LEHIGH UNIVERSITY

Center for Optical Technologies

Nanoindentation Investigation of the Au-Sn System for Optoelectronics Applications

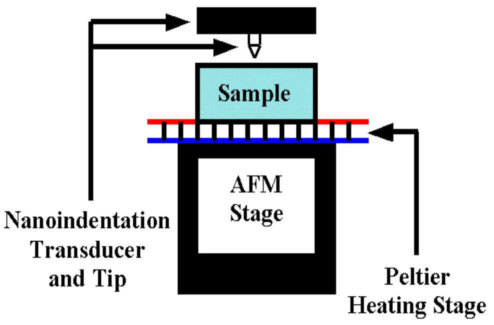
Richard R. Chromik, Richard P. Vinci, Dong-Ning Wang, Aaron Shugar and Michael R. Notis
Department of Materials Science and Engineering

Introduction and Motivation

Nanoindentation is a powerful technique where sensitive measurements of both load and displacement are made as a diamond indenter makes contact with a specimen. The length scale of contact is sub-micron and loads as small as 10 μN may be used. This allows for measurements of mechanical properties of small included phases within a matrix, or a thin film of nanometer length scale on a substrate.

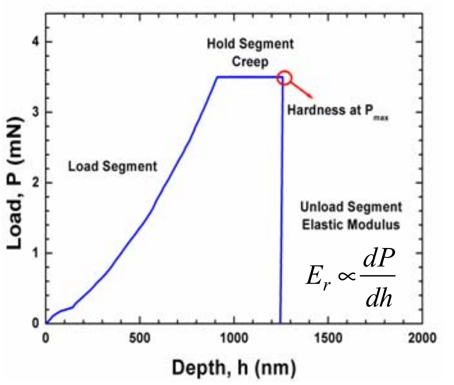
We have used nanoindentation to measure the mechanical properties of various phases that exist in solder joints. Despite only scant property data in the literature and only for bulk samples, failure of a microelectronics package is often attributed to "*brittle intermetallics*". Nanoindentation has allowed us to characterize the deformation behavior of Au-Sn solder, and various intermetallics (Au-Sn, Cu-Sn and Ag-Sn) at length scales similar to that in a real solder joint. A mechanical property database has been compiled, where our measurements may be used for modeling solder joints at a microstructural level.

Nanoindentation System



- Loading: 10 μN to 10 mN with 100 nN sensitivity
- Tip displacement: 5 μm full scale with 0.2 nm sensitivity
- Diamond indenter: Berkovich pyramidal geometry
- Heating stage: 20°C to 150°C
- Imaging: Coupled to atomic force microscope (AFM)

Nanoindentation Experiments



At the maximum load (P_{max}), a hardness, H, is also measured:

$$H = \frac{P_{\max}}{A(h_c)}$$

where $A(h_c)$ is the projected contact area measured by calibration testing on a fused quartz standard. During the unload segment, a reduced modulus, E_r, is measured that is based on elastic recovery of both the sample and the indenter:

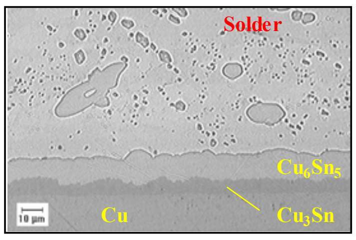
$$E_r = \left(\left.\frac{1-\nu^2}{E}\right|_{sample} + \left.\frac{1-\nu^2}{E}\right|_{indenter} \right)^{-1}$$

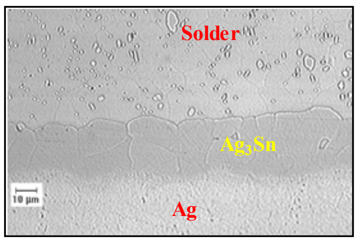
where, with knowledge of Poisson's ratio, ν, the Young's modulus, E, may be calculated.

Mechanical Properties of (Cu, Ag)-Sn Intermetallics

Sample Preparation and Experiment

Nanoindentation testing was carried out on Sn-Ag-Cu solder, Sn, Cu and three intermetallics (Cu_6Sn_5, Cu_3Sn and Ag_3Sn). Samples were prepared by annealing diffusion couples that were prepared between a metal (Cu or Ag) and solder material (Sn-Ag-Cu or pure Sn). This resulted in the formation of continuous layers of intermetallic at the solder/metal interface. To the right are light optical micrographs of the resulting intermetallic layers in annealed diffusion couples. By using the imaging capabilities (AFM) of the nanoindentation system, one can locate these layers, indent and then examine the position of the indent. This procedure ensured testing of the correct phase and also helped avoid edge effects. At least 40 or more indents were performed on each of the three intermetallics. At least 10 or more indents were performed on Cu, Sn and Sn-Ag-Cu solder, where some experiments for Sn and the solder were performed on bulk specimens.





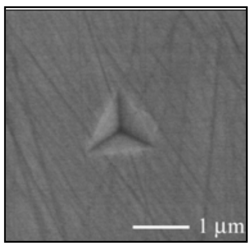
Mechanical Properties and Reliability

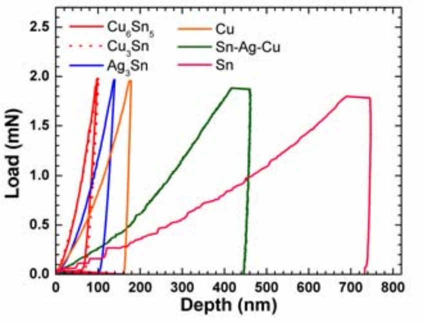
To the right is a plot of load versus depth for all of the materials studied for indentation tests with a 2.0 mN maximum load. For the given load, the maximum depth obtained in the Sn-Ag-Cu solder is approximately 4.5 times that obtained in the Cu-Sn intermetallics. The solder is quite soft and exhibited significant plasticity, as seen in the AFM image above.

From the load-depth curves, measurements of Young's modulus and hardness were made for all of the materials (table below right). The high hardness of the Cu-Sn intermetallics indicates a potential for brittle behavior (crack nucleation). With a lower hardness of 2.9 GPa, Ag_3Sn is observed to be much more ductile and is, thus, not likely to exhibit brittle behavior. Also, for the loads (> 9.5 mN) and length scales of nanoindentation testing, no cracking was observed (see SEM image below). Thus, using the Tabor relation, a yield stress may be calculated from these hardness values.

The Young's modulus values measured for Cu, Sn and Sn-Ag-Cu are consistent with literature values for these materials. The Young's modulus measured for Cu_6Sn_5 is similar to that for pure Cu, while the modulus for Cu_3Sn is slightly higher. The Young's modulus measured for Ag_3Sn is similar to that of pure Ag.

The results presented here represent a consistent database of mechanical properties for these materials measured by a single technique and at length scales similar to those in a real microelectronics joint.





Material	Young's Modulus (GPa)	Hardness (GPa)
Cu_6Sn_5	119±6	6.5±0.3
Cu_3Sn	143±7	6.2±0.4
Ag_3Sn	88±5	2.9±0.2
Cu	123±7	1.7±0.2
Sn-Ag-Cu solder	45±8	0.16±0.06
Sn	44±5	0.11±0.05

Mechanical Properties of Au-Sn Solder and Au-Sn Intermetallics

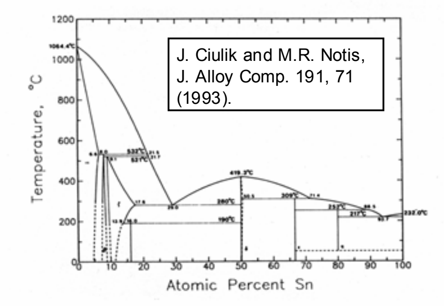
Sample Preparation and Experiment

The Au-Sn system contains a eutectic point at 29at% Sn with a melting point of 280 C. This composition is a common "first-attach" solder for hierarchical soldering processes requiring multiple reflows at different temperatures. It is also an ideal solder for optoelectronics application, due to it's higher creep resistance.

An investigation of the mechanical properties of intermetallics in the Au-Sn system was carried out. Two samples were prepared:

<u>Sample #1</u> *Sn rich side*
- Au plate in molten Sn for 80 seconds
- Diffusion anneal @ 185 C for 48 hrs

<u>Sample #2</u> *Au rich side*
- Au plate in molten Au-Sn eutectic for 80 seconds
- Diffusion anneal @ 180 C for 1 week



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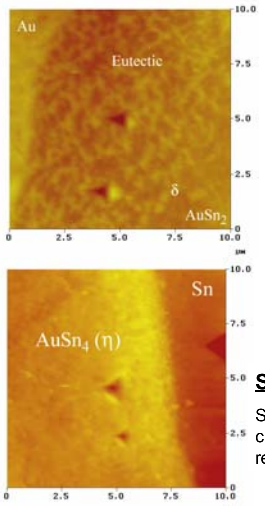
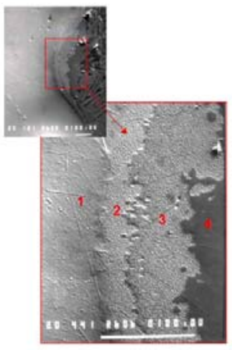


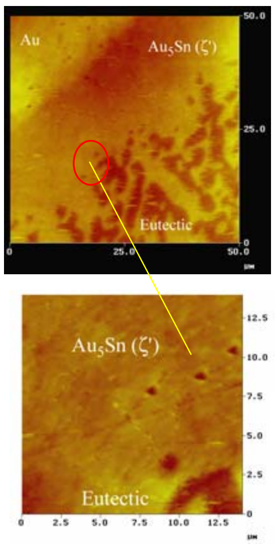


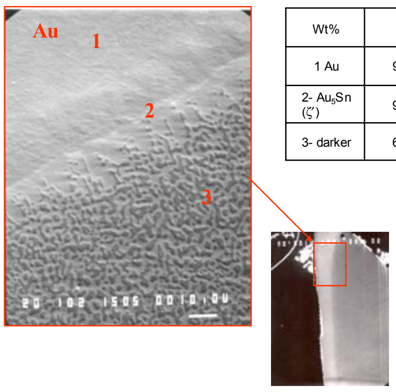
Wt%	Au	Sn	Total
1 Au	97.60	0.10	97.70
2- δ AuSn	62.10	37.00	99.10
3- ε $AuSn_2$	45.30	54.80	100.00
4- η $AuSu_4$	30.20	75.10	105.20

Point measurement 2

Sample #1, Phases present to test: **$AuSn_4$ (η), $AuSn_2$ (ε), "eutectic", Au**

Shown above are electron microprobe images and a table of the measured chemical compositions of the layers in this sample. To the left, atomic force microscopy images of residual indents from nanoindentation tests performed on this sample are presented.



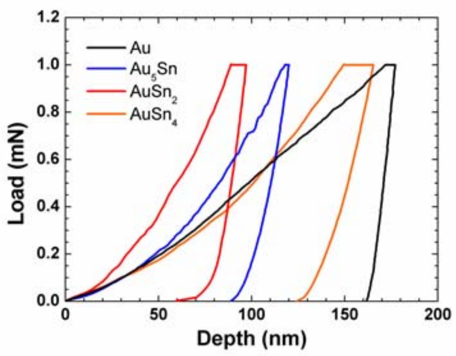


Wt%	Au	Sn	Total
1 Au	96.5	0.2	96.7
2- Au_5Sn (ζ′)	90.7	10.3	101.0
3- darker	63.3	38.0	101.3

Sample #2, Phases present to test: **Au, Au_5Sn (ζ′), "eutectic"**

Electron microprobe analysis revealed the Au_5Sn phase in sample #2. Images and measured chemical compositions are shown above. To the left, AFM images of residual indents in the Au_5Sn layer are shown.

Nanoindentation Results, Mechanical Properties and Reliability



Material	Indentation Modulus (GPa) $\frac{E}{1-\nu^2}$	Hardness (GPa)
Au_5Sn (ζ′)	91 ± 5	2.5 ± 0.2
AuSn (δ)	*Similar to Au_5Sn*	
$AuSn_2$ (ε)	116 ± 10	2.9 ± 0.4
$AuSn_4$ (η)	43 ± 4	1.2 ± 0.2
$(Au,Ni)Sn_4$	48 ±3	1.8 ± 0.1
Au	84 ± 5	0.95 ± 0.05
Sn	51 ± 5	0.11 ± 0.05

The Au-Sn intermetallics have mechanical properties that appear non-deleterious to solder joint reliability. The hardnesses of the Au-Sn intermetallics are comparable to Ag_3Sn, which is very ductile. This is contrary to repeated reports in the literature of "Au embrittlement" of solder joints. This leads to the hypothesis that the "Au embrittlement" phenomena is due to weak interfaces between Au-Sn compounds and surrounding phases. A supporting argument for this is the $(Au,Ni)Sn_4$ phase, which commonly forms in Pb-Sn/Au/Ni solder joints. This phase, also with reasonable mechanical properties, poses reliability problems when formed next to Ni_3Sn_4. Alone, the Ni_3Sn_4/solder interface is strong, but when the $(Au,Ni)Sn_4$ forms, this interface becomes weak. For future solder joint design for reliability, a clearer picture of failure mechanisms will be necessary, where intermetallic properties and interface strength are both considered.