Advanced hardness techniques for small-scale-joints characterization

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Introduction

The need for the design of special mechanical and metallurgical methods for small-scale joints was established in recent years. There is a rising understanding [1] that testing standard specimens does not sufficiently satisfy the knowledge on mechanical behavior of structural joints that consists of several components, either in the macro or micro scale. Two examples of joining methods could be given to demonstrate this assumption. The first example discussed here involves the soldering of miniature joints. For the last decade, the electronics industry has been giving serious thought to replacing lead-tin eutectic solders with lead-free solders. This creates a need for critical data on the industry's new lead-free solder mechanical behavior for the development of reliability models [2]. The metallurgical parameters involved have greatly influence the mechanical behavior of the joints. The quality and thus the reliability, of a joint depend strongly upon microstructure, thermal cycle, metallurgical procedures, materials, and more. One of the parameters expected to be of great importance is the size effect. Most of the available data on mechanical properties of joints alloys were obtained using standard specimens having dimensions that do not represent the actual size of a typical joint. To duplicate the microstructure (grain size and orientation) of smaller joints the large specimens are [3] used for microhardness and crystallographic measurements to assure that equivalence to small joints has been established. However, in the case of soldered joints, for example, Madeni and Liu [4] found that the recommended procedures, as well as complementary mathematical models developed, do not produce tensile test specimens that resemble actual solder joints. Variations in the general metallurgical history of the test specimens are probably the main reason for the wide range of mechanical properties values in the literature [5-7]. Cheng and Siewert [8] also suggested that the reason for the wide fluctuations in mechanical parameters (around room temperature) could be the temperatures at which the solders were tested. These temperatures, higher than half of the solders' homologous temperature, and viscoelastic/viscoplastic deformations, which are rate-sensitive, become important and significant.

The second example is that related to the magnetic pulse welding (MPW) process. This process was developed in the late 1960s and early 1970s [1]; MPW is identical to explosive bonding in the formation of the joint. But, instead of chemical explosive energy, it uses magnetic fields to drive the materials together. The technology is on the verge of becoming a high-volume production process, being ideal for electrical, automotive and aerospace applications [9]. Typically, any round part such as a tube-to-tube joint, a tube-to-end joint or a wire crimp joint would be an ideal candidate for MPW. It has gained the attention of the welding community because it enables the joining of similar as well as dissimilar materials in microseconds, without the need for any costly consumables. The emphasized interest is the ability to join materials that are metallurgically incompatible or sensitive to heat input, as this welding process creates a very narrow fusion zone. The process is fast, clean, very energy efficient and creates no heat affected zone to change the properties of the welded components [9-13].

A possible suggested solution was to test the connected joints using "miniature" specimens having dimensions as close as possible to those of typical real-life joint and focus on the interface and the microstructure that is in close proximity with it. There is a need to use dedicated methods (such as the new variations of hardness tests) having the resolution to conduct test across the interface and its surroundings and get results from each individual component of the joint. This might be used in conjunction with tensile testing in various strain rates and temperature ranges, while maintaining "constant" parameters such as size, shape, and microstructure (thermal cycle) of the specimens, as a complimentary method that enables gaining some more knowledge on the mechanical properties of joints made using different methods, soldering and magnetic pulse welding (MPW), in this case.

Experimental and analysis

Nanoindentation and micro-hardness measurements and analysis

Measurements of hardness and modulus were made by instrumented Nanoindentation (Nano Indenter XP, MTS Systems® Oak Ridge, TN, USA), using a Berkovich indenting tip (Micro Star technologies®, USA). The integrated optical system with ×40 magnification was used to locate and define the testing positions. Prior to each experiment the indenter head was calibrated with the optical view to ensure stage movement accuracy and the precise placement of the indenter tip. Indentations were performed using "Continuous Stiffness Method" (CSM) whereby a small (2 nm) modulation is applied to the indenting tip and the instantaneously modulus and hardness are calculated continuously using the known tip area function. Indentations were performed at a constant strain rate of $0.1 s^{-1}$ under displacement control, with maximum displacement set to 200 nm, the minimal depth required to get consistent results, without influence from local surface roughness. Under these conditions, the entire loading cycle is finished within about 100 seconds. Values were corrected for thermal drift by measuring the drift at 10% of maximum load during the unloading segment. Typically, a series of indentations were programmed to cross from one phase through the interfacial region and into the other phase. A gap distance of 2 µm was allowed between indentations to avoid interference from the plastic deformation of the neighboring indents.

Four basic equations [14] are used for the calculation of indentation modulus and hardness:

$$E_r = \{[(1-\nu^2)\, E^{-1}]_{sample} + [(1-\nu^2)\, E^{-1}]_{indenter}\}^{-1} \quad (1)$$

$$E_r = [S\, (\pi)^{1/2}] \,/\, [2\beta\, (A)^{1/2}] \quad (2)$$

$$h_c = h_{max} - \varepsilon P_{max} (S)^{-1} \quad (3)$$

$$H = P_{max}\, (A)^{-1} \quad (4)$$

where E_r is the reduced modulus of the indenter/sample system, E is Young's modulus, ν is Poisson's ratio, P_{max} is the maximum applied load, S is the contact stiffness, A is the contact area at a given contact at the depth h_c over which contact exists, h_c is related to the indentation depth hmax by eq. (3), with ε a geometric factor related to indenter shape, β a small correction factor, H is the nanoindentation hardness (note that this differs from microhardness as area at maximum load is considered rather than relaxed load).

Nanoindentation and conventional micro-hardness tests were conducted, (a) Across the interfaces of miniature joints of copper soldered using Sn-0.7Cu lead-free alloy; the specimens were in the as-received, pre-deformed in various strain-rates, and, aged (150 ^oC for 1000 h) conditions; the tests were conducted at the intermetallic compound (IMC) layer along the interface, at the IMC precipitates in the eutectic solder, and, at the eutectic solder; (b) Across the interface of a MPW joint of Al-A1050 alloy to Mg-AZ91 alloy.

Instrumented hardness measurements

This method is based on acquiring load and displacement (depth) data during the indentation of a spherical 0.5mm ball into the metal. Load and depth are used to calculate the mechanical parameters and plastic deformation of the metal tested. This method was used to obtain the mechanical properties of lead free solder alloys following an experimental simulation of the soldering process: (a) Sn-4Ag; (b) Sn-0.7Cu; and, (c) Sn-4Ag-0.7Cu. The evaluation [15] of the estimated mechanical parameters of the plastic deformation is based on using Holomon's equations:

$$\sigma = K\, \varepsilon^n \quad (5)$$

where, K and n are the strength coefficient and the strain hardening exponent, respectively, σ is the flow stress, and, ε_p is the plastic strain.

Eq. 5 can be simplified for $n = e_p$ when the flow stress reaches its maximum value (ultimate tensile stress - UTS); e_p is the plastic strain at the UTS,

$$\sigma_{UTS} = K\, (n\, e_p^{-1})^{r} \quad (6)$$

The yield stress is calculated using the relation between the indenter load P, measured continuously during the test,

$$\sigma_y = B_m\, A \quad (7)$$

$$P_i\, d_i^{-2} = A\, (d_i\, D)^{m-2} \quad (8)$$

where:

B_m and A - material constants;
d_i - indentatation diameter;
D - indenter diameter.

The hardness, HB, value is obtained using Eq. 9:

$$HB = 2P_{max}\, (\pi\, D)^{-1}\, (D-(D-d_{max}^{2})^{0.5})^{-1} \quad (9)$$

Results and discussion

Nanoindentation and micro-hardness

A typical load-displacement curve, obtained from a nanoindentation test is shown in Fig.1 and an example of the indentations, made across the intermetallic layer of a soldered joint, is shown in Fig. 2. Hardness values obtained from

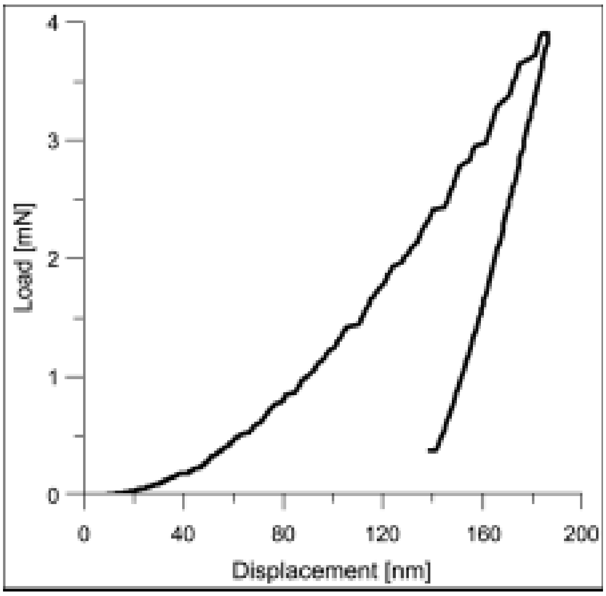

Figure 1. Typical load-displacement curve obtained from nanoindentation test of a Cu_6Sn_5 intermetallic scallop in the interface.

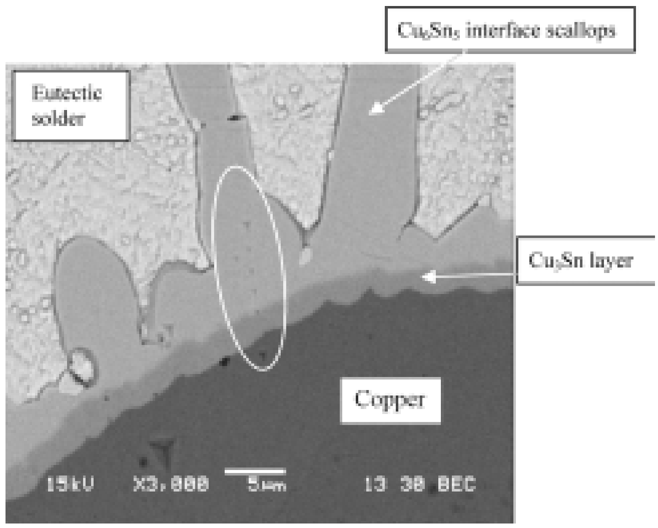

Figure 2. SEM micrographs of nanoindentation tests across the interface (copper substrate-Cu_3Sn intermetallic layer-Cu_6Sn_5 scallop) of an aged (1000h@150^oC) miniature soldered joint.

nanoindentation measurements are shown in Tables 1. Table 1 shows results from as-soldered and aged miniature joints.

Results from experiments with as-soldered miniature joint components, deformed to fracture in tension at three different strain-rates, where measurements were carried out at areas of maximum plastic strain, as close as possible to the fracture face of the miniature soldered joints, are summarized in ref. 16. Measurements near the fracture face in the eutectic solder revealed a striking dependence of nanoindentation modulus on strain rate. This parameter showed a strong positive correlation with strain rate. The hardness rose with strain rate and was more than 100% larger for the fastest rate compared to the slowest rate. The possible explanations for this phenomenon were published earlier [16].

The eutectic solder obviously absorbed most of the plastic deformation being the soft weakest link of the joint while some of the intermetallics, almost intact, "slide" relatively to the eutectic solder. Eutectic solder can be treated as a "composite" where the intermetallic phases, (Cu_6Sn_5) shaped as plates or small particles along grain boundaries, have a strengthening effect and the indentation modulus and hardness are "apparent" values (an "average" of the values typical to pure solder or intermetallic); this assumption can explain the changes in the mechanical parameters measured when (a) solder flows differently than intermetallics under different strain rates, and, (b) changes in microstructure as a result of environmental conditions (time and temperature effect on grain size). Nanoindentation proved to be a good, fairly sensitive method to determine all occurring structure parameters when testing miniature lead free soldered joints.

Table 1. Hardness values, obtained from nanoindentation measurements in as-soldered and aged miniature joints components. Each value is the mean of at least 8 indentations.

	nanoindentations location	hardness, [GPa]
As soldered	Cu_6Sn_5: interface scallop	5.6±2.1
	Cu_6Sn_5: precipitate in eutectic solder	4.8±1.9
	Eutectic solder	0.3±0.1
Aged, 1000h@150°C	Cu_6Sn_5: interface scallop	6.2±1.9
	Cu_3Sn*: layer between the substrate and the Cu_6Sn_5 layer	7.2±0.3
	$Cu_6Sn_5$5: precipitate in eutectic solder	4.8±2.0
	eutectic solder	0.4±0.1

A discontinuous pocket type or a continuous interfacial layer was formed during MPW of Al-A1050 to Mg alloys (Fig. 3). The bonding interfacial layer is the product of local melting and rapid solidification and consists completely of IMC, identified as $Mg_{17}Al_{12}$. In all cases, the most significant feature of the transition zone created during the welding process is the high hardness of the interfacial layer (Fig. 4). The increase in hardness is a result of the formation of IMPs and the fine-grained microstructure [9-13]. The hardness, measured using nanoindentation in the interfacial layer, was 3.3±1.5 GPa.

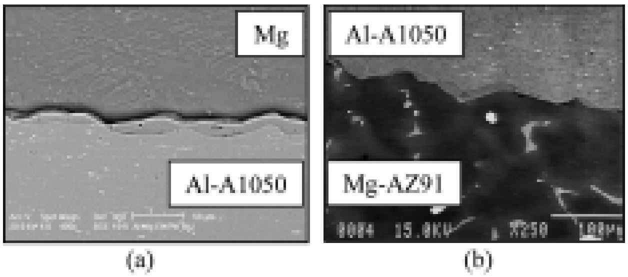

Figure 3. Micrographs of a (a) discontinuous pocket type, and, (b) continuous interfacial layer, formed during MPW of Al-A1050 alloy to Mg-AZ91 alloy.

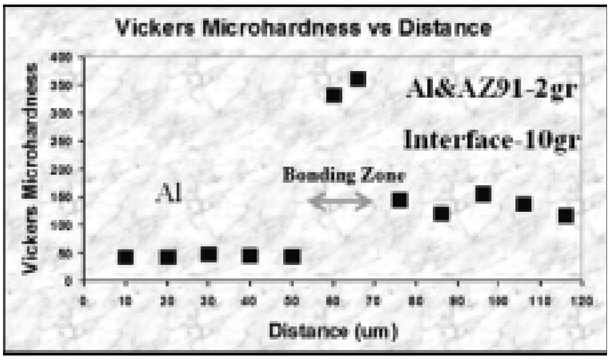

Figure 4. The high hardness of the bonding zone at the interfacial layer created during the MPW.

Instrumented hardness measurements and analysis

Load and depth data, acquired during instrumented tests in the eutectic solder of lead free soldered alloys were analyzed and the mechanical parameters were obtained (Table 2). The results can be used as part of an evaluation of the mechanical response of the soldered joint, where its eutectic solder component is considered as the *weakest link*.

Table 2. Mechanical parameters of the plastic deformation obtained from instrumented hardness test.

	Yield Stress, [MPa]	UTS, [MPa]	n	K, [MPa]	HB, [MPa]
Sn-4Ag	22.7 ±0.1	47.4 ±2.6	0.201 ±0.009	80.2 ±5.7	140 ±4
Sn-0.7Cu	14.9 ±0.2	30.0 ±0.7	0.195 ±0.005	50.7 ±1.1	91 ±0.5
Sn-4Ag-0.7Cu	19.3 ±2.3	38.8 ±1.2	0.190 ±0.010	64.8 ±0.8	118.0 ±8.5

Conclusions

As mentioned before, the main goal of this research was to evaluate advanced hardness test methods for data acquisition and analysis from small-scale interfaces in joints. Examples of applications of these methods in soldering and MPW were given. This goal was satisfied, as demonstrated by:

- Nanoindentation can be used to obtain specific mechanical parameters from ***individual microscopic components*** (e.g. intermetallics compounds) across any area of a connected joint;

- Instrumented hardness results from ***macroscopic components*** of a connected joint can serve as a first-order data source for useful mechanical parameters;
- Micro-hardness is a traditional test method that yield svaluable results from ***macroscopic components*** of the joints;
- Combination of specially-designed mechanical testing, accompanied by other test methods (such as tensile testing of miniature specimens); can serve as a reliable approach for obtaining the overall response of joints, made almost by most of the recognized joining methods.
- The use of microhardness, combined with nano-indentation, was sufficient to reveal the gradient of properties across MPW joints of Al alloy to Mg alloy.

This research was motivated mainly by the importance of full characterization of the elastic and plastic deformation behavior. In order to determine the mechanical response of a miniature lead-free soldered joint, it is essential to collect data from experiments that test the overall mechanical properties of the soldered joint and its individual components. The combination of mechanical testing (a must for determination of a complete stress-strain curve) and nano-indentation (for measuring local hardness) in different metallurgical conditions is highly recommended.

As-soldered, aged and deformed-to-fracture joints were used to explore the possible effects on nanoindentation hardness. Noticeable changes in indentation hardness were observed in the eutectic solder; this was found in an area far from the substrate interface and near the fracture faces (area with maximum plastic deformation) in post-tension joints. The major reason for this behavior was related to the nature of the eutectic solder where rearrangement of its phases during plastic deformation is proportional to strain-rate.

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