ESRF	**Experiment title: Temperature resolved microstructural characterisation of Pb-free solder joints using fine-focus X-ray diffraction**	**Experiment number**: ME532
Beamline: ID11	**Date of experiment**: from: 25/04/2003 to: 29/04/2003	**Date of report**: 16/04/04
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Report

The reliability and ultimate performance of a solder joint is hugely dependent on intermetallic layers located at the interfaces between the joint and circuit board surface pads and the electronic device. This introduces a requirement for a method that detects and characterises intermetallics within the solder joint. Our special interest in using the European Synchrotron Radiation Facility was to exploit the potential for a non-destructive application to detect intermetallics in Pb-free solders using fine-focus X-ray diffraction; traditional methods for intermetallic detection are destructive. Furthermore, temperature resolved experiments that allow intermetallic detection during solder joint formation are possible on ID11 by virtue of a fast capture X-ray detector coupled with micron stepped sample translation and an in-situ heating/cooling stage.

Key successes from this project include (a) Successful gathering of in-situ temperature resolved data for a Pb-free solder joint, (b) development of software to analyse the generated data and (c) the dissemination of the research at two international conferences and the submission of a journal paper.

The analysis of the data is continuing and an MSc project is currently working on this at the University of Greenwich. The development of the software took longer than anticipated, but the data analysis is progressing and results from this will be presented at the Material Science and Technology in New Orleans in September 2004. Dialogue is also underway between the partners to submit an EPSRC proposal and to bid for further beam time to build upon the results so far obtained.

Experimental methods

The experiments were conducted using the micro focus XRD apparatus to detect and to map intermetallic composition in small lead-free solder joints. High energy X-rays (70keV) were used so that penetration of the solder and circuit board was achieved. Model systems, with known intermetallic compositions, were chosen to explore and demonstrate the capability of non-destructive intermetallic detection and temperature resolved experiments using XRD. The samples comprised two small (1mm diameter) single Pb-free solder bumps, with SnAgCu composition, each soldered to either a Cu or a Ni/Au surface pad along with samples that had been isothermally aged; in total over 30 pre-prepared samples corresponding to a variety of solder joint systems were utilised. The experimental configuration of the experimental set up is shown in figure 1.

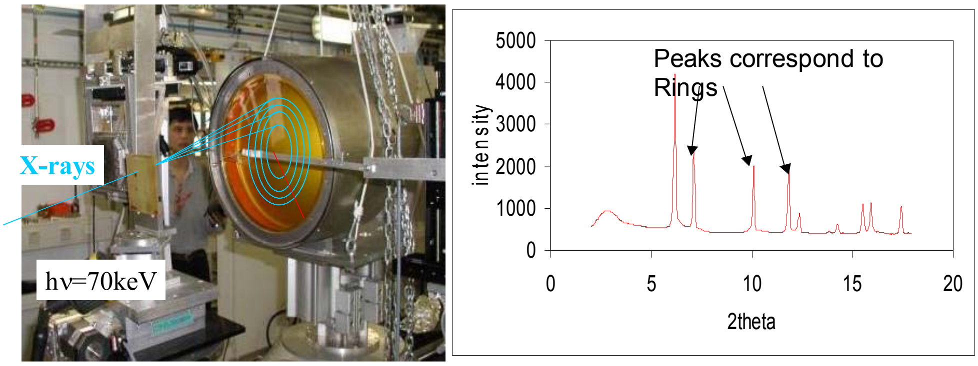

Figure 1: Experimental configuration for XRD on ID11 and an example of a ‘caked’ 1D diffraction pattern.

The 2D diffraction data captured by the X-ray detector for each position in the solder joint, was analysed as follows, each of the rings corresponds to a diffracted peak from a characteristic crystal, i.e. 111 planes from Cu in Cu_6Sn_5 compounds. The ring formation is generated as each micro-crystal of a particular compound diffracts X-rays by angle 2theta (2θ), which corresponds to a particular radius on the 2D detector. A complete ring formation was achieved for fine grain structures because each particular crystal is oriented in many directions in the sample. For larger grain sizes, such as the bulk solder, spots were formed on the 2D diffraction pattern and a complete ring pattern was not always achieved. Our aim was to utilise the XRD technique so that different phases in the solder joint could be detected in two dimensions by detecting the diffracted rings. A scan matrix then allowed the collection of 2D diffraction patterns at selected locations in the solder bump in 5µm steps. The 2D diffraction patterns (rings) were then caked into 1D diffraction patterns (peaks) so that information on the intermetallic compositions as a function of composition and distribution could be obtained.

Results

Information was collected from the 2D pattern by ‘caking’ the data to form 1D 2-theta verses intensity plots, an example is shown in Figure 1. The data caking takes an average of the ring intensity around the entire radius to produce a single peak on a 1D plot. The 2-theta value of this peak is then compared with the measured 2-theta values from the Joint Committee of Powder Diffraction Standards (JCPDS), which then allow positive identification of the phases present. After positive identification of a particular intermetallic species, spatial distributions over the entire bump were then plotted. An example of the Cu_6Sn_5 intermetallic distribution is shown in figure 2. The intermetallics are distributed at the solder/surface pad interface and at the surface of the solder bump. Furthermore, the resolution of the distribution plots was around 5µm, which was made possible by the narrow X-ray beam on ID11.

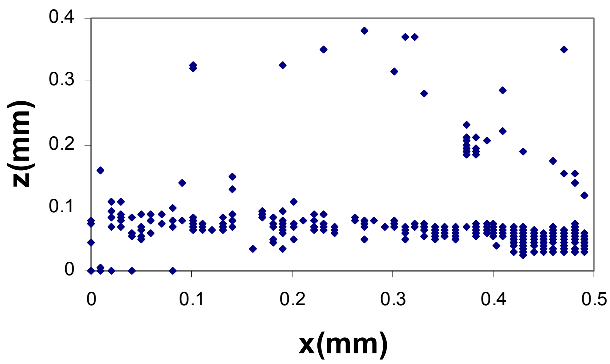

Figure 2: Distribution plot of intermetallics in a Pb-free solder bump.

The XRD technique was also utilised in a temperature resolved mode to observe the formation of intermetallic compounds, in situ, during the solidification of the solder. In this experiment, the samples were prepared by printing solder paste onto Cu pads, which were then placed on a hot-plate in the beamline’s experiment chamber. The hot-plate was controlled remotely away from the beamline hutch and thermal couples were attached to the samples so that the temperature could be monitored. The samples were scanned, in the X-ray beam, as the temperature was increased to reach the solder melting temperature and then allowed to cool. A broad thin beam (100×5μm) was used to scan the sample along the vertical direction of the solder deposit. XRD scans were recorded during the solder joint formation process at the solder/solder pad interface so that the growth sequence of the IMC could be investigated, a complete solder melting and solder joint solidification sequence is shown in figure 3.

Figure 3a displays diffraction rings from the Cu surface pads, the complete ring formation is generated as electrodeposited Cu has a very fine grain structure. Figure 3b shows the diffraction pattern from the solder paste prior to any heating, in this case the sample has been moved in the z direction so that the X-rays are incident on the solder paste. The observed diffraction pattern is typical of powder diffraction from small particles. As the solder paste melts to form molten solder figure 3c displays the diffraction pattern typical of a liquid, i.e. anomalous short-range order. As the solder bump begins to cool initial formation of the IMC's are detected, in figure 3d spots corresponding to IMC are shown to co exist with the molten solder. Furthermore, figure 3e displays the IMC's in the initial formation stage at the Cu pad interface. Finally, figure 3f displays the IMC at the solder pad interface after cooling.

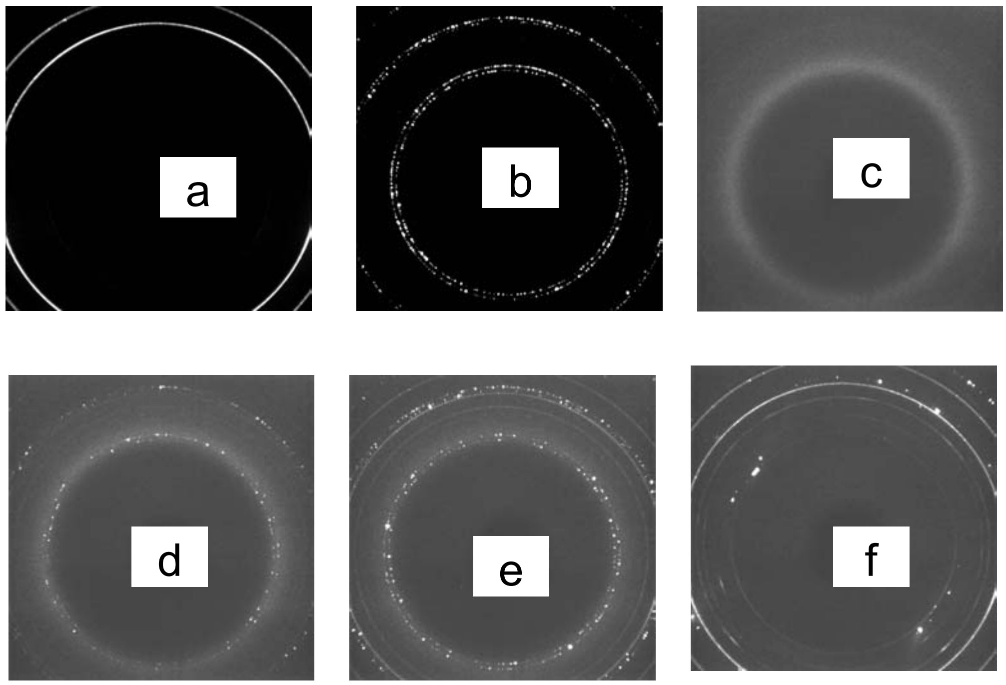

Figure 3: In situ melting and solidification experiment. a) XRD pattern of Cu pad, b) XRD pattern of Sn in solder paste, c) molten solder, d) crystalline structures appear shortly after melting, e) during solidification, f) PCB pad / solder interface after solidification.

Conclusions

Intermetallic phases were successively detected 'non-destructively' within our test models. Our data does demonstrate the validly of the fine-focus XRD method, on ID11, for this identification. The preliminary work on the in situ temperature resolved experiments shows that it is possible to detect IMC, as a function of solder joint preparation, i.e. heating and cooling. The sequence of images in the temperature resolved experiments have captured the initial growth formation of the intermetallics in the molten solder. Further work, using this method, would be able to assess the formation of the intermetallics in more detail to assess the sensitivity of using alternative Pb-free solders, solder joint configurations and heating/cooling profiles.

Publications

[1] G.J.Jackson, H.Lu, *et al*. '*Intermetallic Phase Detection in Lead-free Solders Using Synchrotron X-ray Diffraction*', submitted to Journal of Electronic Materials, March 2004

[2] Details of this work have also been presented in a Pb-free solder session at the 2004 annual TMS symposium, March 14-18th, Charlotte, NC, USA

[3] At the invitation of Materials Science and Technology (MS&T) 2004 conference organisers (TMS), a paper will be submitted detailing this work at the next MS&T conference in New Orleans, September 2004.