Nanoscale Lead-free Solders (“Nano-Solders”): Synthesis, Characterization, and Reflow Properties

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Nanoscale Lead-free Solders ("Nano-Solders"): Synthesis, Characterization, and Reflow Properties

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The Toxics Use Reduction Institute
Academic Research Program

2008

The Massachusetts Toxics Use Reduction Institute
University of Massachusetts Lowell

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Abstract

Nanoscale lead-free solders ("nano-solders") have been proposed and investigated in the development of nano-soldering technique for nanoscale assembly and integration. Tin (Sn)-based and indium (In)-based lead-free nano-solders have been synthesized directly onto multi-segmented nanowires using electrodeposition method in nanoporous templates. The diameter of nano-solder nanowires ranges from 50nm to 200 nm and length from 1-10 μm. The microstructure of nano-solder nanowires have been studied using optical microscope and electron microscopes (FESEM and TEM) along with energy-dispersive x-ray spectroscopy (EDS). Thermal reflow properties of nano-solders were characterized with a temperature programmable furnace tube. It was found that flux plays an important role in the nano-solder reflow process.

KEYWORDS: Nanotechnology, Lead-free Nano-solders, Self-assembly, Nanowires, Nanoelectronics

1. Introduction and Background

Nanotechnology has been one of the most exciting and rapidly growing areas in the past decade. Nanoscience and nanotechnology involve the design, fabrication, and engineering of materials and systems at the nanometer scale (1-100 nm). Materials and systems at this scale may exhibit novel optical, electrical, electronic, magnetic, and mechanical properties.[1-8] There have been significant developments in new nanomaterials fabrication and novel nanotechnology development, e.g., the emerging and evolving of carbon nanotubes, nanoparticles, and nanowires. On the other hand, many assembling methods have been developed to integrate nano-components into ordered one-dimensional (1-D), 2-D and 3-D structures. However, joining and interconnect formation between these self-assembled structures are still less developed and understood, which is critical for electronics and device integration. *Significant improvement is needed in order to realize nanoscale manufacturing (nanomanufacturing). In addition, related health and safety rules/policies of nanomanufacturing have to be developed or created for environmentally-benign and green manufacturing.*

There has been some progress in joining nano-components. Two main techniques are: (1) annealing[9,10] and (2) focused E-beam (FEB) or focused Ion-beam (FIB) irradiation. Annealing at high temperature is an efficient way to lower contact resistance between components, especially metals to metals, but the annealing temperature is normally very high (up to 400-500 °C), which will damage many components including organics and polymers. FEB/FIB soldering has been successfully used to bond carbon nanotubes to substrates, nanotubes to nanotubes, and gold nanowires;[11-18] however, this technique suffers from contamination of uncompleted precursor decomposition. For both methods above, slow processes, harsh processing conditions, and high cost make them almost impossible for manufacturing-scale production other than in laboratory-scale integration.

Solders and soldering are widely used in electronics industry for product assembly and packaging.[19-24] Solders are low-melting point metals or metal alloys; they are excellent joining materials to form electrical interconnects in electronics and also have good mechanical property. The mostly widely used solders are eutectic Sn/Pb (60/40). One major application of soldering is assembling electronic components to printed circuit boards (PCBs). Another common application of soldering is to make permanent but reversible connections between copper pipes in plumbing systems. Due to environmental concerns, health and safety issues (Pb is very toxic), "no-lead" solders are becoming more and more widely used. For example, Europe has required many new electronic circuit boards to be Pb-free by July 1st, 2006. Unfortunately, many Pb-free solders are not eutectic formulations, making it more difficult to create reliable joints. Thus, **there is tremendous need to develop new Pb-free solders with higher or similar quality as the traditional Sn/Pb (60/40) solders for electronics and other industries**. With the emerging and evolving of nanotechnology, the importance of developing nanoscale Pb-free solders is clear and the demand is huge for the future nanoelectronics industry. **Nanosolders may not only have better properties, but also have lower melting points, which will greatly lower the operating temperature and save energy, and make the solder reflow process more economical**.

In this TURI funded project, we have proposed and fabricated two types of Pb-free nanosolders; tin (Sn)-based and indium (In)-based solders, which have great potential to serve as new base materials and novel platforms for nano-component assembly and integration. The fabrication process involves electrodeposition in nanoporous templates. The nanosolders fabricated have been characterized with optical microscope and electron microscope (SEM and TEM) along with Energy Dispersive X-Ray Spectroscope (EDS). Preliminary solder reflow properties have been studied in a temperature programmable furnace tube. Particle exposure test was monitored during nanowire fabrication with a digital particle analyzer (through collaboration with Toxics Use Reduction Institute, TURI).

2. Experimental Section

2.1 Materials Used

Commercial plating solutions of silver (Ag) (Techni Silver E-2), gold (Au) (Techni Gold 25E), nickel (Ni) (Techni Nickel Sulfamate Bath RTU), copper (Cu) (Techni Copper U Bath RTU), and Sn (Techni Tin) were purchased from Technic, Inc (www.technic.com). Indium Sulfamate was purchased from Indium Corp. (www.indium.com). Sodium hydroxide (NaOH), dichloromethane, and ethanol were purchased from Fisher Scientific.

Alumina and polycarbonate membranes are purchased from Whatman (now GE Healthcare). Silver (Ag, 99.99% purity) evaporation target was purchased from Alfa Aesar. De-ionized water was made from Nano-pure (18MΩ).

2.2 Nanowire Synthesis and Fabrication

Nanoscale Pb-free solders ("nano-solders") have been synthesized directly onto nanowires. Nanowires are one type of the most promising nano-building blocks in the field of nanoscience and nanotechnology, including nanoelectronics.[25-29] Metallic, semiconducting, and polymeric nanowires can be synthesized using various techniques. Direct integration of nanoscale solders onto nanowires has several advantages: precise placement of solders at any location of nanowires, less solder volume being used, and excellent size control, etc.

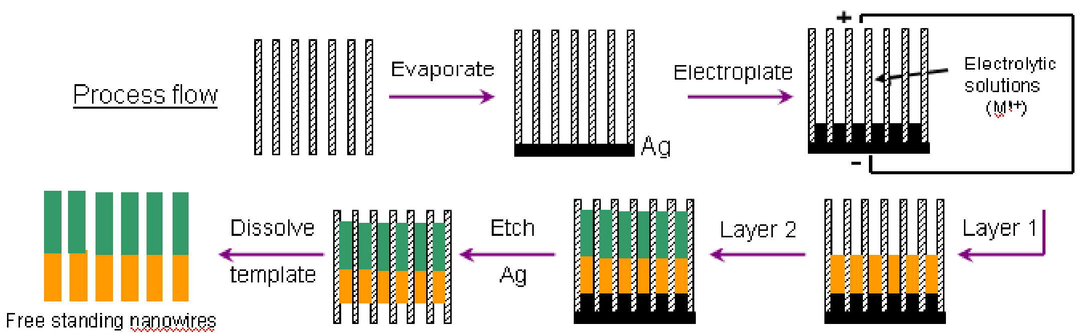

Figure 1. Process flow for the fabrication of single or multi-segment nanowires with functional solder segments and the release of nanowires from nanoporous templates

Electrodeposition in nanoporous templates has been utilized to fabricate nanowires and nano-solders. A brief process is as follows (**Fig. 1**): [30-33] First a thin layer of Ag was evaporated (Nanomaster NTE3000) on one face of a commercial alumina or polycarbonate membrane (**Fig. 2**). The membrane was placed in contact with a copper plate and restrained with a glass joint and viton o-ring seal. The membrane was soaked in water for 5 minutes and then filled with the electrolytic solution of choice. Plating solutions for silver (Ag), gold (Au), copper (Cu), nickel (Ni) were utilized. Typical plating conditions were 0.8 to 1.2 mA/cm^2, for Au, Ag, and Ni. The Ag on the other side of the membrane was dissolved in 6M HNO_3 after electrodeposition. After rinsing several times in water, the membrane was dissolved in 2 M NaOH (for AAO membrane) or dichloromethane (for polycarbonate membrane) to release the nanowires. The nanowires were rinsed repeatedly with water and ethanol (using sonication and centrifugation cycles) and then stored in ethanol for further assembly or utilization.

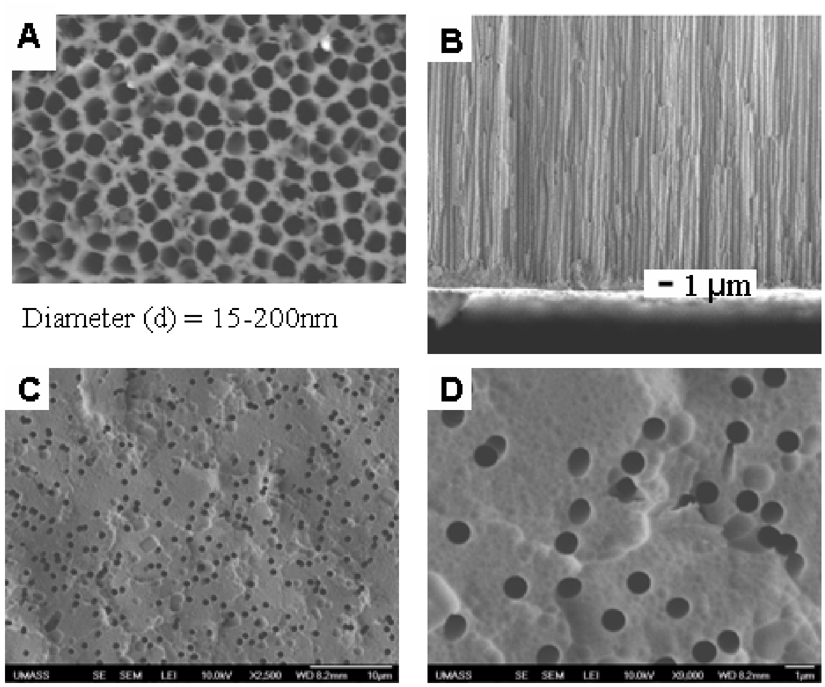

Figure 2. (**A-B**) Top view and cross-section view of an alumina membrane; (**C-D**) top view of a polycarbonate membrane.

2.3 Materials Characterization

Optical images of nanowires fabricated were taken using an Olympus CX-41 microscope equipped with DP-71 CCD camera. JEOL JSM-1401F Field-emission scanning electron microscope (FE-SEM) and Philips EM400 transmission electron microscope (TEM) were used to characterize the size and structures of nano-solders and nanowires.

2.4 Thermal Properties – Solder Reflow

Solder reflow is a process being used in industry to solder the joints. There are normally four stages ("zones"): preheat, thermal soak, reflow and cooling. Solder manufacturers normally supply the reflow profiles to industrial customers. In this project, we have initiated the study of reflow properties of both Sn-based and In-based nano-solders with different melting temperatures, which will provide more insight into new applications especially for hybrid systems that need a lower solder reflow temperature. Process windows are defined as the temperature range between the maximum reflow temperature and level where damage to electronic components will occur. It is clear from the figure that the current lead-free solders have a smaller process window for electronics and are not suitable for “soft” materials processing. The process window for lead-free nano-solders are expected to be much larger than lead-free solders currently being used and thus will increase the reliability and lifetime of systems and devices. It is also possible that the melting points of nano-solders can be significantly lowered with smaller size (or shape) of nano-solders and thus can be used to process “soft” materials. Our intent is to develop these reflow profiles for lead-free nano-soldering applications for this research.

Nano-solder reflow was carried out in a high-temperature tube-furnace (Thermo Scientific Lindberg Blue tube furnace). The nano-solders were dispersed on a silicon substrate and introduced into the furnace. The dwell time above the peak reflow temperature was minimized to reduce inter-diffusion of metals. In this project, the reflow process for nano-solders followed similar reflow profile as the conventional eutectic solder reflow process. Table 1 shows the melting points of bulk lead-free solders.

Table 1. Melting points of several solders

Solders	**Sn**	**In**	Sn/In	Sn/Ag/Cu	“Sn/Pb”
Melting points (ºC)	**232**	**157**	118	217	183

3. Results and Discussion

3.1 Single- and Multi-segmented Nanowires

Figure 3 shows different images of the nanowires fabricated using templated electrodeposition technique.

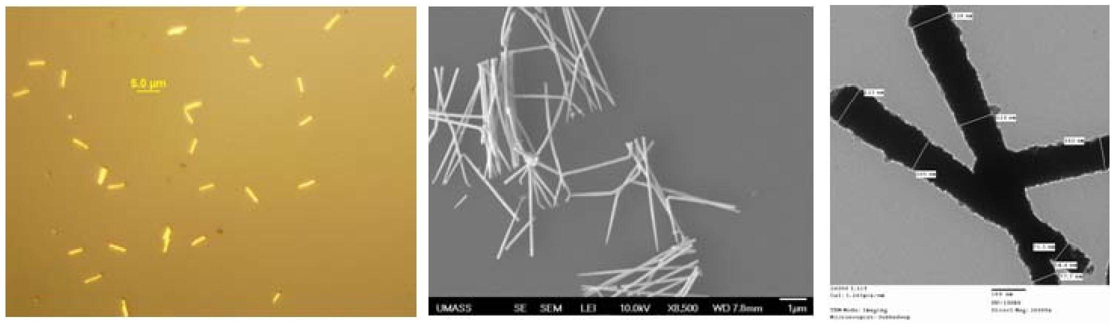

Figure 3. Various images of gold nanowires nanowires: (A) optical image (d=200 nm), (B) SEM image of gold nanowires (d=50 nm), and (C) TEM image of gold nanowires (d=50 nm)

3.2 Lead-free Nano-solders on Nanowires

In this project, we have successfully fabricated many kinds of nanowires including both Sn-based and In-based nanosolders. The diameter is from 200 to 50nm with different lengths. For Sn-based nanowire, they are: pure Sn, Au-Sn, Au-Ni-Au-Sn, Sn-Au-Sn; Sn/3Ag alloy-solders were also fabricated onto nanowires. (see Figure 4 images) In the future, we plan to fabricate more alloys such as SAC (Sn/Ag/Cu).

- **Tin (Sn)-based lead-free solders**

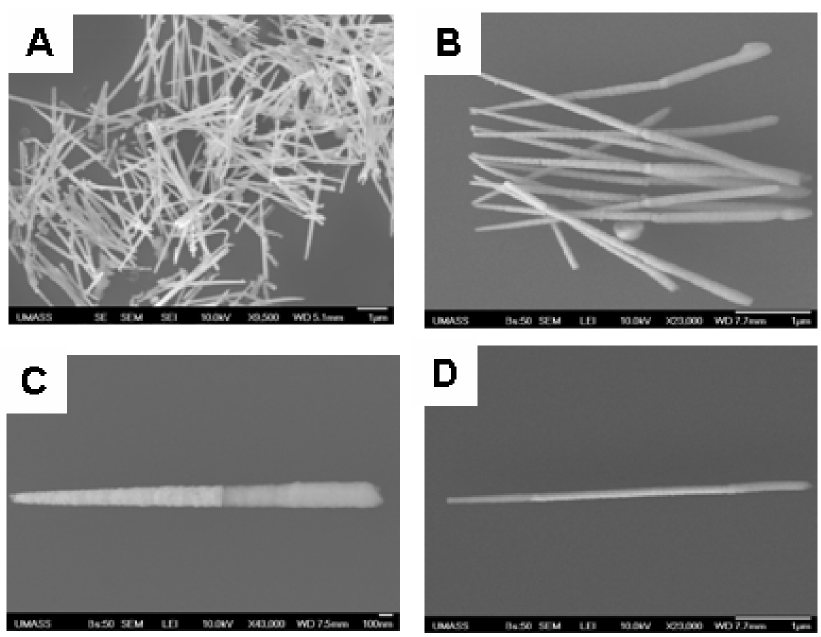

Figure 4. Lead-free nano-solders on nanowires. (A) Pure Sn nanowires, (B) Au-Sn nanowires, (C) Au-Ni nanowires, and (D) Sn-Au-Sn nanowires

- **Indium (In)-based lead-free solders**

Low melting point indium (In) has been fabricated onto nanowires using similar way. Their structures were studied. One interesting phenomenon is that voiding was observed (Figure 5) on nanowires during electrodeposition. Figure 5B shows the EDS data of the elements in each segment of nanowires. It is clear from the XPS data that indium solder was successfully deposited onto nanowires.

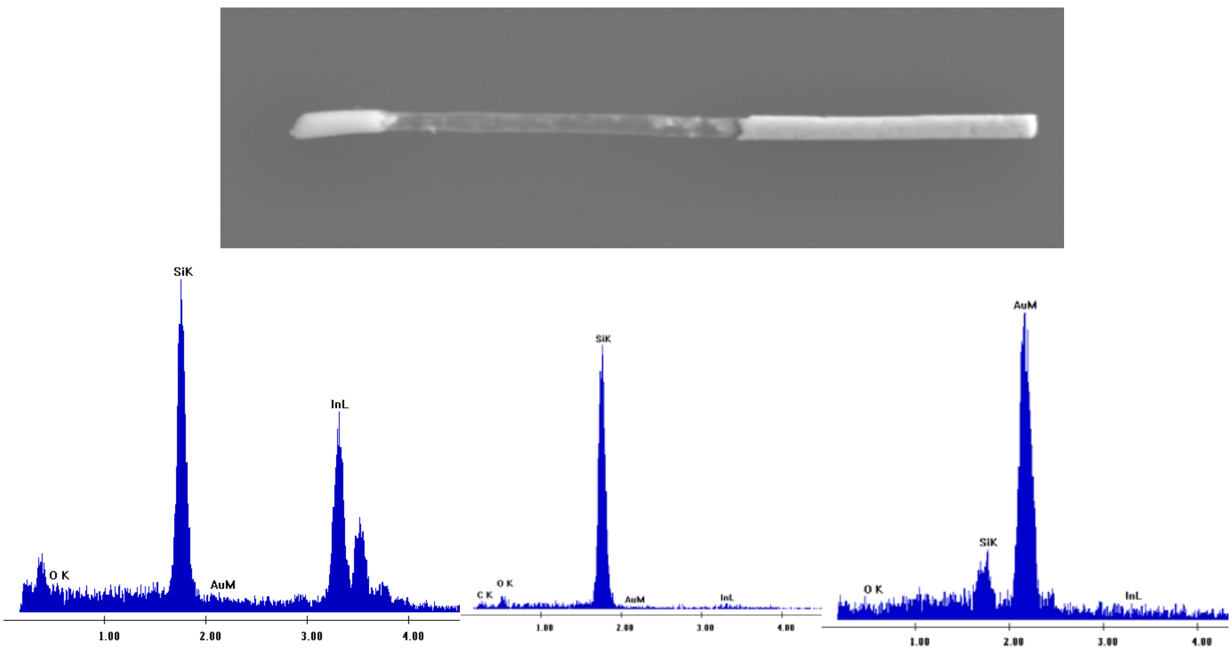

Figure 5 Au-In nanowires. A void (dark segment in the SEM image) was observed in such system.

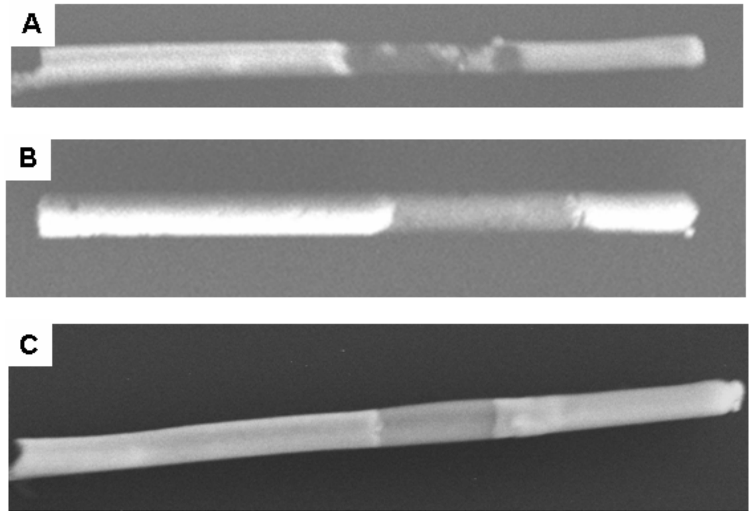

Figure 6. In-based lead-free nano-solders on nanowires. (A) Au-In nanowires, (B) Au-Ni-In nanowires, (C) Au-Ni-Au-In nanowires

3.3 Thermal Properties – Solder Reflow Results

The major impact of the lead-free conversion will be on the solder reflow process step. Most lead-free alloys under investigation melt at temperatures 30° to 40°C higher than eutectic Sn/Pb solder. Based on the elemental constituents of the lead-free candidates, lead-free solder should be less lustrous and slightly "grainier" than the baseline Sn/Pb alloy. In practice, this is exactly what was found.

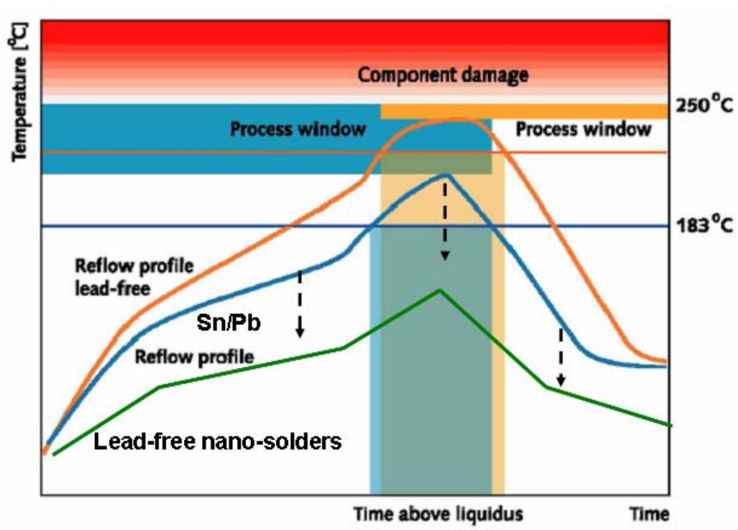

Figure 7. Solder reflow profile

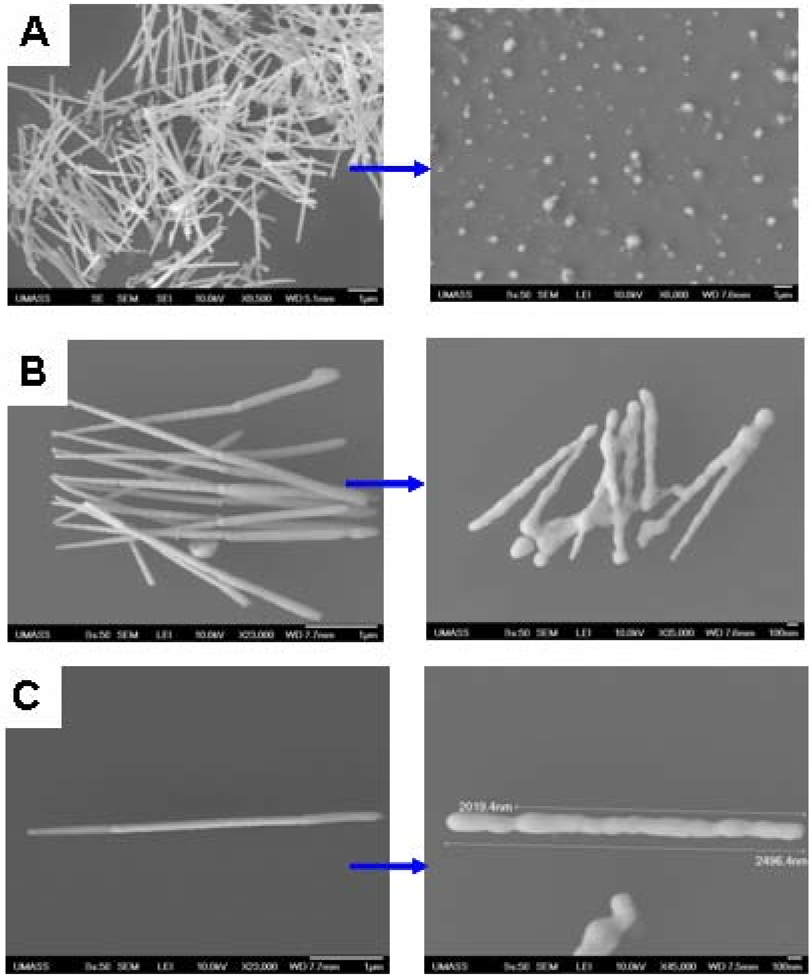

Figure 8. Solder reflow profile

Rule of flux

Solder manufacturers have found that there is no lead-free "drop-in" replacement as far as solder paste fluxes are concerned. Currently, vendors are developing fluxes that will perform well with the new alloy chemistries and process conditions. Each element, and resultant alloy, has unique characteristics with regard to oxidation, surface tension, reflow and wetting. Solder paste fluxes must be formulated to address the specific alloy(s) of choice. Some solder paste manufacturers have made great advances and presently offer viable lead-free solder paste products.

Achieving a low residue is becoming a major control issue with the move toward clean fluxes in the aim of discontinuing the use of carbons. Nevertheless, during the experiment, one drop amount of flux in the reflow system will easily separate nanosolders from interface of and the residue of flux is difficult to move away from the surface of nanosolders. Compare with industrial solder paste which contains solder weight nearly more than 80% (that means the flux only has less than 20%), the flux used in the nanosolders looks much more than the common value. So how to control the quantity of flux consuming to fit nanosolders and clean it after reflow process are the critical problems to solve.

We also observed interesting results in the reflow of indium nano-solders. Without the presence of flux, it showed that solder reflow was difficult to achieve. Figure 9 shows that under high temperature (i.e., >500 C) annealing, indium diffused into nickel segment. Further investigation of solder reflow and intermetallics should be conducted.

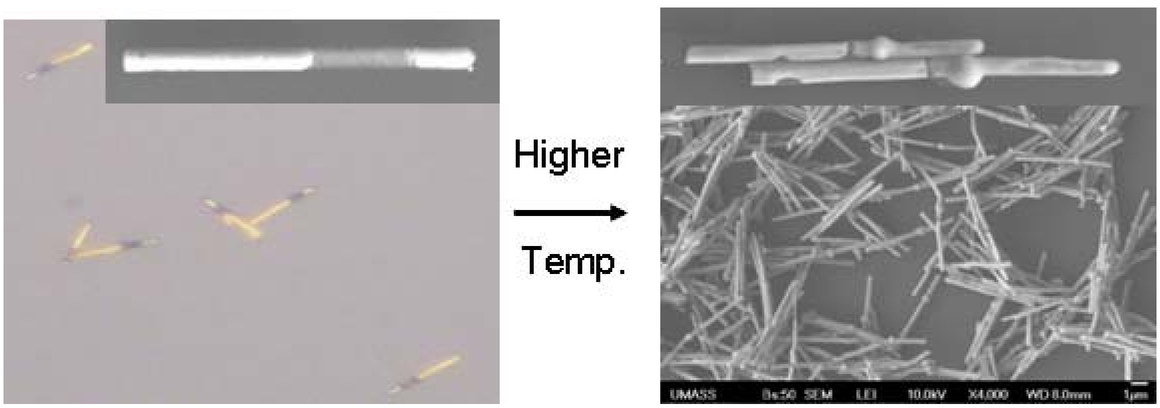

Figure 9. Solder reflow profile

3.4 Nanowire Size Analysis through TURI Collaboration

A TSI Fast Mobility Particles Sizer (FMPS) was used to measure number concentration of nanowires by particle size. The FMPS measures submicron size range (5.6 to 560 nm) aerosols. It reports total number concentrations and size distributions for a total of 32 channels every 1 second. Figure 10 shows the background levels and the concentrations at the points close to the sources. Elevated concentration values were noticed only when the nanoporous templates were being cut into pieces using scissor. For that step, 30 consecutive 1 second periods were chosen, and average number concentrations were calculated by particle size. The total concentration for the cutting step was about 19,000/cc, which was 7.3 times that of background level at 2,600/cc.

The particle size distribution for the cutting step was compared to that of the background level as shown in Figure 10. As you can see, small particles, mostly in the range of 20-90nm, were generated from the cutting.

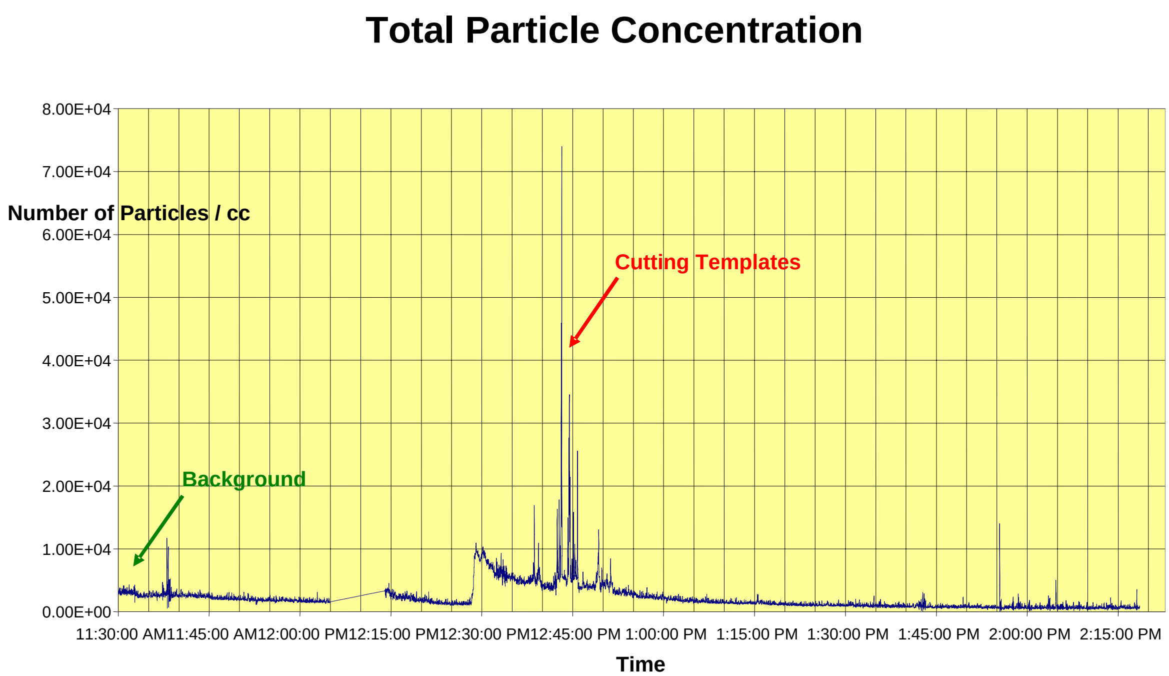

Figure 10. particle size analysis

4. Summary

Tin (Sn)-based and indium (In)-based lead-free nano-solders have been synthesized onto multi-segmented nanowired using electrodeposition method in nanoporous templates. The diameter of nano-solder nanowires ranges from 50nm to 200 nm and length from 1-10 µm. The microstructure of nano-solder nanowires are studied using optical microscope and electron microscopes (FESEM and TEM) along with energy-dispersive x-ray spectroscopy (EDS). Thermal reflow property of nano-solders was characterized with a temperature programmable furnace tube.

5. Presentations and Publications through this Project

Significant results have been obtained in the FY08 TURI Sustainability Research Project. A journal paper is in preparation to publish the original findings from this project, and the two graduate students who worked on the project will be lead authors. In additions, several oral and poster presentations have been made in various educational and technical conferences on the topic of lead-free nano-solders.

Journal Publications

(1) F. Gao, S. Mukherjee, Z. Gu. "Synthesis, Characterization, and Reflow Properties of Nanoscale Lead-Free Solders on Multi-Segmented Nanowires", **2008**, in preparation.

Conference presentations (oral presentation or poster presentation as indicated)

(2) E. Chin (**Poster**), F. Gao, S. S. Mukherjee, and Z. Gu*. "Synthesis and Characterization of Multifunctional Nanowires for Sensor and Electronics Applications", *35th NEACerS/IMAPS Symposium & Exhibition*, Boxborough, MA, May 6, 2008.

(3) E. Chin, X. Li (**Oral**), F. Gao, S. S. Mukherjee, and Z. Gu*. "Synthesis and Characterization of Multifunctional Nanowires for Sensor and Electronics Applications", *2008 AVS New England Chapter Annual Symposium*, Burlington, MA, June 2, 2008.

(4) S. S. Mukherjee (**Oral**), F. Gao, and Z. Gu. "Nanoscale Lead-free Solders ("Nano-Solders"): Synthesis, Characterization, and Reflow Properties", *TURI Continuing Education Conference*, Westborough, MA, April 8, 2008

(5) E. Chin, Xiaopeng Li, F. Gao, S. S. Mukherjee, and Z. Gu* (**Poster**). "Synthesis, Characterization, and Assembly of Multifunctional Nanowires for Sensor and Electronics Applications", *236th American Chemical Society (ACS) National Meeting*, Philadelphia, PA, August 17-21, 2008 (accepted).

(6) F. Gao (**Oral**), S. Mukherjee, and Z. Gu*. "Exploration of Lead-Free Nanosolders for Nanowire Assembly", *2008 AIChE Annual Meeting*, Philadelphia, PA, Nov. 16-21, 2008 (accepted).

(7) F. Gao (**Oral**), S. Mukherjee, and Z. Gu*. "Electroplated Lead-Free Nanosolders onto Multi-Segmented Nanowires and their Reflow Properties", *Materials Research Society (MRS) Fall Meeting*, Boston, MA, Dec. 1-5, 2008 (abstract submitted)

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