Part – 2: Whisker characterization

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Outline

• Review part 1
• TEM characterization
  – EDS
  – SADP
• Conclusions
Hypothesis: massive nucleation and co-growth of multiple thin filaments = nanostructure of whiskers

Possible hollow

~10 nm

striations

~1 micron

Possible neutral spot

Charge patch

One anticipated consequence: the multi-filament structures can occasionally split: Observed and related by Steve Smith and Bill Rollins

If the charge patch is related to an individual grain, then the well-known log-normal distribution of grain sizes translates into that of MW diameters
Whisker FIB-TEM analysis

FIB-milling:

Two goals:
1. Whisker cross-section analysis.
2. TEM sample preparation.

Milling (ion-beam) and imaging (electron beam) done simultaneously

Ion-beam can be used to take images (gives more details)

Whisker FIB-TEM analysis

Cross-section analysis (Evaporated sample)

The smaller SEM image to the left is the original whisker before the FIB work. Red arrows in all the 3 figures point to voids, which should not be confused with IMCs. The bottom one is an ion beam picture (blue box) of the same whisker and it clearly shows the contrast difference between different materials, uniform Cu layer, inherent voids and the layers with no signs of IMCs. The scale bar in all the three images is 1µm.
Whisker FIB-TEM analysis

Cross-section analysis (Evaporated sample)

SEM image of original whisker before Pt deposition is shown to the left (red box). Image to the right is the SEM scan of the same whisker after depositing Pt and milling using ion-beam (yellow box).

Above are the close-up scans of the cross-section clearly showing the layer separation, no signs of IMCs.
Whisker FIB-TEM analysis

Cross-section analysis (Electroplated sample)

- **Z-contrast and ion images** showing more details of the layers.
- **Cross-section reveals no signs of IMCs.**
Whisker FIB-TEM analysis

FIB-milling: general TEM sample preparation steps

- Sample lift-out was carried using a in-situ nano-manipulator.
- After final cleaning, sample is transferred to TEM.
Almost all whisker nucleation diameters appear to be in the micrometer range, which is substantially greater than crystalline nuclei sizes in solids.

We used FIB-TEM to verify this.

Longitudinal ridges or striations on the whiskers are quite common regardless of their diameter.

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Whisker FIB-TEM analysis

FIB-milling: Whisker TEM sample

Whisker attached to TEM grid

During Pt deposition

Pt deposited

Mounting on TEM grid

Lift-out

Trenching

Top-view (trench)
Whisker FIB-TEM analysis

FIB-milling: Whisker TEM sample

Top view

Before thinning

After thinning

Side view
Whisker FIB-TEM analysis

TEM analysis

Low magnification TE image

A schematic representation of the whisker’s internal structure

TE image

ZC image
Whisker FIB-TEM analysis

The internal structure observed indicates alignment along the length of the whisker.

Filament of size 20-50nm appear to comprise the volume of the whisker.
Whisker FIB-TEM analysis

TEM analysis

• Longitudinal internal structure is consistent along whisker’s length.

Low magnification TE image

TE image

ZC image
Whisker FIB-TEM analysis

EDS line analysis

Chemical composition analysis as a function of the location across the whisker

Weight % Vs Line scan #

O  Pd  Sn  Pt  Au
Whisker FIB-TEM analysis

EDS mapping

(b) to (f) show the relative chemical elements distribution over the area shown in (a).
Whisker FIB-TEM analysis
Selective-Area Electron Diffraction (SAED)

(b) & (c) SAED patterns taken from the whisker in (a).

The SAED spot patterns indicate well-aligned crystalline material, which is in agreement with the filaments internal structure interpretation of the TEM images.
Whisker FIB-TEM analysis

Conclusions:

• FIB cross-sectioning is found to be an effective way of studying the internal structure of Sn whiskers (obtained from pure Sn films, deposited on Cu substrates) at the nanoscale.
• A combination of complementary imaging techniques (SEM, TEM, ion-beam imaging, backscattered electron imaging), chemical analysis (EDS), and structure (SAED) techniques were applied in this study.
• The results indicate that the whisker internal structure consists of aligned, highly oriented crystalline filaments that consist of pure Sn material.
• The results support the hypothesis that Sn whiskers (and metal whiskers, in general) can grow as result of massive nucleation and co-growth of thin filaments.
• No evidence of the existence of IMCs in material in the vicinity of the whiskers was found.