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Yield Assessment of Protective Coatings for Atom Probe Analysis

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Specimen preparation is the cornerstone to allow successful atom probe tomography (APT). Focused-ion-beam (FIB) based specimen preparation has become the most widely used method for atom probe specimen preparation, surpassing the traditional electrochemical polishing that is limited to conductive specimens. Although the literature discusses FIB-based preparation in detail, the pros and cons of capping materials and methods are often overlooked since the capping layer itself is usually not the subject of an APT analysis. Capping layers have four main functionalities 1) to prevent damage caused by ion implantation, 2) to preserve and mark the original surface, 3) to help control specimen geometry, e.g. shank angle, 4) to act as a sacrificial material in low energy cleaning and APT alignment. The focus of this work is to assess various surface capping options and their influences on yield and data quality.

Various capping methods and materials exist for surface deposition. The common ones for APT include FIB-based deposition, physical vapor deposition (PVD), chemical vapor deposition (CVD), and atomic layer deposition (ALD) [1, 2]. FIB-based deposition, using GIS precursors, involves the incoming ions, also electrons, interacting with precursor gas injected from a gas nozzle positioned near the specimen surface. Depending on the type of precursor used it enables various materials to be deposited. We will be investigating tungsten, platinum, carbon, and silicon oxide based insulator deposition material. The ability to conduct deposition on a selective area imaged by SEM is the biggest benefit of FIB-based deposition. In contrast, sputter coating applies a thin layer, up to hundreds of nanometers, of deposition materials that covers the entire surface. The sputter coating occurs in a vacuum and involves bombardment of the sample with energetic positively charged ions that came off from a target. The target is interchangeable, and we will be comparing nickel and tungsten in this work.

To facilitate the sharpening process, capping was conducted on CAMECA® microtip arrays that host 2 µm wide posts etched out from dopped Si wafers [3]. Studying capping options on Si is directly relevant to many semiconductor applications. In addition, the single-crystal Si post material has been well-studied in APT showing excellent survivability across a wide range of acquisition conditions. The milling routine shown in **Table 1** was performed to sharpen all the specimens to keep a consistent specimen geometry. This semi-automated milling recipe, in which gradually reduces the inner and outer pattern diameters as the sharpening proceeds, greatly improves geometric consistency and reduces milling time. For each coating option, seven or more specimens were analyzed to provide an estimate of the yield and data quality. APT analysis was conducted using the CAMECA's "chain acquisition" and "scripted acquisition" automation features in AP Suite 6.1™ software to increase the throughput. SEM images prior to atom probe analysis were taken to acquire accurate information about the specimen geometry including cap thickness, radius at the interface of cap and substrate, shank angle of the specimen, etc.. Post analysis images were also taken to categorize the mechanism of failure.

Survivability, Ga ion stopping power, relative field to Si, and cap composition are the most valuable results from this experiment. Survivability can be further categorized as a smooth transition to Si with minimal change in voltage curve, a rough transition with substantial fluctuation in voltage curve, or a sudden transition with the potential of material loss. Overall, sputtered Ni, W, and GIS deposited Pt, and Si oxide insulator were shown to provide good to moderate survivability. Survivability was also able to be correlated to, the cap thickness. For example, a 125nm GIS-Pt cap failed all seven trials, however, a 40nm thick GIS-Pt cap succeeded in all nine trials with the same data acquisition conditions. Ga stopping power was assessed by measuring Ga concentration in the first 10 nm of Si beneath the cap interface. There are two ways to obtain meaningful information to inform the relative field to Si, which are to compare the shank angle produced by low energy cleaning, and to compare the voltage fluctuation before and after the transition from the cap to the substrate. **Figure 1** shows the typical specimen geometry of a Si specimen capped with sputtered Ni and the corresponding APT reconstruction. This work is meant to provide valuable information on choosing methods and materials for capping, which can affect the subsequent analysis in APT.

Application	Voltage	Current	Outer (um)	Inner (um)	Depth (um)
eBeam depo	10kV	0.2nA	1.9	0.0	0.1
iBeam depo	30kV	26pA	1.8	0.0	0.3
Milling	30kV	90pA			0.2
1			3.2	1.5	
2			3.0	1.0	
3			2.8	0.5	
4			2.6	0.4	0.05
5			2.4	0.3	
6			2.2	0.2	
7			2.0	0.1	0.05-0.1
Cleaning	500V	8.0nA	10.0	0.0	Variable

Table 1. FIB fabrication recipe designed to improve consistence in specimen geometry and reduction in milling time.

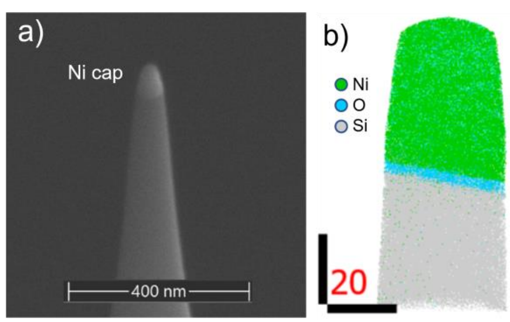

Figure 1. a) is the representative specimen geometry of a sputtered Ni cap (brighter contrast) on top of a Si post that provides good yield in APT. b) is the correspond APT reconstruction showing Ni cap, Si post and native oxide in between.

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