilibria of age-hardened Cu-based alloys are shown. Figure 6 shows the calculated Cu-Be binary phase diagram in which the miscibility gap of the β(B2) + γ(A2) is reproduced because of the ordering of the bcc phase in the Cu-rich portion. Figure 7a shows the calculated, vertical section diagram at 3wt.%Ni in the well-known Corson alloy system, Cu-Ni-Si. It is seen that Ni_5Si_2, Ni_2Si, and

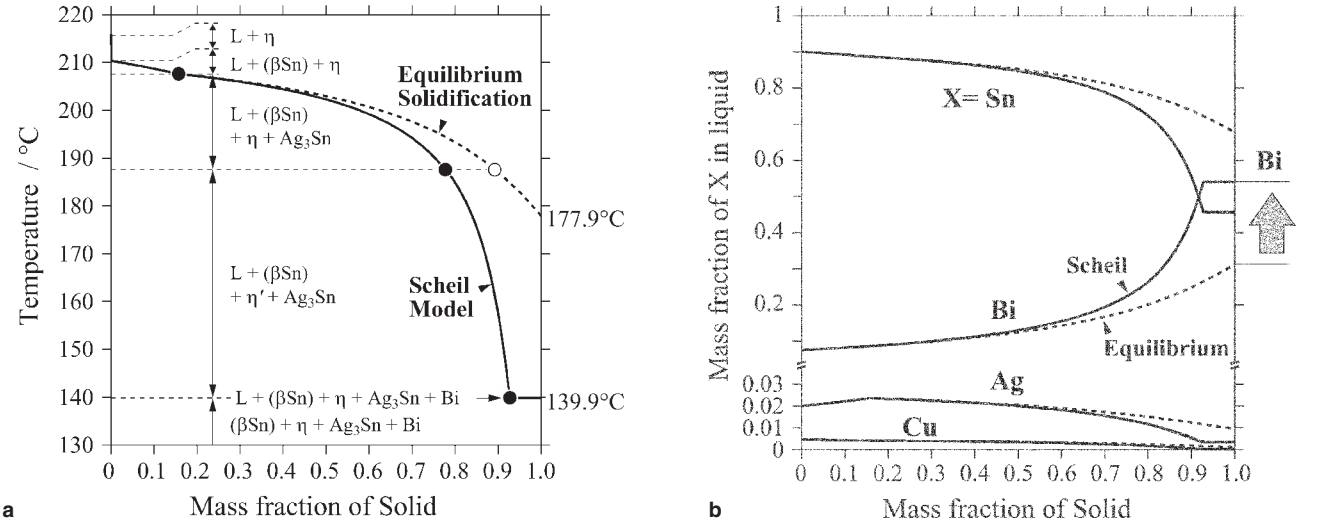

Fig. 4. The solidification simulation of the Sn-2.0Ag-0.5Cu-7.5Bi (wt.%) alloy.

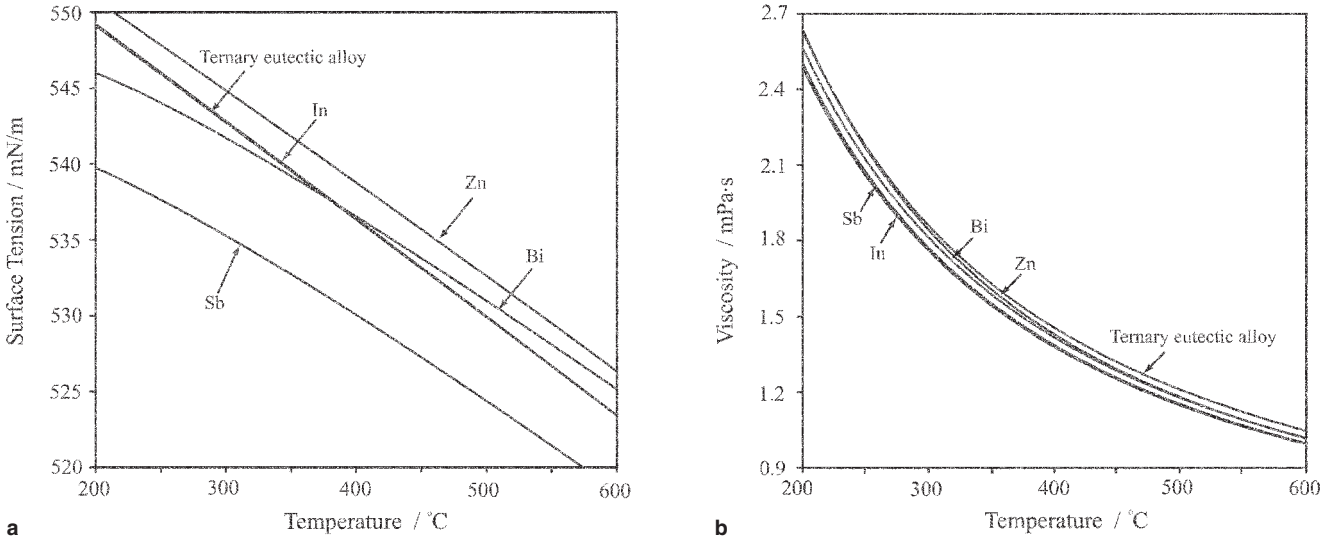

Fig. 5. The effect of alloying elements on the (a) surface tension and (b) viscosity of the ternary-eutectic Sn-3.24Ag-0.57Cu-2.0X (wt.%) alloy.

Ni_3Si compounds may precipitate from the fcc phase during cooling because of a small addition of Si. Figure 7b shows the calculated change of phase fraction with temperature in the Cu-3Ni-0.7Si (wt.%) alloy, which is composed of the fcc phase and these two Ni_2Si and Ni_5Si_2 compounds. These compounds are often used to enhance the strength of Cu-based alloys because of precipitation hardening. Another example of the phase equilibria is that of high-strength spinodal-type Cu-Ni-Sn alloys. The calculated vertical section at 9wt.%Ni is shown in Fig. 8a in which the miscibility gap of the fcc phase exists in the Cu-rich portion at a lower temperature, and Ni_3Sn precipitates from the fcc phase during cooling. Figure 8b is the calculated phase fraction in the Cu-9Ni-2.3Sn (wt.%) alloy, which is composed of the fcc phase and the Ni_3Sn compound in a wide temperature range, and the microstructure with the miscibility gap of the fcc phase may be obtained at a low temperature.

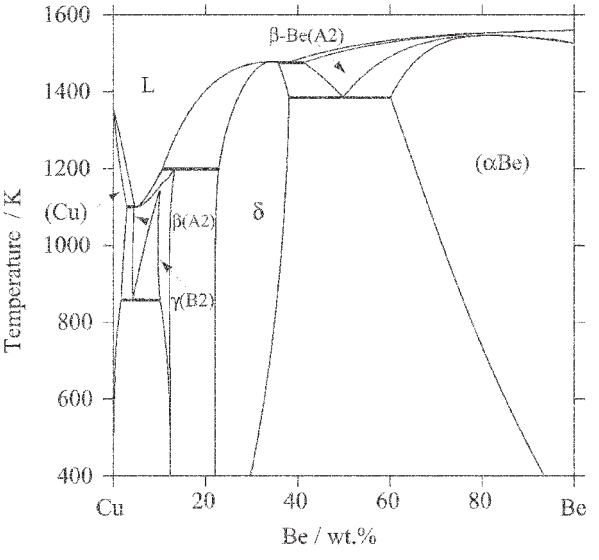

Fig. 6. A calculated Cu-Be binary phase diagram.

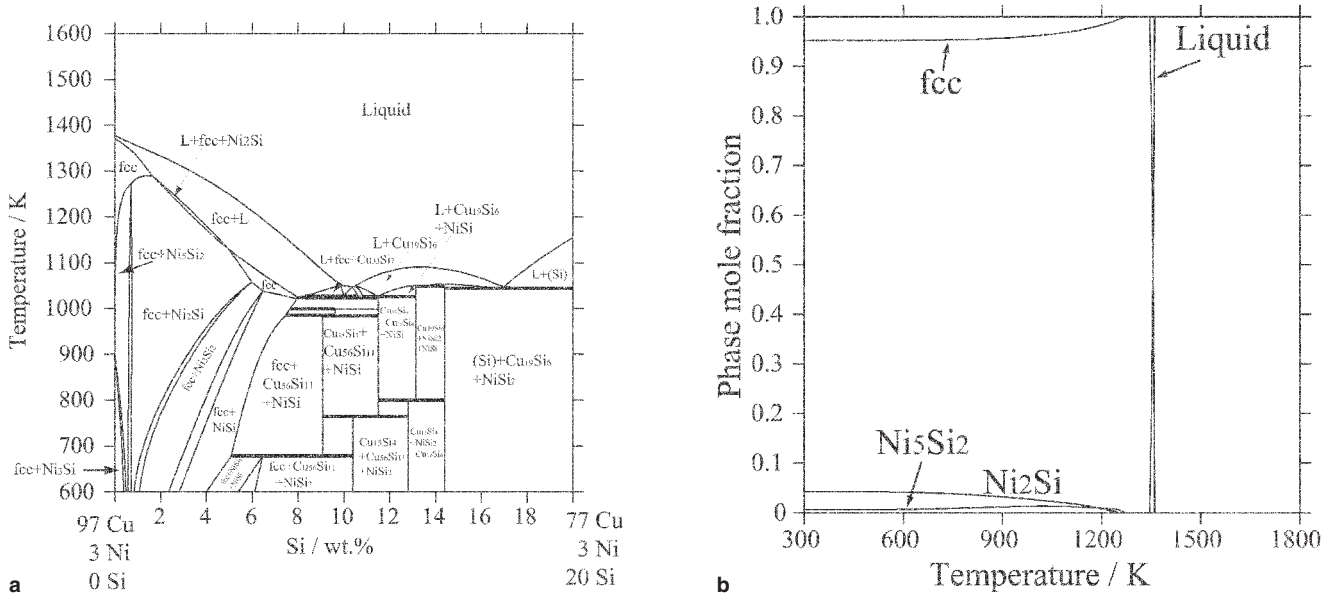

Fig. 7. (a) A calculated vertical section at 3wt.%Ni and (b) phase molar fraction for Cu-3Ni-0.7Si (wt.%) in the Cu-Ni-Si system.

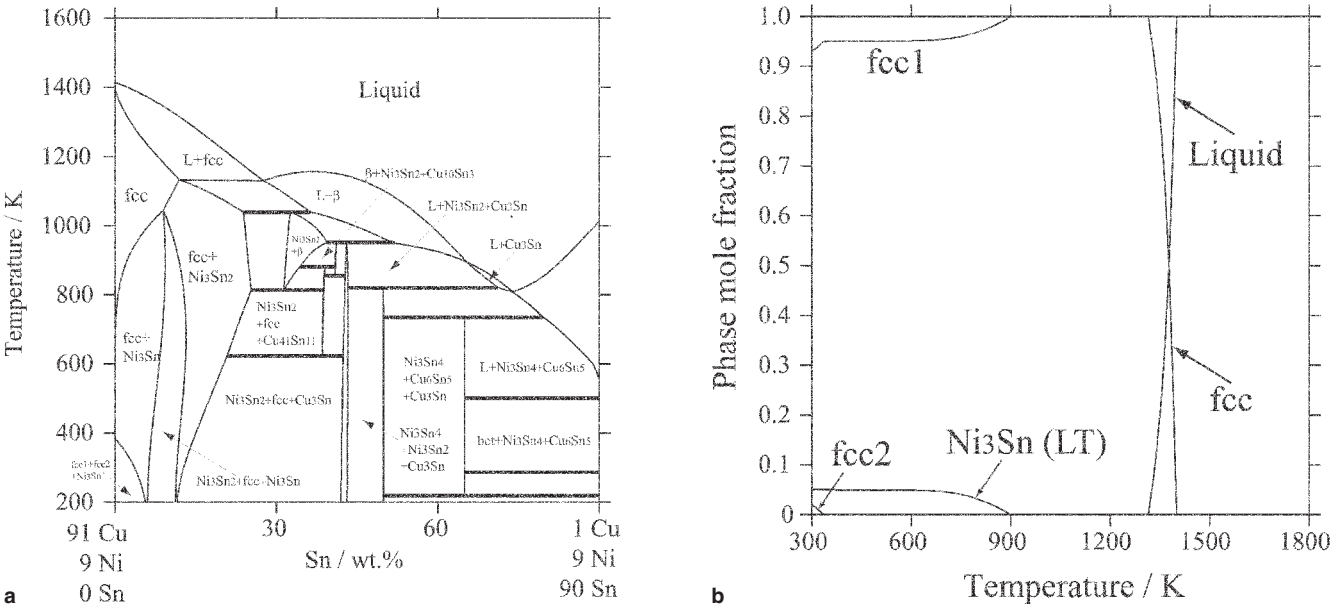

Fig. 8. (a) A calculated vertical section at 9wt.%Ni and (b) phase molar fraction for Cu-9Ni-2.3Sn (wt.%) in the Cu-Ni-Sn system.

Dissolution of Cu in Molten Solders Simulated by Diffusion Controlled Transformation

The dissolution reaction between a substrate and solders is important for the reliability of semiconductor electronic packaging. Such a problem can be predicted by simulation of diffusion on the basis of *Diffusion Controlled Transformation (DICTRA)* software (Stockholm, Sweden).[34] As an example, for a diffusion couple Cu (solid)/ Sn (liquid) heat treated at 250°C for different lengths of time, the simulation of the dissolution behavior of Cu in molten Sn was carried out.[35] Figure 9a shows the change of the concentration of Cu in liquid Sn with increasing time. It is seen that the concentration of the liquid phase almost reaches the equilibrium composition when the time is about 500 sec. In addition, the movement of the Cu/Sn (liquid) interface to the Cu side can also be observed. Figure 9b gives the calculated position and speed of movement of the fcc/liquid interfacial boundary. It is seen that the Cu/Sn boundary moves at a larger negative speed at the beginning of diffusion, which means that this boundary rapidly moves to the Cu side. Thus, Cu rapidly dissolves into liquid Sn at the beginning stage of diffusion, and the speed of Cu dissolution decreases with increasing time.

Simulation of the Solidification Process by the Phase-Field Method and Advanced Solidification Technology for Foundry Aided by Numerical Simulation

The evolution of microstructure during solidification, which is essential for understanding lift-off

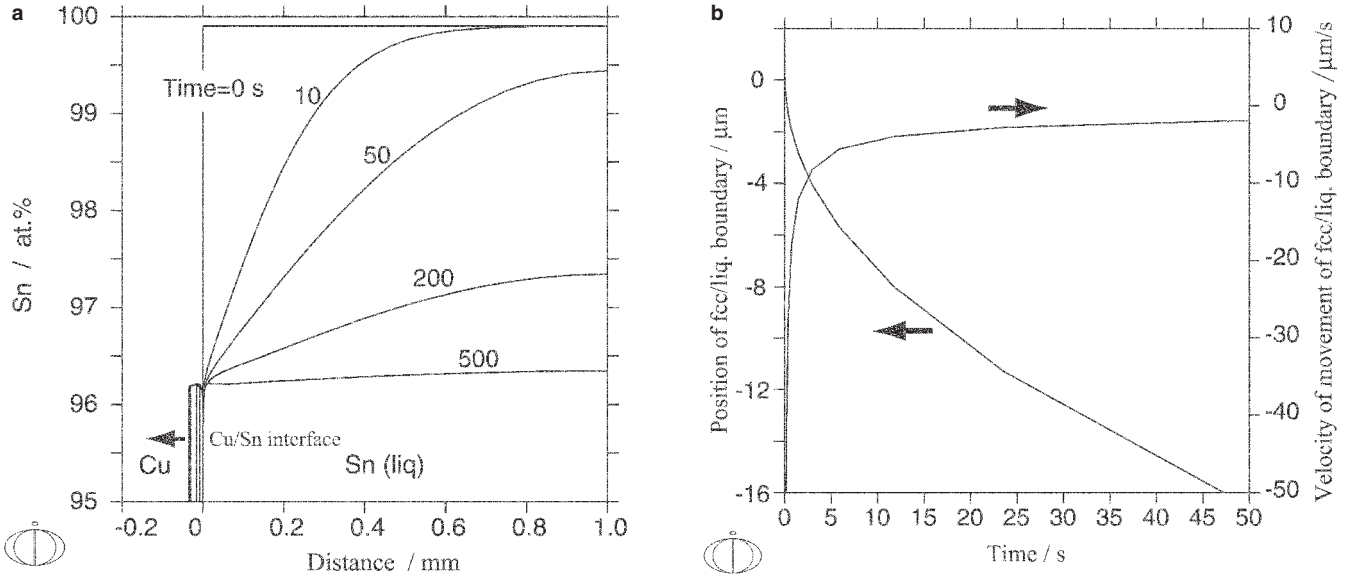

Fig. 9. The calculated results of dissolution behavior of Cu in molten Sn: (a) the change of concentration in liquid phase and (b) velocity of movement and position of the fcc/liquid boundary.

phenomena,[36,37] can be quantitatively predicted by the phase-field method.[38,39] Ode et al. have applied phase-field simulation to microstructure evolution during solidification of the Sn-5wt.%Bi alloy in reflow soldering.[40] Figure 10 shows a schematic diagram of the calculation area. The system was assumed to possess rotational symmetry, and the triangular shape corresponds to the area that was simultaneously frozen with the final solidification point in the solidification simulation. The simulation results of microstructure evolution during solidification at the tilted angles of 45° and 0° are shown in Fig. 11, respectively, corresponding to the microstructures at 3 sec after solidification. From Fig. 11a, it is seen that the tip of the dendrite for the tilted 45° angle is slightly bent at the land because of solute enrichment. When the tilted angle is 0°, however, the tip of the primary dendrite approaches the land without bending (Fig. 11b), and the secondary dendrite arms grow along the land surface. The results show that microsegregation does not always prevent the growth of the solid in the vicinity of the land.

In addition, the information from the database, such as the phase fraction of solid phases, latent heat evolution, composition of growing solid phases, etc., can be employed for predicting the three-dimensional solidification process by Advanced Solidification Technology for Foundry Aided by Numerical Simulation (ADSTEFAN).[41] ADSTEFAN can show the evolution of solidifying soldering materials. The simulated results for the Sn-Ag-Cu alloy were obtained by the present authors.[20]

Pb-Free Solder Ball with Core Structure

Compared with more conventional packages, the ball grid array (BGA) package has the advantage of having higher input/output terminal density, a smaller footprint, and higher reliability.[42] In particular, Cu-core solders plated with a Pb-Sn eutectic alloy for BGA joints have been developed and used.[43] The present authors have recently found that an egg-type powder with core microstructure can be obtained in many alloy systems possessing a liquid miscibility gap.[44] Here, we present a promising BGA ball consisting of a Cu-based core with a Pb-free, low-melting solder periphery designed by ADAMIS. A powder of Cu-15Sn-50Bi (wt.%) was prepared using conventional nitrogen-gas atomization under an argon atmosphere, where a stable liquid-miscibility gap appeared as predicted by ADAMIS. Figure 12 shows the microstructure of this alloy powder, clearly indicating the egg-type structure of the Cu-Sn-rich core with a Bi-Sn-rich periphery. The commercial size of the Cu-core ball plated with the Pb-Sn eutectic solder is about 700 μm, but a size less than 100 μm, which is very difficult to produce by the conventional plating method, is required for the chip scale package. As shown in Fig. 12, the size of the egg-type powder is about 80 μm, which should prove to be a very attractive ball for electronics packaging technology in the future.

Automatic Search Program for Alloy Composition

In addition to the melting temperature of soldering alloys, the difference between the liquidus and solidus (ΔT_{ls}) is important because it influences the microstructure of solidifying solders. Generally, however, it is difficult to find the suitable melting point and ΔT_{ls} for meeting the requirement of new soldering materials in a system with more than three components. Recently, an automatic search program for alloy composition with the aid of PanEngine[45] has been developed[46] and combined with

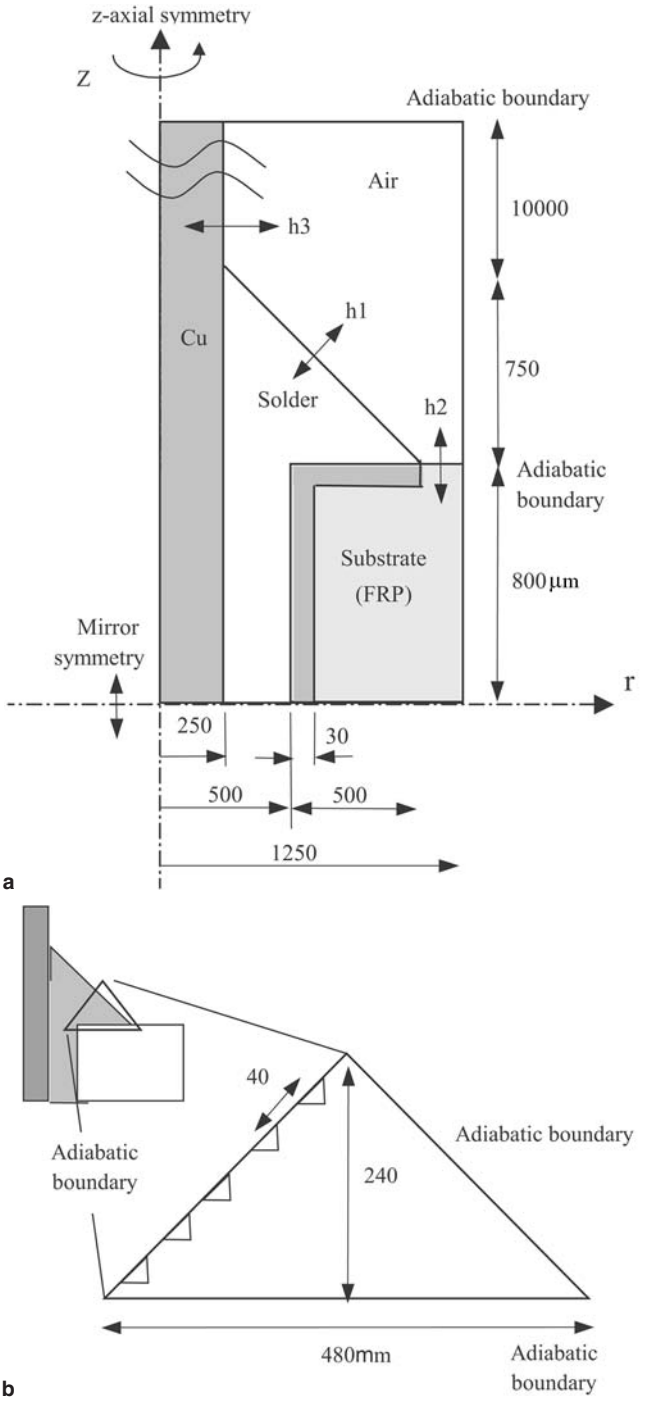

Fig. 10. The calculation area for (a) microscale solidification simulation and (b) calculation area for the phase-field simulation.

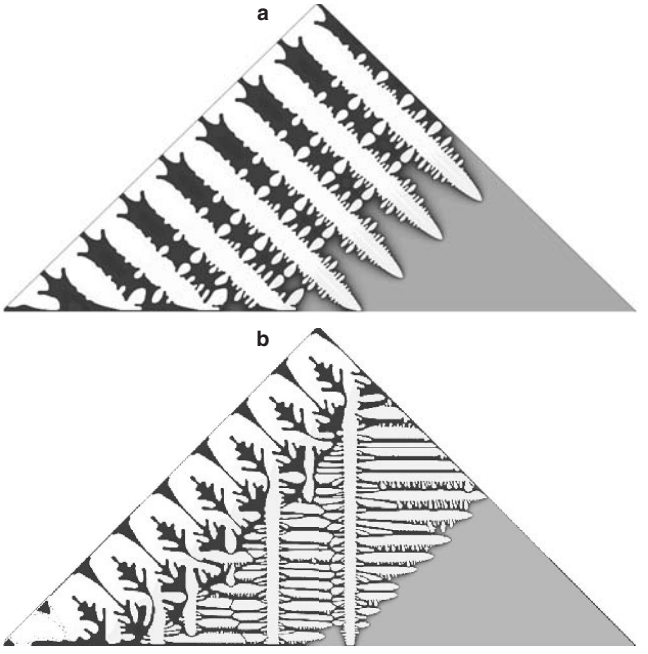

Fig. 11. The microstructure evolution in the fillet above the Cu land. Tilted angle: (a) 45° and (b) 0°.

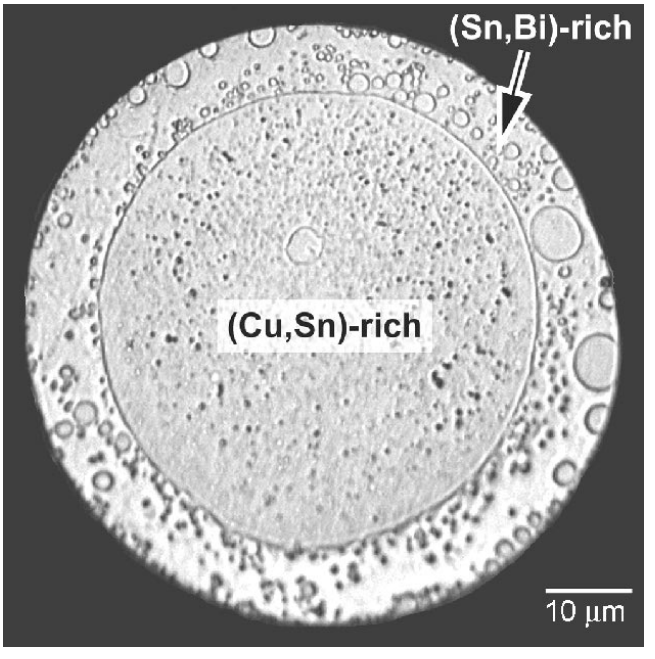

Fig. 12. The egg-type microstructure of the Cu-15Sn-50Bi (wt.%) alloy powder.

ADAMIS and the database of Cu-based alloys. This program can give a range of compositions corresponding to the required melting point, and ΔT_{ls} and is a very convenient tool in the design of solders and Cu-based alloys.

CONCLUDING REMARKS

Thermodynamic databases for microsoldering alloys and Cu-based alloys have been developed on the basis of the CALPHAD method. These databases can provide much valuable information on not only thermodynamics, such as phase equilibria, phase fraction, etc., but also on kinetics and evolution of microstructures when they are combined with some appropriate software and models, such as DICTRA and the phase-field method. Examples have been given to demonstrate the great utility of these databases for designing Pb-free solders and high-strength Cu-based alloys and for predicting the dissolution of Cu and simulation of evolution of microstructure.

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