Metallic Nanoneedles Arrays for “Lift-Out” TEM Sample Preparation

Romaneh Jalilian, Brian Miller, David Mudd, Neil Torrez, Jose Rivera, and Mehdi M. Yazdanpanah
NaugaNeedles LLC, Louisville, KY
Purpose

• Introduction to NaugaNeedles technology
• Review Standard probe for TEM Lift-out
• Using Needle Array to sharpen W probe (Needle-Tipped Probe )
• Needle-Tipped Probe for TEM Lift-out
• Summary
An Introduction to NaugaNeedles Technology

Time lapse images of an Ag$_2$Ga needle growing from a melted drop of gallium

Ag$_2$Ga Nanoneedles

Properties

1. Mechanical
   • Spring constant (K) = 0.1 - 1 (N/m),
   • Resonance frequency ($f_0$) = 20 - 10 (MHz)
   • Young’s modulus (E) = 84 ± 1 (Gpa)

2. Electrical
   • Electrical Resistivity ($\rho$) = 2 × 10$^{-7}$ (Ωm)
   • Max Current Density (J) : 1.5 × 10$^{-11}$ (A/m$^2$)

3. Thermal
   • Melting point (experimental) = 950 °C

4. Resistive to Reactive Ion Etching (RIE)

Applications

• AFM, SGM, SECM, EFM, STM, nano-cantilever, ...
Example of Nanoneedle Application for AFM (EFM)

Enhancement of electric force microscopy (EFM) imaging on CNT nano-composite

NaugaNeedles Array Technology

- Grow arrays of Ag$_2$Ga nanoneedles on prefabricated structures
- Nanoneedles Diameter (W, Ag, Cr, Pt coated): 500nm-1000nm
- Nanoneedles Length: 15µm to 50µm
- Array contains minimum of 1000 nanoneedles

[R. Jalilian et al, Nanotechnology 22 (2011)]
Problems With Lift-Out Using Standard Tungsten Probes

• Sharpening process usually takes several minutes
• A tungsten probe may be replaced after a few uses
• The extended sharpened tungsten tip is on axis with the tungsten probe, which limits application
Fabrication of Needle-Tipped Probe

1. Locate a selected nanoneedle on array
2. Rotate the needle array at the desired angle
3. Weld a tungsten probe to the needle
4. Cut the needle free from the array substrate
Lift-Out Process Using Needle-Tipped Probe
Improved Sample Viewing Using Needle-Tipped Probes

Improved sample viewing in SEM when the needle is in contact with the specimen

Clear view of connection in FIB when the needle is in contact with the specimen
Needles-Tipped Probes are Flexible and Durable

Needle bend without plastic deformation

Keep similar properties after cutting and re-welding

Before bending

First bending test

After cutting, back to original shape

Bending test after second welding

1 µm

1.8 µm

1.7 µm
Vibration Test of the Welded Needle During Sample Movements

The Welded Needles can survive severe mechanical vibration and shocks
A Nano-Fork Made from Needles for Weldless Lift-Out

The movie demonstrates the use of fork made by needles for weldless lift-out
Advantages of Using NaugaNeedles Array for Lift-Out

• Better mechanical properties than W probe
• More FIB uptime due to less W tip changes
• User maintained tip by sharpening inside TEM
• Curve the shape for best sample viewing
• Needles have constant small diameter
• Needles flexibility during touch down
• No sample welding using nano-Fork
Summary

• Introduced Ag$_2$Ga Nanoneedles technology
• Reviewed Nanoneedles properties and application
• Demonstrated W probes sharpening using needle array
• Demonstrated Lift-Out using Needle-Tipped Probe
• Examined the Needle-Tipped Probe properties
• Reviewed Needle-Tipped probes advantages for Lift-Out
Thank You
Procedure

- **Needle-Tipped Probe fabrication:**
  1. Locate a nanoneedle on array in the TEM system
  2. FIB-weld the W probe to the nanoneedle
  3. FIB-cut the needles from the array

- **Needle-Tipped Probe for Lift-Out:**
  1. Bring the Needle-Tipped Probe in contact
  2. Weld the nanoneedle to the sample
  3. FIB-cut the specimen
  4. Transfer the specimen to TEM grid
  5. FIB-cut the Needle-Tipped Probe
More examples of Needle-Tipped probe in action
Robust Nanoneedles, Less Buckling

The needle pushed against the specimen
→
The specimen displaced severely
→
But Nanoneedle buckles slightly