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Application of push-off shear test for evaluation of wetting-interface structure-bonding relationship of solder joints

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The relationship between wetting behavior, interface structure and mechanical properties of solder/substrate couples has been studied on example of Sn-alloys and Cu substrates. The sessile drop method was used to investigate the solder wetting and spreading on polished Cu substrates in vacuum at a temperature of 503 K. The sessile drop samples after solidification were bisected perpendicularly to the substrate at the mid-plane of the contact circle. The first half of each sample was used for structural characterization of interfaces and evaluation of their mechanical properties by improved push-off shear test. The second half was used for investigation of the effect of thermocycling on structural stability and corresponding mechanical behavior of model solder/Cu joints. A comparison with the results obtained on standard solder joints has shown the usefulness of the improved push-off shear test performed directly on solidified sessile drop samples as an express test for evaluation of technological and mechanical compatibility of solder/substrate couples, particularly at the first stage of solder candidate selection.

1. Introduction

Since lead and lead-containing compounds are among the most toxic substances, a global economic forces the development of new generation solders to replace widely used lead-containing ones. However, none of the known lead-free solder alloys meets all requirements in terms of specific combination of melting temperature, solderability, thermal and electrical conductivity, thermal-expansion coefficient compatibility, corrosion and thermal-fatigue resistance, adaptivity to existing joining procedure, flexibility in designing joints. Because lead replacement problem is complicated by enormous number of interacting parameters that must be considered, a number of expensive and time-consuming tests should to be done for proper selection of the solder alloy and corresponding processing parameters. Therefore, the development of cost-effective research procedure for the selection of new generation solders is of practical importance.

The aim of this paper was to check the adaptivity of the procedure recently proposed in [1] for evaluation of wetting-interface structure-bonding interrelationship of metal/ceramic couples as express test for solder selection. In this procedure the sessile drop specimens produced in wettability test under repeatable and well-controlled conditions are used as model metal/substrate joints, which mechanical properties can be estimated by the proposed improved push-off shear test. Such procedure was selected, based upon prior experience on ceramic joining of several metal/ceramic systems (Al/AlN [2], Al/Al_2O_3 [1, 3–4], Al/Si_3N_4 [5], Al/TiO_2 [6], Ni/Al_2O_3 [7] and Cu/Al_2O_3 [8]) and importance of shear properties because the strain in solder joints of electronic interconnects is mostly shear due to CTE mismatch between the solder and the substrate [9]. For the reasons that solder joints are the weakest link in the hierarchy of the electronic interconnects and thermomechanical fatigue is the major cause of their failure [9] the effect of thermal cycling on shear properties of model solder/Cu joints has been studied.

2. Experimental procedure

The materials used were Cu substrates and several Sn-alloys, the chemical composition and properties of which are given in Table I.

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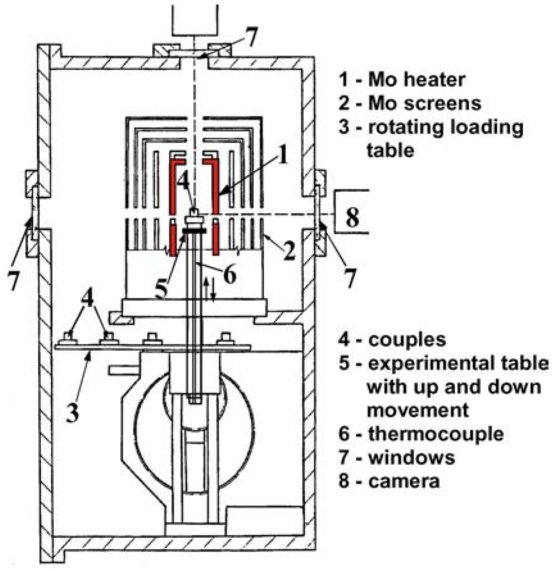


Figure 1 Experimental set up for wettability studies [9].

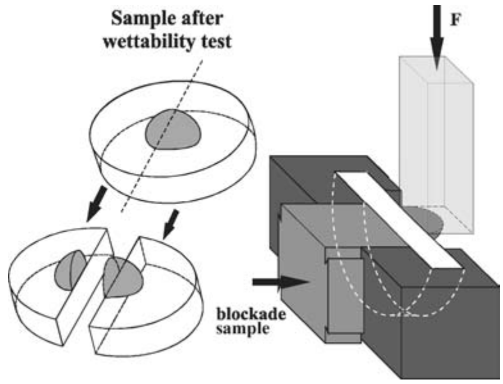


Figure 2 Schematic of improved push-off shear test of solidified sessile drop samples.

Wettability of Sn-alloy/Cu substrate couples was studied by a sessile drop method at 503 K for 5 min under vacuum of 2–3 × 10^{-6} Pa. Design of apparatus used [9] (Fig. 1) allowed to control the time of drop/substrate contact because the Sn alloy/Cu substrate couples before and after testing were stored on loading table in the cold part of the vacuum chamber. One couple was introduced inside the molybdenum furnace when experimental conditions (vacuum and temperature) were reached. The time of drop/substrate contact was measured from the moment of visually estimated melting of Sn alloy. After wettability test for 5 min, the couple was again removed into the cold part. Under conditions used the rates of heating and cooling of the couples were about 20 K/min.

Directly before wettability tests, the Cu substrates were polished up to an average roughness of a few nano-meters while the Sn-alloy samples were cleaned mechanically and next both Cu substrate and Sn-alloy samples were cleaned ultrasonically in acetone.

Solidified sessile drop samples were adopted as model Sn-alloy/Cu joints for bond strength measurements using improved push-off shear test [1], schematically shown in Fig. 2. The drop/substrate samples were carefully cross-sectioned at the mid-plane of the contact circle perpendicular to the substrate surface and polished. The first half of each sample was used for shear test while the second half of the same sample was used for structural analyses of interfaces by optical and scanning electron microscopy. After structural analyses examination the second halves of all samples were thermally cycled (1000 cycles according to temperature profile shown in Fig. 3a), next their microstructure was again examined in order to identify the structural changes involved by thermal cycling. Finally, the couples were sheared in the same way as the first halves of each sample.

In the improved push-off shear test, the sample is placed in a holder of special design and loaded in INSTRON 1115 machine. A load was applied to the flat end of the bisected couple at a constant rate of 1 mm · min^{-1} and the load versus displacement data were digitally recorded until failure under shear occurred. Assuming uniform distribution of shear stress along the line during the push-off test, the shear strength was calculated by dividing the maximum load with lateral interface area estimated from geometry of the drop/substrate contact under ×10 magnification.

3. Results and discussion

For 5 min wettability tests, the contact angles of examined alloys on Cu (Table I) are comparable with those of literature data for Sn, Sn-Ag and Sn-Ag-Cu alloys given in [9–12].

Fig. 3b shows a few examples of shear behavior of Sn alloys (as-cast and after thermal cycling) as a function of shear stress τ *vs* displacement l. Maximum shear stress on each $\tau - l$ curve presents the shear strength of

TABLE I Chemical composition and properties of selected alloys

								τ(MPa)	
Alloy	Sn	Cu	Ag	Ga	Pb	T_M (K)	θ(°) 503 K	Before TC	After TC
Sn	99.99	–	–	–	–	458.1	99	13.9	15.0
SnCu0.5	99.44	0.5	–	–	–	455.1	36	16.2	18.3
SnAg4	96	–	4	–	–	450.1	49	24.9	26.3
SnAg3.95Cu0.65	95.4	0.65	3.95	–	–	444.5	59	32.7	30.1
SnAg3.95Cu0.65Ga1	94.4	0.65	3.95	1	–	441.6	77	43.4	49.6
SnPb40*	60	–	–	–	40	456	35**	–	–

*Solder containing flux (Cynel Unipress®);
**Wettability test at 453 K in air.

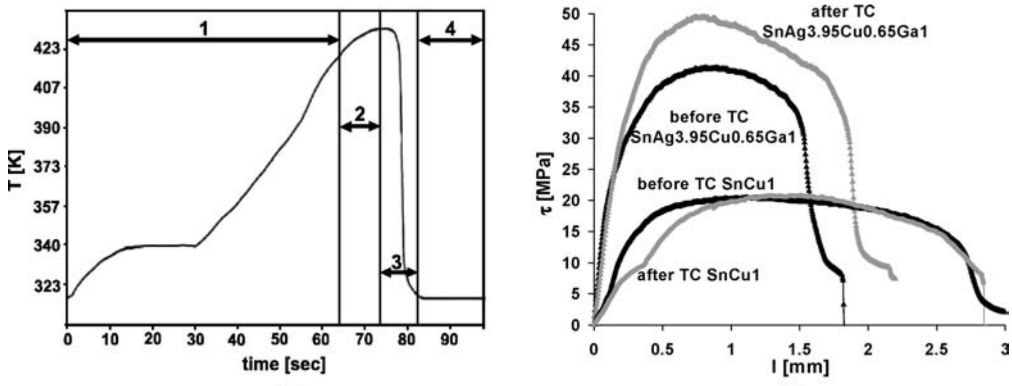


Figure 3 (a) Temperature profile of thermal cycling (TC, 1000 cycles): 1—heating, 2—movement from a furnace to a water pool, 3—cooling in water, 4—movement from a water pool and drying in air; (b) effect of alloying additions on shear behavior of selected Sn-alloys before and after TC.

the couple. The results of shear tests of examined solder alloys are collected in Table I. All alloying additions result in strengthening the Sn alloys while thermal cycling improves shear strength of SnAg3.95Cu0.65Ga1 alloy and it slightly weakens SnAg3.95Cu0.65 alloy.

Comparison of shear curves of the Sn-alloy/Cu model joints (i.e. the sessile drop samples) after wettability tests demonstrates shear behavior similar to that of corresponding monolithic alloys. Despite the fact that the Sn-alloys have about 30–34% higher shear strength than that of consequent Sn-alloys/Cu joints, at least qualitatively, the effect of alloying additions on increase in shear strength of joints raises according to the same direction as for the alloys, i.e.:

$$\text{Sn} \Rightarrow \text{SnCu0.5} \Rightarrow \text{SnAg4} \Rightarrow \text{SnAg3.95Cu0.65} \Rightarrow \text{SnAg3.95Cu0.65Ga1}.$$

However, after thermal cycling, the shear strength of the Sn-alloy/Cu couples changes in different way showing stronger bonding with the SnAg4 and SnAg3.95Cu0.65 alloys and weak bonding with the SnAg3.95Cu0.65Ga1 alloy.

Structural characterization of interfaces before and after thermal cycling suggests that the needle-like Ag_3Sn phase, nucleated at the interface of SnAg4/Cu and SnAg3.95Cu0.65/Cu couples and which amount increases during thermal cycling, might be responsible for the improved shear strength of the couples after thermal cycling (Fig. 4).

In the case of the SnAg3.95Cu0.65Ga1/Cu couples an interaction during wettability test results in the formation of two layers of intermetallic phases between the alloy and the Cu substrate. SEM analysis (Fig. 5) indicates that the substrate-side layer of about 5 μm thickness presents the $SnCu_2$ phase with about 5 at% dissolved Ga. The drop-side layer of an average thickness of 15 μm contains about 34 at% Ga and 2 at% Sn that correspond to $GaCu_2$ phase with dissolved Sn. After thermal cycling the interface of the same couple has structural discontinueties in the forms of small pores or even cracks located between the two reaction layers (Fig. 6). These structural defects can be formed during thermal cycling due to Kirkendall effect caused by uncompensated diffusion of elements across the interface and leading to the excess of vacancies in the materials with the highest diffusion rate.

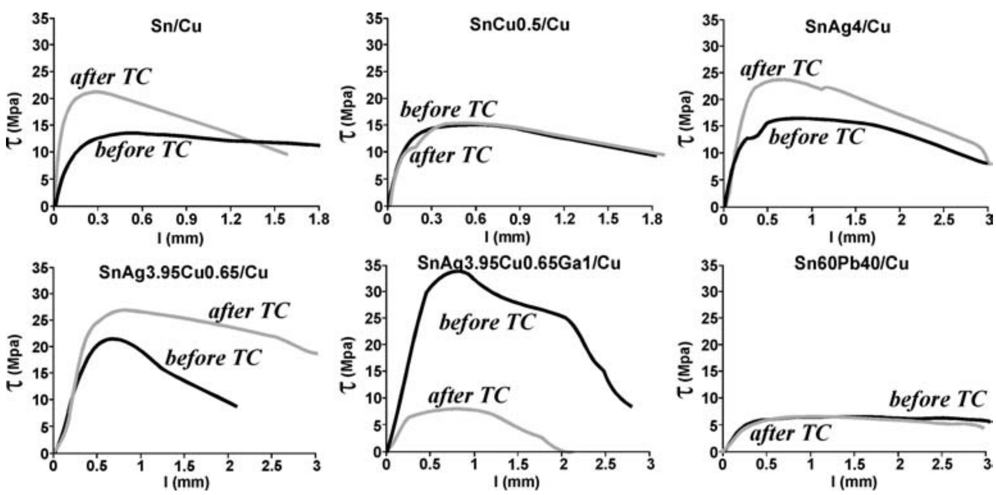
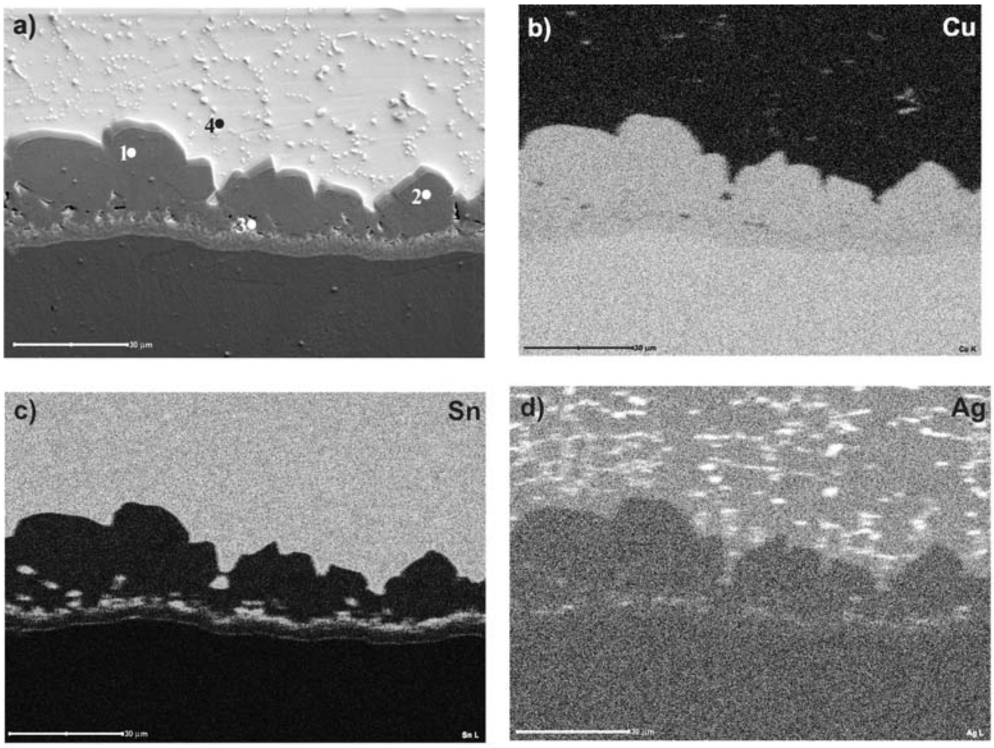
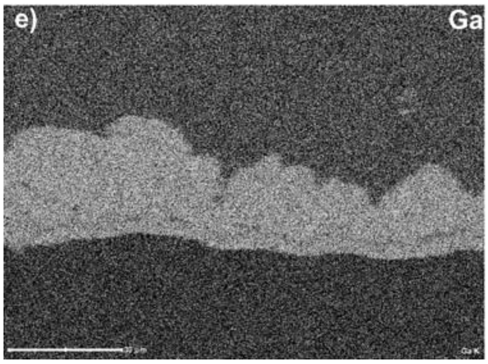


Figure 4 Effect of thermal cycling on shear behavior of Sn alloy/Cu sessile drop samples.





EDS analysis

Point	Cu mass (%)	Cu at (%)	Sn mass (%)	Sn at (%)	Ag mass (%)	Ag at (%)	Ga mass (%)	Ga at (%)
1	60.73	64.38	5.72	3.25	0.13	0.08	33.43	32.30
2	59.93	63.03	2.27	1.28	1.58	0.98	36.22	34.71
3	44.39	59.22	53.50	38.21	—	—	2.12	2.58
4	1.30	2.29	83.39	78.36	9.08	9.39	6.23	9.96

Figure 5 SEM image (a) of SnAg3.95Cu0.65Ga1/Cu interface and corresponding distributions of Cu (b); Sn (c); Ag (d) and Ga (e).

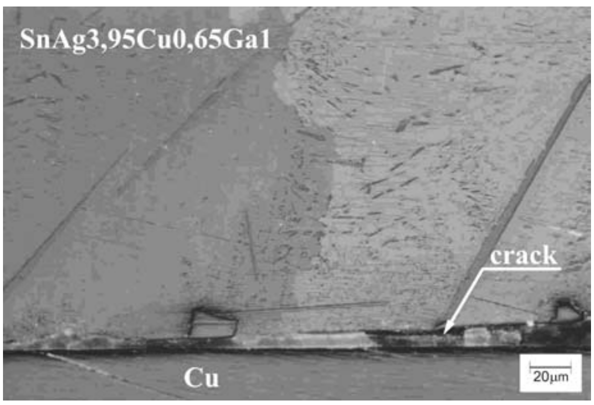


Figure 6 Microstructure of SnAg3.95Cu0.65Ga1/Cu interface after thermal cycling showing the formation of crack at the interface.

Moreover, heating during TC stimulates the vacancies to coalesce and form porosity while each cooling of TC contributes to the formation of cracks between series of neighbouring pores due to thermal stresses produced by mismatch in coefficients of thermal expansion between two contacting reaction layers. These results suggest that the formation of the Ga-rich phase at the interface is beneficial for the improvement of shear strength of the SnAg3.95Cu0.65Ga1/Cu couples. However, such couples show low resistance to thermal fluctuations. Hwang *et al.* [13] noted similar unbeneficial effect of segregation of Bi (as alloying addition to Sn-Ag solder) at the interface, which leads to weakening of the Ni/SnAg3Bi/Ni joints produced at 523–573 K for 3600 seconds and examined by tensile strength measurements.

It should be noted that despite of some possible problems to assure uniform distribution of shear stress on the interface area (that can be achieved for a perfect specimen and loading system only) and due to not taking into account the local stress concentration effects (which among others depend on the relative mechanical properties of the drop material and the substrate and on the wetting properties of the couple), the proposed improved push-off shear test delivers an important information about wetting-bonding relationship for a given group of solder/substrate couples, particularly useful on the first stage of the solder candidate selection.

TABLE II Comparison of the results of shear strength tests of Sn alloy/Cu joints with literature data

[this work]		[11]*		[12]**		[13]***		
							τ (MPa)	
							Cooling rate (K/s)	
Alloy	τ (MPa)	Alloy	τ (MPa)	Alloy	τ (MPa)	Alloy	1.5	10
Sn	14.1	Sn	22					
SnCu0.5	16.1	SnCu1	28.5	SnCu0.7	23	SnCu0.7		29.8
SnAg4	18.2	SnAg3.5	39	SnAg3.5	27	SnAg3.5	20.9	61.2
SnAg3.95Cu0.65	21.9	SnSb5	38.7	SnAg3.5Cu0.7	27	SnAg3.8Cu0.7		63.8
SnAg3.95Cu0.65Ga1	34.7	SnBi58	48.0			SnAg4.7Cu1.7	47.0	58.0
SnPb40	17.3	SnPb40	34.5	SnPb37	23	SnPb40	37.4	36.5
						SnAg3.6Cu1	54.0	67.0

*Ring and plug test, cross-head speed: 1 mm/min.
**No data on method, cross-head speed: 0.1 mm/min.
***AFPB method, cross-head speed: 0.1 mm/min, gap thickness: 76.2 μm.

Comparison of the results of shear tests with the literature data of similar couples using standard methods shows quite good qualitative relationship for selected alloys and subsequent joints with Cu, especially taking into account additional factors, such as different cooling rates, cross-head speeds in shear tests and sample size, all of which affect the values of shear strength of both solders and solder joints. The most representative results of the literature data with relatively similar conditions of processing and testing are collected in Table II.

4. Conclusions

An application of improved push-off shear test on solidified sessile drop samples to solder joints has been demonstrated. It could be a routine test, particularly when the cost and availability of the specimens are important. This test may be recommended as express test for evaluation of technological and mechanical compatibility in solder/substrate couples, especially useful at the first stage of solder candidate selection.

Among the selected Sn-based solder alloys the shear strength of model solder/Cu joints increases in the direction of corresponding solder: Sn $\Rightarrow$ SnCu0.5 $\Rightarrow$ SnAg4 $\Rightarrow$ SnAg3.95Cu0.65 $\Rightarrow$ SnAg3.95Cu0.65Ga1. Thermal cycling results in strengthening of SnAg4/Cu and SnAg3.95Cu0.65/Cu couples and in weakening of SnAg3.95Cu0.65Ga1/Cu ones.

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