ESRF	**Experiment title: Detection and characterisation of intermetallic compounds in Pb-free solder joints using high-energy micro-focus X-ray diffraction**	**Experiment number**: ME361
Beamline: ID11	**Date of experiment**: from: 25/04/2002 to: 29/04/2002	**Date of report**: 31st August 2002
Shifts: 12	**Local contact(s)**: Dr. Gavin Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Gavin Jackson*+1, Rajkumar Duriairaj*+1, Ndy Ekere+1, Mike Hendriksen*+1 Nick Hoo*2 Chris Bailey3, Hua Lu*3

1. Electronics Manufacturing Engineering Research Group, Medway School of Engineering, University of Greenwich, Chatham Maritime, Kent ME4 4TB, UK

2. Tin Technology (ITRI), Kingston Lane, Uxbridge, Middlesex UB8 3PJ, UK

3. Computing and Mathematical Sciences, University of Greenwich, Park Row, Greenwich, London SE10 9LS, UK

+Previously at: School of Aeronautical, Civil and Mechanical Engineering, University of Salford, Salford M5 4WT, UK

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 the intermetallic composition (IMC) within the solder joint.

Traditionally, intermetallics in solder joints are detected 'destructively' via cross-sections and optical observations. However, once a destructive test is made the evaluation cannot be continued. For example, further ageing and independent joint strength tests will not be possible.

Our special interest in using the ESRF is to exploit the potential for non-destructive intermetallic detection in new Pb-free solders using fine-focus X-ray diffraction. Different phases existing within the solder joint/pad arrangement will be shown by diffracted X-rays at a particular value of two-theta (the diffraction angle). The undulator on ID11 provides the high energy in order to penetrate the solder material and also the high spatial and spectral resolution required to exploit the diffraction effect; ID11 also provides a fine-focussed beam and micron-stepped sample manipulation, which is vital for IMC detection. Furthermore, 2-dimensional full area diffraction patterns will provide information on the micro-texture, such as grain size and preferred growth orientations for the bulk solder and intermetallic compositions; traditional methods for intermetallic phase characterisation don't report on this micro texture.

Results

Model systems were chosen to explore and demonstrate the capability of non-destructive intermetallic detection using XRD. The samples comprised two small (1mm diameter) single Pb-free solder bumps, with Sn/Ag/Cu composition, each soldered to a Cu and an Electroplated Nickel Immersion Gold (ENIG) surface pad. Traditional destructive cross-section methods (Harris et al, Soldering & Surface Mount Technology, 10/3

(1998) 38-52) combined with Scanning Electron Microscopy (SEM) have reported that these systems comprise of a β-Sn solid solution with dispersed γ-Silver Tin (Ag_3Sn), $Cu_{10}Sn_5$ intermetallics in the bulk of the solder bump and η-Tin Copper (Cu_6Sn_5), ε-Tin Copper (Cu_3Sn) intermetallics at a Cu pad interface and Ni_3Sn_4, Ni_3Sn_2, Ni_4Sn at the Ni pad interface. Our initial objective was to test these results and validate the whole idea of the non-destructive XRD method

Figure one displays our experimental results from the SnAgCu solder on Cu system, in the form of 1-dimensional diffracted X-ray spectra. The data was collected at (x,z) points in the solder joint starting from the interface and moving upwards (z-direction) in 5µm steps into the bulk. At the substrate side of the interface, point (0,5), the diffracted peaks correspond only to values indicating Cu. As the X-ray beam is moved upwards into the interface/bump region (0,10) & (0,20), diffracted peaks are observed from the eta-Tin Copper intermetallic phase. Moving further upward (0,30), gamma-Tin Silver and beta-Sn peaks are detected. This result is consistent with data obtained from the destructive methods and theoretical considerations.

After positive identification of IMC from the diffracted data, spatial distribution plots for each peak over the entire bump were then obtained. Copper, eta-Tin Copper and gamma-Tin Silver peak spatial distributions are shown in figure two, it can be clearly seen that the Cu and eta phase reside at the interface whilst the gamma phase is dispersed throughout the solder bump. This method was also used for the solder on the ENIG surface, see figure 3. Further X-ray diffraction experiments were performed on aged samples, these being Pb-free solder bumps on Cu and ENIG pads that had been isothermally heated at 450K for 150 hrs. The results showed that a much thicker IMC layer had grown from the interface into the bulk.

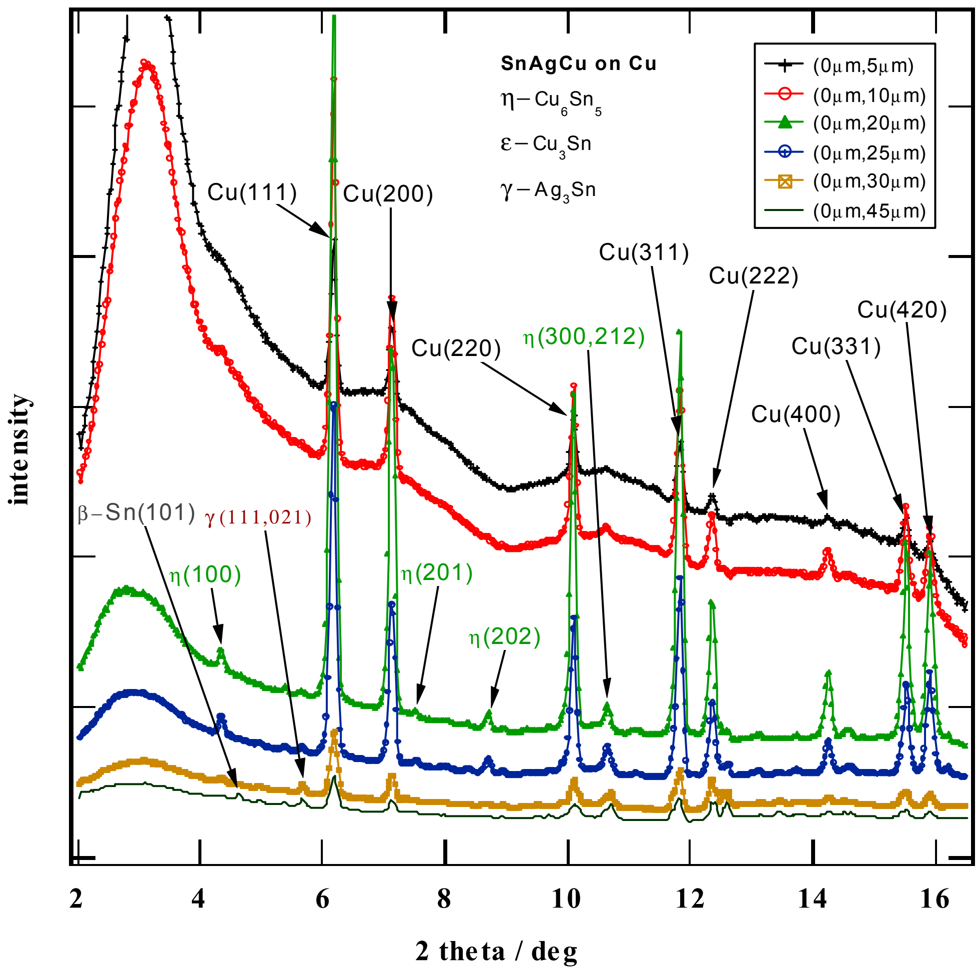

Figure one: 1-dimensional XRD peaks from the interface region of a SnAgCu alloy soldered onto a Cu pad. Comparisons between the measured 2-theta values and ones from the Joint Committee of Powder Diffraction Standards (JCPDS) allowed positive identification of the present phases.

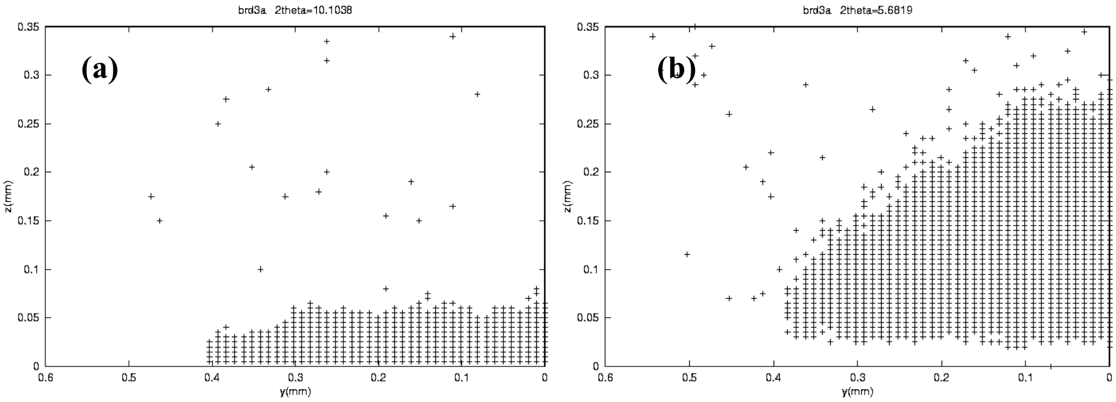

Figure two: Spatial distribution plots of diffracted peaks from: **(a)** eta-Tin Copper phase **(b)** gamma-Tin Silver phase. It can be clearly seen that the eta phase is confined to the Cu interface region whilst the gamma phase is present throughout the bulk.

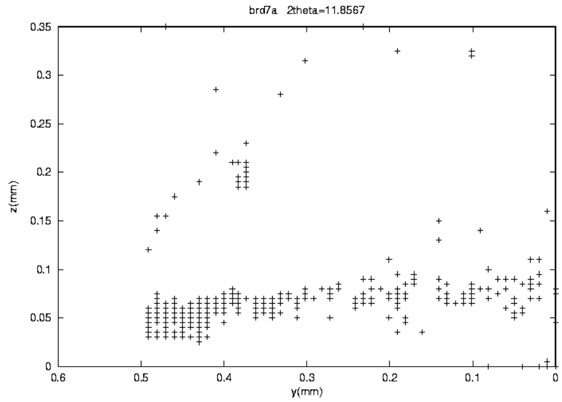

Figure three: Spatial distribution plot of diffracted peaks from NiSn intermetallic

Information regarding the microstructure of the joints was examined using the 2-dimensional full area diffraction patterns. The eta, gamma and Cu phases displayed sharp ring patterns in the 2-d data, suggesting a fine grain size and smooth texture. Conversely the beta Sn and Ni compounds displayed more spotted regions in the 2-d diffraction pattern, thus indicating lager grains and a granular texture. This information is extremely useful when correlating micro texture with solder joint preparation methods.

Further measurements were also made on immersion Ag pad finishes along with additional samples that had been further aged, these data are currently being analysed.

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. Our rather encouraging results make this technique very attractive as it allows for non-destructive investigations to be performed on expensive state-of-the-art electronic components, thereby allowing these new materials to be characterised. Additional information on the micro texture can also be gleaned by this method.

Further work has been planned, by virtue of a second proposal for beamtime on ID11. We aim to perform in situ temperature resolved experiments that will allow IMC detection, as a function of solder joint preparation, i.e. heating and cooling, to be performed non-destructively in Pb-free solder joint assemblies. Furthermore, grain size information and any preferred growth mechanisms would also be assessed.

Publications

[1] G.J.Jackson, H.Lu, N.Hoo, M.W.Hendriksen, N.N.Ekere, C.Bailey,' Intermetallic phase detection in Pb-free solders using fine-focus synchrotron X-ray diffraction', in preparation.