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Intermetallic Compound Layer Growth Kinetics in Non-Lead Bearing Solders[1]

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Abstract

The introduction of alternative, non-lead bearing solders into electronic assemblies requires a thorough investigation of product manufacturability and reliability. Both of these attributes can be impacted by the excessive growth of intermetallic compound (IMC) layers at the solder/substrate interface. An extensive study has documented the stoichiometry and solid state growth kinetics of IMC layers formed between copper and the lead-free solders: 96.5Sn-3.5Ag (wt.%), 95Sn-5Sb, 100Sn, and 58Bi-42Sn. Aging temperatures were 70-205°C for the Sn-based solders and 55-120°C for the Bi-rich solder. Time periods were 1-400 days for all of the alloys. The Sn/Cu, Sn-Ag/Cu, and Sn-Sb/Cu IMC layers exhibited sub-layers of Cu_6Sn_5 and Cu_3Sn; the latter composition was present only following prolonged aging times or higher temperatures. The total layer growth exhibited a time exponent of n=0.5 at low temperatures and a value of n=0.42 at higher temperatures in each of the solder/Cu systems. Similar growth kinetics were observed with the low temperature 58Bi-42Sn solder; however, a considerably more complex sub-layer structure was observed. The kinetic data will be discussed with respect to predicting IMC layer growth based upon solder composition.

Introduction

Soldering provides the primary means for assembling electronic devices, sub-systems, and products for both consumer and military applications. Cost-effective

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manufacturing processes, which are required of companies that wish to compete in the global marketplace, require the minimization of assembly defects that can lead to post-fabrication repair and rework or, worse, service failures in the field. The Sn-Pb solders, 63Sn-37Pb (wt%) and 60Sn-40Pb, have provided a reliable attachment technology for electronic systems. Also, Sn-Pb solders have readily accommodated ongoing device and system miniaturization as well as the demand for low-cost, high volume manufacturing processes.

However, Pb, which is a principle component of electronic-grade solders, poses a hazard to human health as well as to the environment. Restrictions on the use of Pb in a wide range of manufactured products, either by means of legislation or tax incentives, will cause the electronics community to develop alternative solders for their products. The logistics for replacing such a well established technology as tin-lead soldering with alternative non-lead bearing alloys will require that the following tasks be exercised: (1) development of suitable alloy compositions with desired thermal, mechanical, and physical metallurgy properties; (2) implementation of cost-effective manufacturing processes for electronics assembly; (3) establishment of a reliability database to predict the service lifetime of solder joints, and (4) the growth of an industry-wide infrastructure that will provide support technologies such as fluxing materials as well as alternative, Pb-free circuit board and package lead finishes. Already, considerable effort has been given to these aspects; the reader is referred to citations [1,2,3,4,5] for detailed discussions.

An important aspect of the physical metallurgy of alternative solders is the reaction layer formed at the interface between the solder and the Cu substrate. This reaction layer is comprised of covalent or "intermetallic" compounds of Sn and Cu. The intermetallic compound (IMC) layer forms, initially, as a part of the wetting process by the molten solder; however, it can also develop further by solid state diffusion processes after joint solidification. The solid state growth rate increases with temperature and time duration. Shown in Fig. 1a is the IMC layer which develops between 63Sn-37Pb and Cu after thermal aging at 170°C (solid state) for 402 days. The Cu_3Sn and Cu_6Sn_5 sub-layers are identified as the ε and η', respectively, from the Cu-Sn binary phase diagram in Fig. 1b. An IMC layer indicates that, in fact, a metallurgical bond has formed between the molten solder and the substrate surface.

In electronic applications, the IMC layer impacts largely the manufacturability of solder joints during product assembly. This effect comes about because, in most instances, the package lead or termination as well as the copper land and/or plated through hole on the printed circuit board have been deposited with a 100Sn or 63Sn-37Pb coating *prior to* assembly. By establishing a layer of Sn or Sn-Pb solder on the base

metal surfaces before the attachment process, wettability of the surfaces has already been assured. In the case of hot solder dipped coatings, poor wetting by either the individual components (i.e., package I/O or circuit board feature) or the circuit board features can be remedied before assembly. This is a more cost effective approach than having to inspect for, and rework, defective joints on the final product.

However, long term storage of electronic components or their exposure to elevated temperatures during such life testing as "burn-in" or thermal cycling accelerate the solid state growth of the IMC layer between the base metal and the Sn or solder coating. Moreover, such coatings are intentionally made very thin in order to prevent excessive solder from accumulating in the joint at the time of assembly, thereby reducing the likelihood of damage to, and/or misalignment of, the very small leads on fine pitch technology packages. On the other hand, a consequence of limited coating thicknesses is that under elevated temperature conditions, the IMC layer can potentially grow to an extent so as to consume the entire Sn or Sn-Pb coating. The IMC layer is then exposed to the atmosphere whereby it readily oxidizes and becomes non-solderable, even under the strongest fluxes allowable in electronics manufacturing. The consequence is defective solder joints on the product. Therefore, it is necessary to establish the growth kinetics of IMC layers formed between Cu and alternative, non-lead bearing solders so as to be able to predict device (and circuit board) solderability following long term storage or life testing procedures.

The formation and thickness of the IMC layer can also affect the reliability of the solder joints; albeit, this factor is of lesser pertinence to electronics applications except for unusual applications. By the covalent nature of the bonds, the Cu_3Sn and Cu_6Sn_5 sub-layers have very limited ductility. The brittle nature of the IMC will affect joint strength under impact load conditions (i.e., forces applied at a high rate). Although fracture of the IMC layer has the potential to be catastrophic from the sense of a loss of mechanical integrity as well as electrical continuity, such a failure mode is not traditionally observed in the service life of most Sn-Pb solder-based electronic products. The mechanical loads experienced by a solder joint are typically limited to residual stresses generated by the thermal expansion mismatch between the circuit board laminate and the device package. Such loads develop as a consequence of assembly processes or thermal cycling conditions, causing time dependent deformation to take place within the solder. The IMC layer typically does not have an explicit role in this mode of deformation.

This study will outline the growth kinetics of the IMC layers which form between Pb-free alloys: 100Sn, 96.5Sn-3.5Ag, 95Sn-5Sb, and 58Bi-42Sn with Cu. A brief

description of the IMC structure will be presented for each bi-metal couple along with the kinetic data of IMC layer thickness versus time. Trends will be sought that correlate the solder alloy physical metallurgy, solder thermal properties, and aging temperature with IMC layer thickness.

Experimental Procedures

The substrates used in this investigation were oxygen-free, high conductivity Cu tabs measuring 0.635 x 0.635 x 0.159 cm. The composition of the Cu was verified by atomic emission spectroscopy to have the following contaminants and maximum content levels: 40 ppm Ag, 50 ppm Al, 10 ppm Mg, 30 ppm Mn, and 10 ppm Si (with an error of a factor of two). The surface on which the thickness values were taken, was optically polished.

The Cu substrates were hot solder coated by dipping each specimen into a bath of the molten metal or alloy. The solders, their liquidus (T_l) and solidus temperatures (T_s), as well as their bath or working temperatures (T_w) were: 100Sn (T_l=T_s=232°C, T_w=270°C); 96.5Sn-3.5Ag (T_l=T_s=221°C, T_w=260°C); 95Sn-5Sb (T_l=232, T_s=240°C, T_w=280°C), or 58Bi-42Sn (T_l=T_s=139°C, T_w=190°C) solder. The details of the coating procedure can be found in Reference 6.

Thermal aging of the solder-coated test samples was performed in air furnaces with a temperature stability of ±0.5°C. The annealing temperatures were 70, 100, 135, 170, and 205°C for the 100Sn, 96.5Sn-3.5Ag, and 95Sn-5Sb solders, and 55, 70, 85, 100, and 120°C for the 58Bi-42Sn alloy.. The time periods were 1 to 400 days with a tracking error of ±30 min. Upon completion of the heat treatments, the specimens were cross sectioned and prepared for metallographic analysis. The composition of the intermetallic layers were determined by electron microprobe analysis (EMPA). Measurement of the intermetallic compound layer thickness was performed by quantitative image analysis. Forty measurements were made per sample. A minimum thickness resolved by this technique was 0.5 mm. The data were represented by a mean and ± one standard deviation.

Results

A brief description of the IMC layer structure and growth kinetics is outlined below for each of the diffusion couples.

100Sn/Cu. An optical micrograph of the IMC layer formed at the 100Sn/Cu interface is shown in Fig. 2. The IMC layer was comprised of the Cu_3Sn and Cu_6Sn_5 sub-layers stoichiometries. The total layer thickness as a function of the square root of

time is shown by the filled symbols in Fig.3 (thickness values from aging at 205°C were not included in this graph). These 100Sn/Cu data were overlaid with the results from similar tests on 63Sn-37Pb (open symbols). The 100Sn/Cu total IMC layer grew with parabolic kinetics ($\sqrt{t}$) at 70, 100, and 135°C and $t^{0.42}$ kinetics at 170 and 205°C. The apparent activation energy was 66 kJ/mol. Growth of the Cu_3Sn sub-layer was parabolic ($\sqrt{t}$) at all temperatures with an apparent activation energy of 43 kJ/mol.

96.5Sn-3.5Ag/Cu and 95Sn-5Sb/Cu. Optical micrographs of the solder/Cu interface for the 96.5Sn-3.5Ag/Cu and 95Sn-5Sb/Cu couples aged at 170°C for 150 days are shown in Fig. 4 (the dark shadow at the interface in Fig. 4a is an artifact of the sample preparation/optical microscopy technique and does not represent a crack). The Cu_3Sn and Cu_6Sn_5 sub-layers were observed after aging at temperatures of 170 and 205°C for 96.5Sn-3.5Ag solder and 135 (time>25 days), 170, and 205°C for 95Sn-5Sb alloy. Both solder/Cu systems exhibited total IMC layer growth kinetics similar to those of 100Sn/Cu, i.e., $\sqrt{t}$ growth at $T \leq 135$°C and $t^{0.42}$ kinetics at 170 and 205°C. The IMC layer thickness as a function of $\sqrt{t}$ for 96.5Sn-3.5Ag/Cu and 95Sn-5Sb/Cu are shown in Figs. 5a and 5b, respectively. The 96.5Sn-3.5Sn/Cu and 95Sn-5Sb/Cu systems exhibited apparent activation energies (for total layer thickness) that were very similar to those measured in the 100Sn/Cu system: 59 kJ/mol and 61kJ/mol, respectively. Growth of the Cu_3Sn sub-layer exhibited $\sqrt{t}$ kinetics in both cases with identical apparent activation energies of 50 kJ/mol which were only slightly higher than that for the 100Sn/Cu couple.

58Bi-42Sn/Cu. A complex IMC layer structure developed in this couple; it is exemplified by the optical micrograph in Fig. 6a and accompanying electron microprobe trace in Fig. 6b for the sample aged at 120°C for 400 days. Generally, the total layer was comprised of one or more Cu_6Sn_5 sub-layers separated by Cu-Sn-Bi sub-layers of varying Cu, Sn, and Bi contents. The source of Bi in the Cu-Sn-Bi sub-layer(s) was from that which was rejected by the Sn-rich phase (containing 5 wt% Bi in solid solution), the Sn of which formed the Cu_6Sn_5 sub-layers. Unlike the Cu_3Sn and Cu_6Sn_5 sub-layers which developed in the Sn, Sn-Ag, and Sn-Sb solders, the IMC layer formed in the 58Bi-42Sn/Cu couple was prone to cracking. Such cracks were observed to have propagated typically between a Cu_6Sn_5 sub-layer and a Cu-Sn-Bi sub-layer. Only the total layer thickness was measured as a function of aging time; those data appear in Fig. 7. The apparent activation energy for total layer growth was very similar to that of the Sn-rich solders, 55 kJ/mol.

Discussion

The growth kinetics of the IMC layers were examined with reference to the solder compositions and their melting properties. A fundamental point that is raised with regards to IMC layer growth is whether the growth behavior responds to the environmental (aging) temperature or to the homologous temperature of the particular solder alloy. The homologous temperature is a ratio of the aging temperature to the liquidus temperature expressed in degrees Kelvin (i.e., T/T_l, with T and T_l in °K). The 100Sn, 96.5Sn-3.5Ag, and 95Sn-5Sb solders, as well as the 63Sn-37Pb (baseline) solder, had similar IMC layer thicknesses after long term aging. For example, at an aging temperature of 170°C and a time period of 200 days, the total IMC layer thicknesses for the 100Sn/Cu, 96.5Sn-3.5Ag/Cu, 95Sn-5Sb/Cu, and 63Sn-37Pb/Cu systems were: 19.9, 17.8, 20.6, and 21.0 µm, respectively. This observation suggests that the growth kinetics were largely dependent upon the *aging or test temperature* rather than upon the homologous temperature (i.e., the composition) of the particular alloy. In addition, similar kinetic parameters of time exponent and apparent activation energy support the observation of little or no composition dependence by the growth process(es). It is worthwhile to point out that, indeed, that the solders represented several different physical metallurgies: Sb as a solid solution element (95Sn-5Sb): Ag as Ag_3Sn intermetallic second phase particles (96.5Sn-3.5Ag); or Pb as an insoluble second phase (63Sn-37Pb).

Next, a comparison was made of the IMC layer thickness values of the 100Sn/Cu, 63Sn-37Pb/Cu, and 58Bi-42Sn/Cu systems. (The 100Sn/Cu data was used to also represent the 96.5Sn-3.5Ag/Cu and 95Sn-5Sb results due to their similar growth kinetics.) The kinetic parameters of time exponent, "n", and the apparent activation energy were listed in Table 1. The time exponents were similar, although, the value of n decreased from 0.5 to 0.42 at higher temperatures for the lead-free solders. The apparent activation energies showed a composition dependence in which the magnitude increased with Sn content. Also included in the data were the thickness "increase" values (i.e., $y-y_0$ where y is the thickness at time, t, and y_0 is the initial thickness at t=0) and growth rate (dy/dt) as a function of both the aging and the homologous temperatures. Given a constant aging temperature of 100°C and time period of 400 days, the thicknesses of the IMC layers for Bi-Sn, Sn, and Sn-Pb solders on Cu, were 14.2, 3.3, and 4.5 µm, respectively. The corresponding growth rates, as determined by the linear best fit (thickness versus $\sqrt{t}$), were 0.63, 0.16, and 0.19 µm/$\sqrt{}$day, respectively. Clearly, IMC layer growth was not strictly dependent upon the aging (or environment) temperature. Otherwise, similar thickness increases and growth rates should have been observed for each of the alloys. However, the layer thickness did appear to increase in the order of the

solders with decreasing liquidus temperature. This fact suggested that the aging temperature relative to the liquidus point (i.e., the homologous temperature) had a more significant impact on IMC layer development.

Therefore, the role of homologous temperature was examined as part of the Table 1 analysis. A value of 0.88 was selected for the homologous temperature because it represented test temperatures of 85, 170, and 135°C which were accessible to the 58Bi-42Sn/Cu, 100Sn/Cu, and 63Sn-37Pb/Cu data, respectively. Clearly, the thickness increases were not the same for the given homologous temperature. Upon further examination, the thickness and growth rate data appeared to exhibit an increase with aging temperature (which is the number in parentheses under the thickness value).

The data in Table 1 suggest that IMC layer growth between the 100Sn (which also represents the Sn-Ag and Sn-Sb solders), 58Bi-42Sn, and 63Sn-37Pb solders was not predicted strictly by either the aging temperature or by the homologous temperature; rather, IMC layer development depended upon both of these properties. This point was further supported when all of the temperature data for each of the solders was included in the analysis. The trends exemplified in Table 1 can be explained, based upon the knowledge of probable microstructural processes responsible for IMC layer formation. The IMC layer developed by two processes: (1) the reaction of Cu and Sn atoms at the solder and Cu boundaries, respectively, to form the IMC composition(s) and (2) the diffusion of Cu and/or Sn atoms through the layer, to the interfaces to support such reactions. If the diffusion mechanism was dominant, then the IMC layer growth would be largely dependent upon the aging temperature, given the same IMC layer composition, since diffusion within the layer would depend upon the structural of the IMC layer and not the solder field composition. On the other hand, a strong dependence upon the homologous temperature implies that those reactions at the solder interface (which directly involve the solder field) control IMC layer growth.

A mechanistic approach can be used to illustrate the data in Table 1. The 63Sn-37Pb/Cu and 100Sn/Cu (which includes the 96.5Sn-3.5Ag/Cu and 95Sn-5Sb/Cu) couples developed the same layer structures. Since the thickness values were similar for a given aging temperature, it was surmised that diffusion processes dominated layer development in these systems. The deviated behavior of the 58Bi-42Sn solder suggested that reaction processes at the IMC layer/solder interface dominated the growth processes of this system. Alternatively, the unusual IMC layer structure generated by the rejected Bi may have caused diffusion kinetics to be sufficiently different from those of the 100Sn/Cu and 63Sn-37Pb/Cu systems that the growth behavior of the 58Bi-42Sn/Cu system did not fit the trend established by the Sn-based alloys.

In summary, the quantitative data of IMC layer growth suggest that when the traditional Cu_3Sn and Cu_6Sn_5 sub-layers were present, the IMC growth kinetics were similar and, thus, determined largely by the aging temperature rather than the solder composition-dependent homologous temperature. The controlling microstructural process was solid state diffusion within the IMC layer. The 58Bi-42Sn/Cu system did not fit this trend. The two likely hypotheses were that (1) an interfacial reaction mechanism dominated the growth process or (2) the presence of the Cu-Sn-Bi sub-layers in the IMC structure caused changes to the diffusion kinetics in the IMC layer.

From the point-of-view of electronic systems design, a simplified rule based upon aging temperature or the melting properties of the solder could not be developed as a universal approach to predicting IMC layer growth for alternative, Pb-free solders. A microstructural analysis is necessary in order to identify the IMC layer structure. Should the IMC layer be based upon the development of the Cu_3Sn and Cu_6Sn_5 sub-layers, then its growth kinetics would, in all likelihood, be similar to those of the 100Sn/Cu or 63Sn-37Pb/Cu systems and, hence, be predicted by the particular aging temperature. However, the development of a different IMC layer structure, as was the case with the 58Bi-42Sn/Cu system, would require an experimental program to document IMC layer growth.

Finally, this study also demonstrated that the presumption that a reduced IMC layer growth rate would be realized by solders of lower Sn contents, is entirely unfounded. The 58Bi-42Sn solder exhibited the smallest Sn content; yet, at a given aging temperature, it consistently presented the fastest IMC layer growth rate than was the case of the other three Pb-free solders. Also, the IMC layer in the 58Bi-42Sn/Cu couple appeared to have a reduced mechanical strength as indicated by the presence of cracks.

Conclusions

(1) The role of intermetallic compound (IMC) layers in the manufacturability and reliability of solder joints has been documented by the extensive history of Sn-Pb solders use in electronic products.

(2) As the electronics engineering community enters the early stages of prototype testing with alternative, Pb-free solders, concerns for the effects of IMC layers will increase in importance.

(3) Isothermal IMC layer growth between 100Sn, 96.5Sn-3.5Ag, or 965Sn-5Sb solders, and Cu was documented for time periods of up to 400 days. Parabolic (or nearly so) growth rates as well as the presence of both Cu_3Sn and Cu_6Sn_5 sub-layers in the Pb-free

solder couples resembled the 63Sn-37Pb/Cu system. The ratio of Cu_3Sn -to-Cu_6Sn_5, however, differed between the solder composition.

(4) Comparable IMC layer structures between the 96.5Sn-3.5Ag, 95Sn-5Sb, 100Sn, and 63Sn-37Pb solders resulted in similar diffusion-controlled growth kinetics that exhibited a similar aging temperature dependence.

(5) The 58Bi-42Sn/Cu couples developed complex IMC layer chemistries with traditional Cu_6Sn_5 sub-layers separated by Cu-Sn-Bi sub-layers. Although the growth rates were faster than those of the other solders, the kinetic parameters (time exponent and activation energy) were similar.

(6) Differing diffusion rates within the complex IMC layer structure of the or the controlling process being atomistic reactions at the interfaces rather than diffusion, provided the two potential mechanisms for the deviated IMC layer growth behavior of the 58Bi-42Sn/Cu system. Thickness predictions in such systems, which have non-traditional IMC layer structures, will require experimental measurements in order to obtain the necessary data for the design and process development of electronic products targeted for assembly with Pb-free solders.

Acknowledgments

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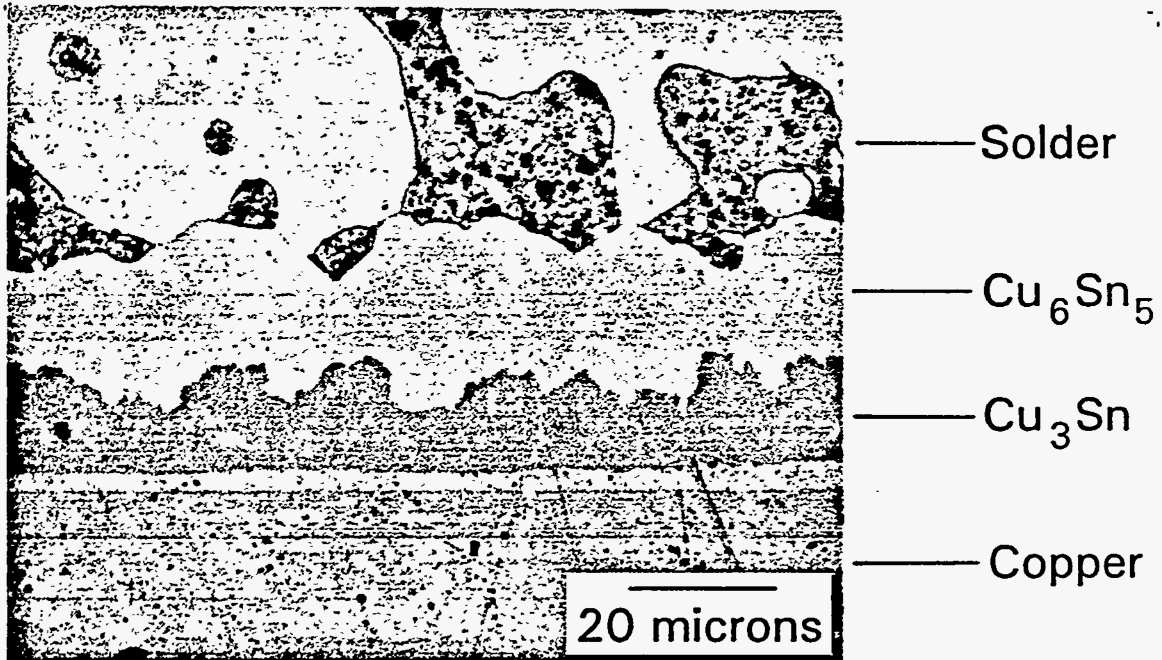

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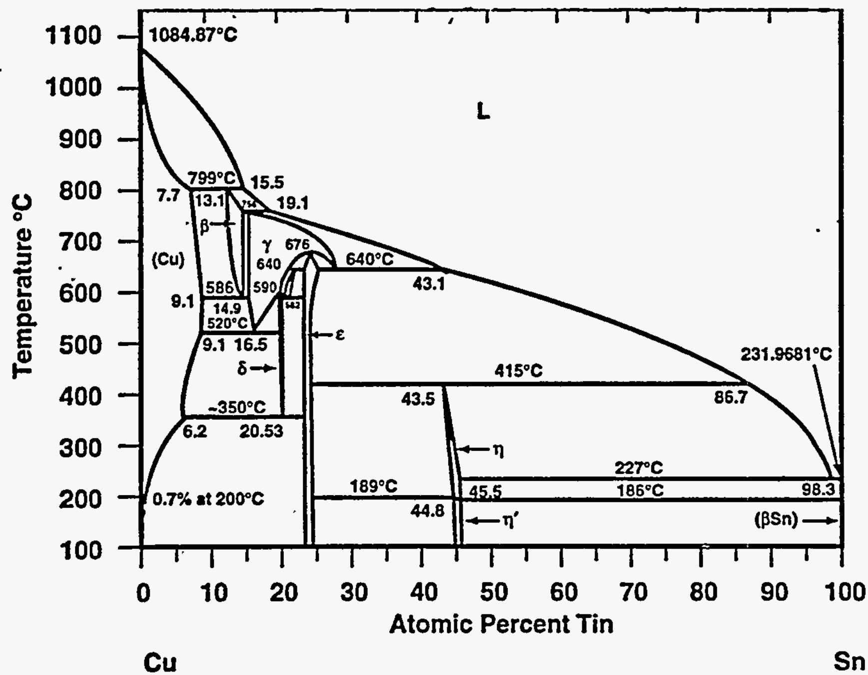


(b)

Fig. 1 (a) Intermetallic compound layer development in the 63Sn-37Pb/Cu couple aged at 170°C for 402 days. The Cu_3Sn and Cu_6Sn_5 sub-layers were identified as the ε and η' phases, respectively, in the Cu-Sn binary phase diagram (b).

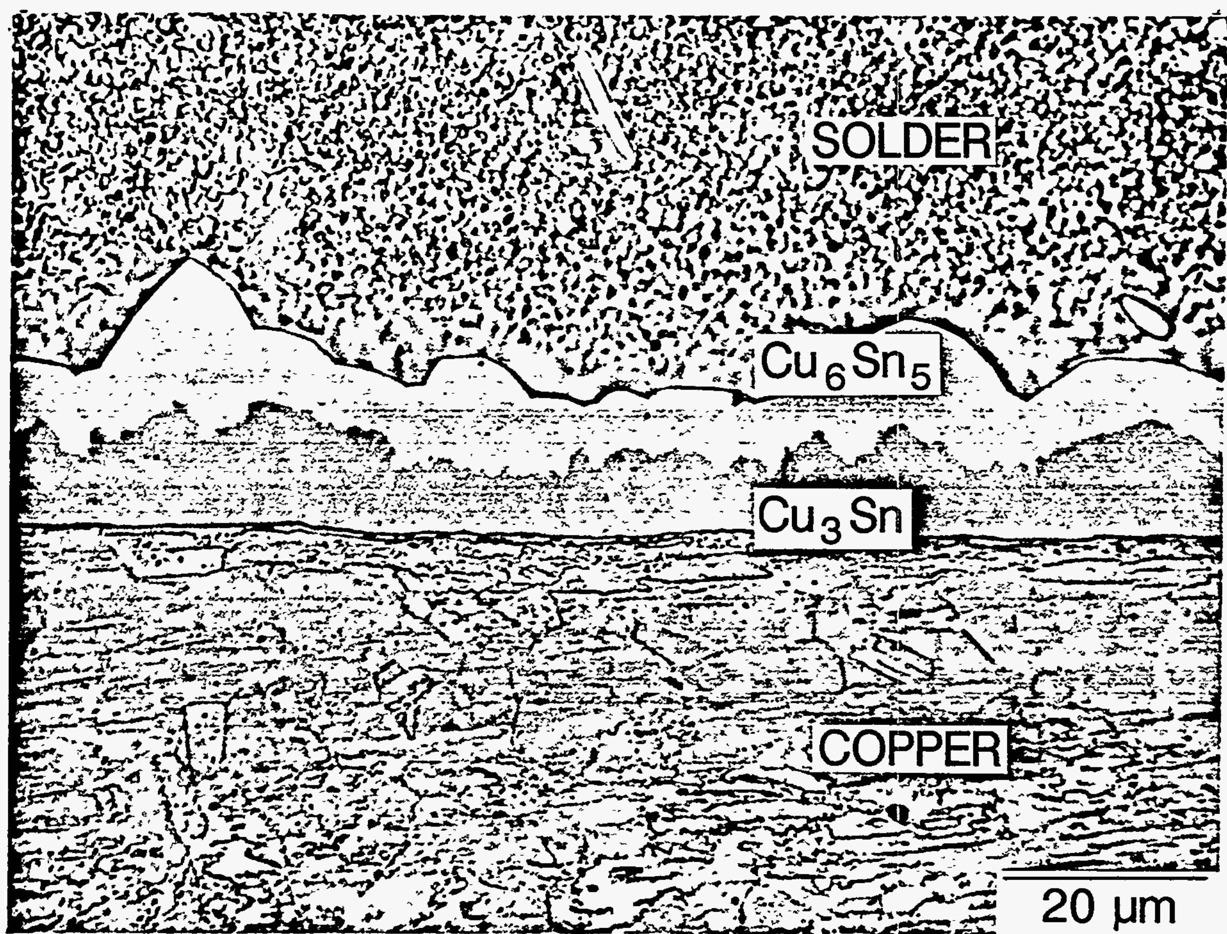


Fig. 2 Optical micrograph of the IMC layer formed at the 100Sn/Cu interface by thermal aging at 170°C for 200 days.

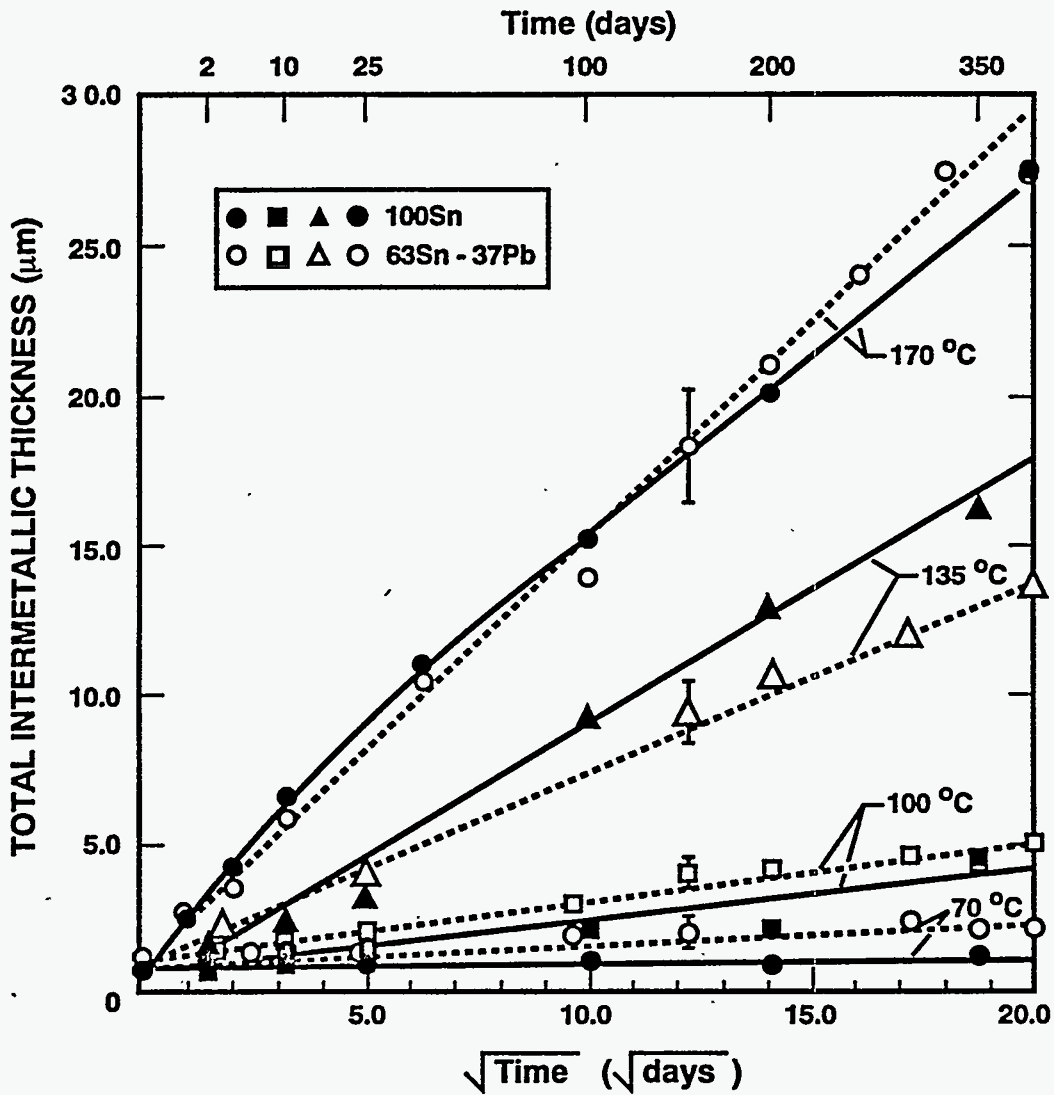
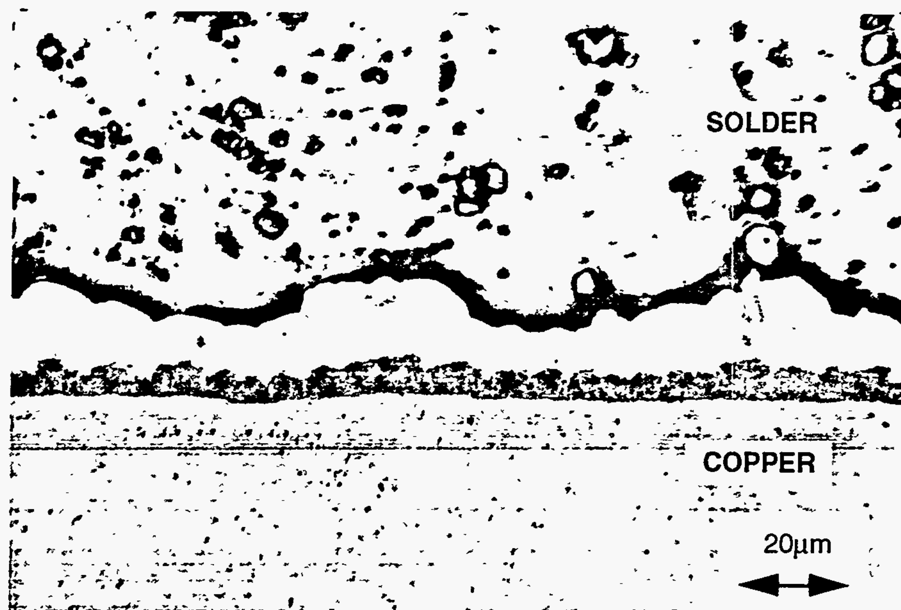
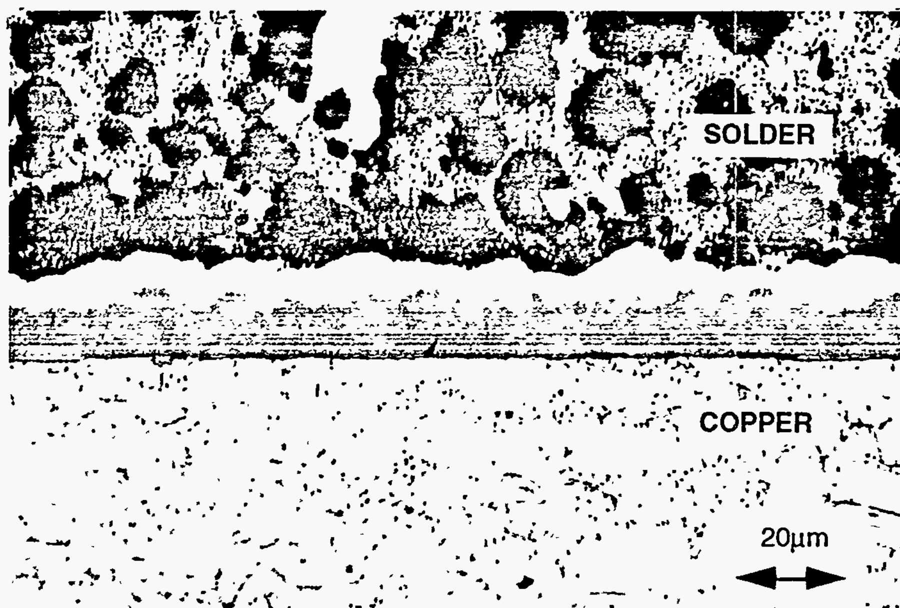


Fig. 3 IMC layer thickness as a function of √t for the 100Sn/Cu system (filled symbols) and compared with the data from the 63Sn-37Pb/Cu couple (open symbols).

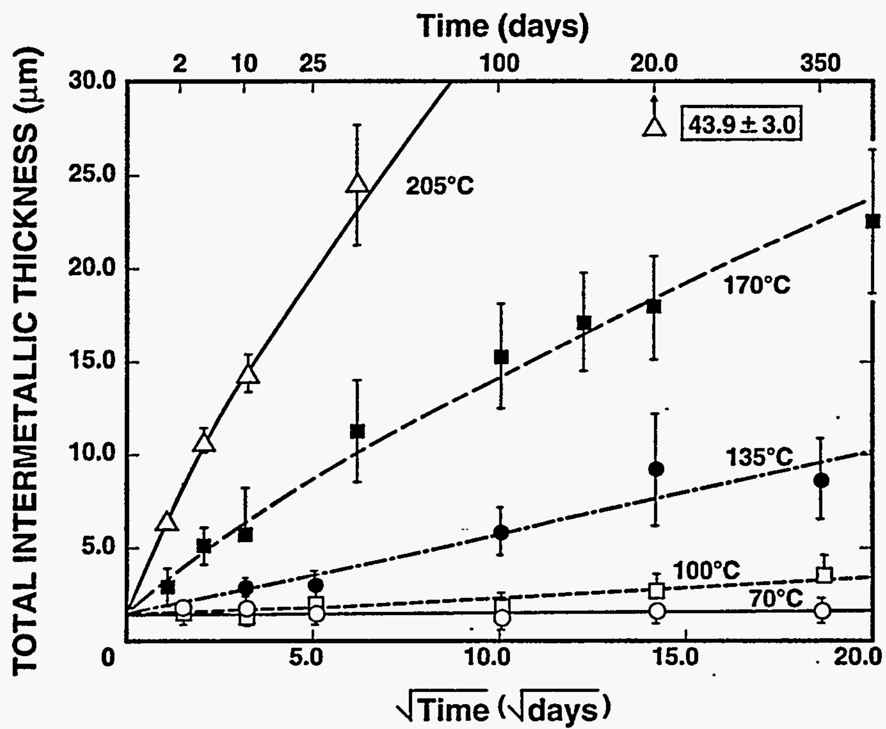


(a)

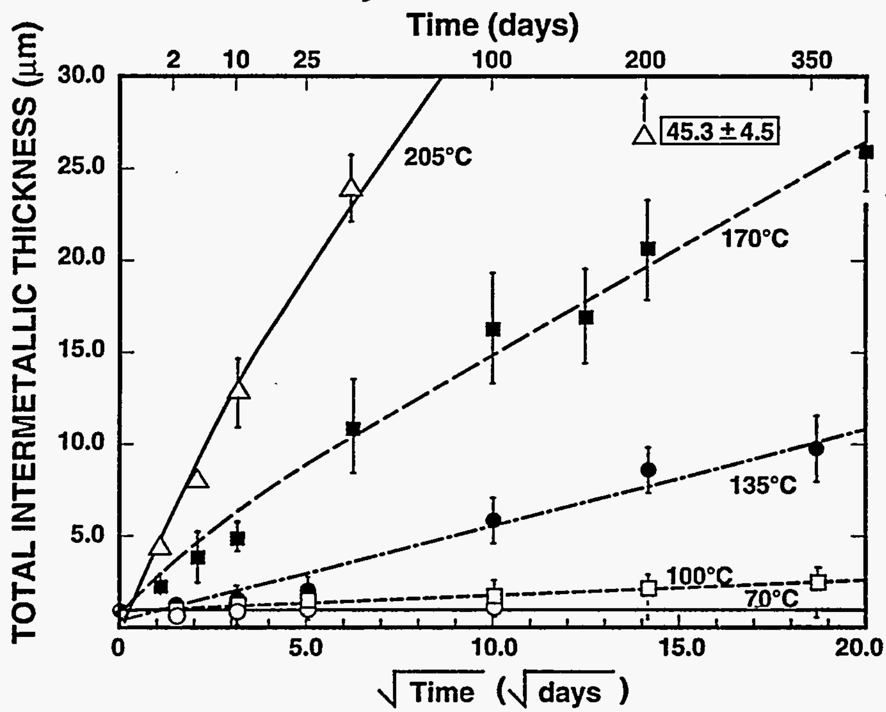


(b)

Fig. 4 Optical micrographs of the (a) 96.5Sn-3.5Ag/Cu and (b) 95Sn-5Sb/Cu systems after aging at 170°C for 150 days. An artifact of the etching process used to accentuate the IMC layer introduced appeared as a crack (a) and precipitates (b) which did not exist in either case.

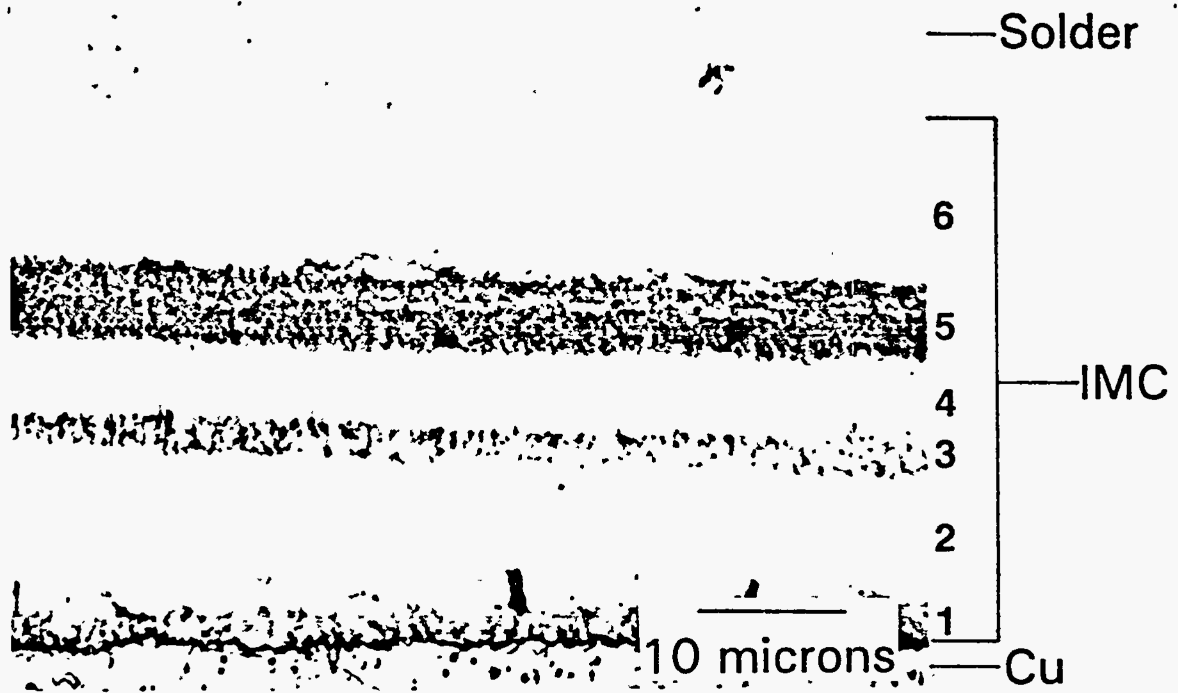


(a)

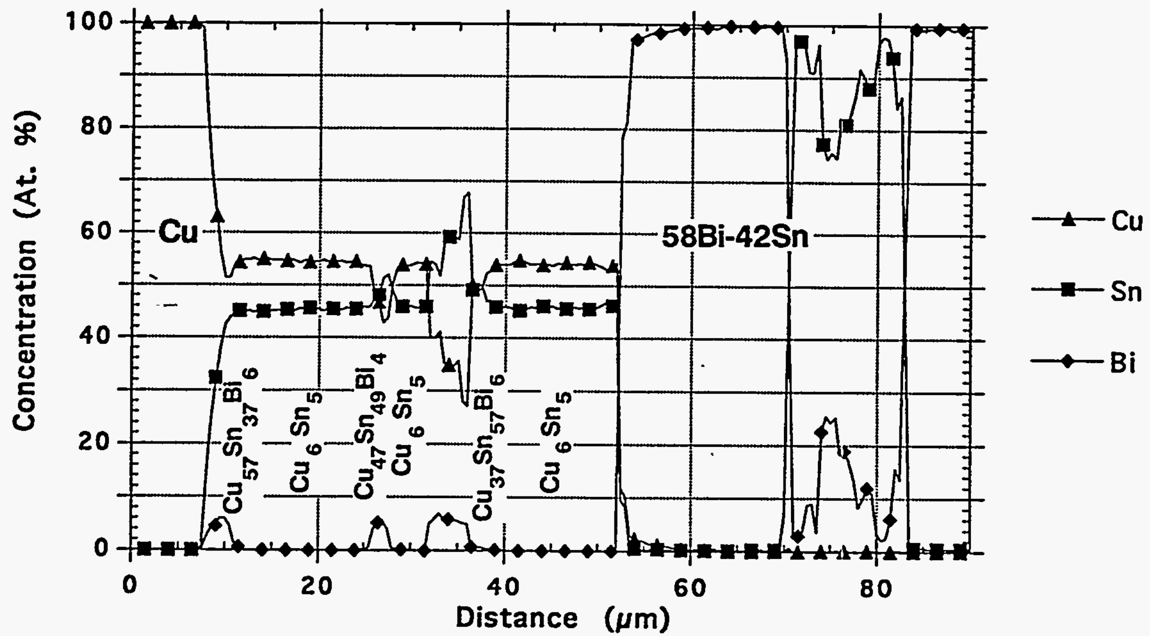


(b)

Fig.5 Growth kinetics of the (a) 96.5Sn-3.5Ag/Cu and (b) 95Sn-5Sb/Cu couples as a function of $\sqrt{t}$.



(a)



(b)

Fig. 6 (a) IMC layer of the 58Bi-42Sn/Cu couple after aging at 120°C for 400 days. (b) Electron microprobe trace across the IMC layer in (a).

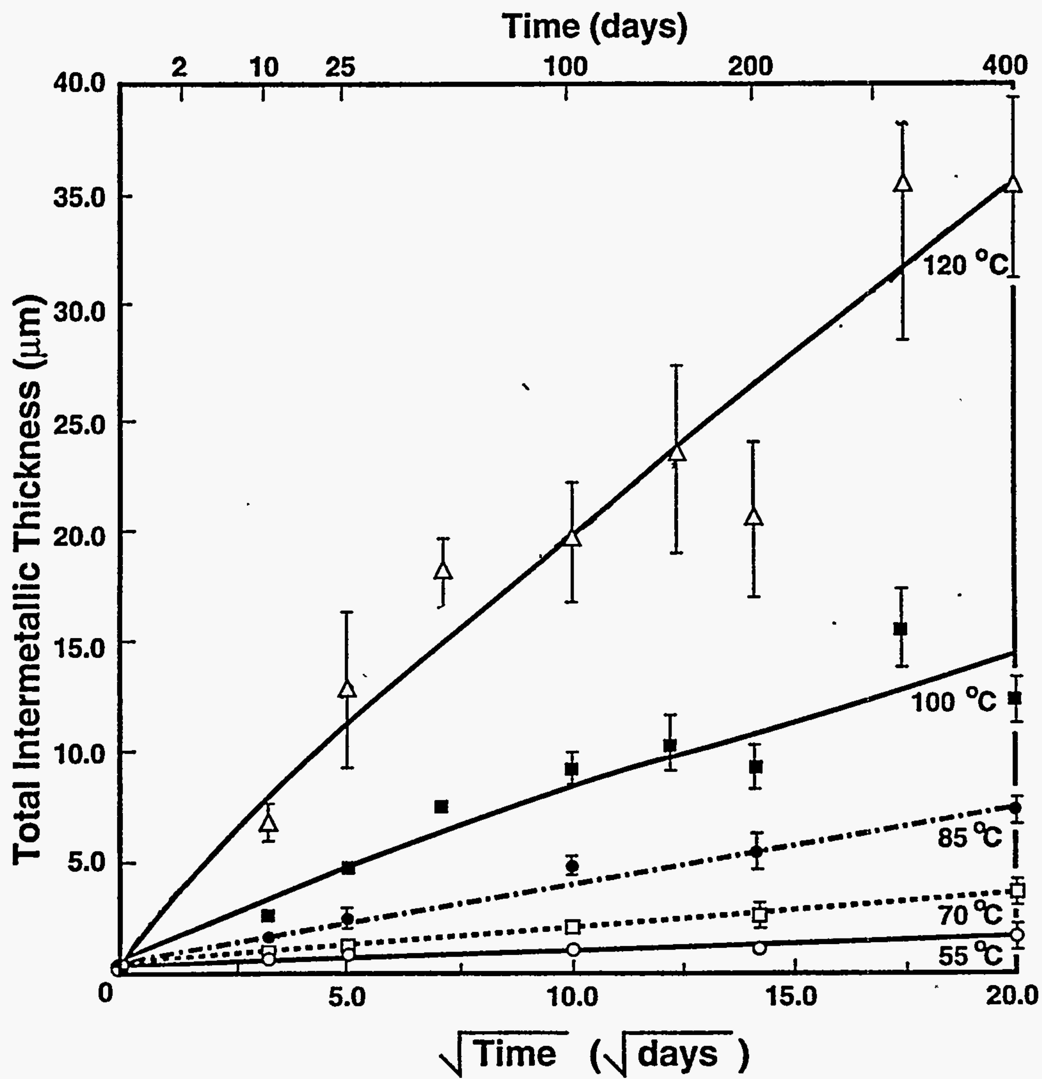


Fig. 7 Growth kinetics of the 58Bi-42Sn/Cu systems as a function of √t.

Description	58Bi-42Sn	100Sn	63Sn-37Pb
Kinetic parameters			
∘ time exponent, n	0.5 → 0.42	0.5 → 0.42	0.5
∘ activation energy (kJ/mol)	55	66	45
Absolute temp. 100C			
∘ thick. increase 100C, 400 days (microns)	14.2	3.3	4.5
∘ rate of increase at 100C ($\mu/\sqrt{day}$)	0.63	0.16	0.19
Homologous temp. 0.88			
∘ thick. increase $0.88T_m$, 400 days (microns)	7.0 (85C)	19.1 (170C)	13.2 (135C)
∘ rate of increase $0.88T_m$ ($\mu/\sqrt{day}$)	0.32 (85C)	0.89 (170C)	0.68 (135C)

Table 1 Growth Properties of Selected Solders.