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Experimental Evaluation of Conditions Affecting Specimen Survivability in Atom Probe Tomography

T.J. Prosa[1], D. Lawrence[1], D. Olson[1], S. Strennen[1], I. Martin[1], D.J. Larson[1], R.L. Martens[2], J. Goodwin[2], A. Portavoce[3] and D. Mangelinck[3]

[1.] CAMECA Instruments, Inc., Madison, WI, USA.
[2.] Metallurgical and Materials Engineering, University of Alabama, Tuscaloosa, AL, USA.
[3.] Aix-Marseille Université, IM2NP, F-13397 Marseille Cedex, France.

It is a miraculous quirk of nature that any material can survive application of the extreme electric fields necessary to extract atoms from a specimen surface one-at-a-time without bulk rupture of the material itself. The field of atom probe tomography (APT) relies upon this natural quirk and continues to find an ever widening variety of materials can be successfully investigated [1], yet the issue of premature specimen failure remains critical for continued field growth and adoption [2].

Since the primary goal of an ordinary APT analysis is to gather compositional information related to a materials science problem, improving the success rate of the investigation is a secondary luxury when a single, high quality dataset is sufficient for the analysis need. In the current study, analysis yield improvement is the primary purpose of the investigation. We report on our results to date relating how analysis conditions affect yield for our standard specimen.

For this study we chose a material that has many material characteristics common to the microelectronics industry but also with a history of low (but non-zero) yield in our laboratory. As illustrated in Figure 1, the material consists of a 12 nm oxide grown on a Si <100> substrate with an additional 100 nm of phosphorous doped poly-silicon was grown and implanted with boron [3,4]. Historically, the analysis yield of the doped and implanted poly-silicon region was relative high while that of the oxide was extremely low. Our goal was to statistically analyze the analysis yield for this specimen type and then consider how various variables, both specimen preparation and analysis conditions, effect yield. The initial considerations included specimen size, as measured by the tip diameter at the oxide, cap size, detection rate (DR), and laser pulse energy.

For the initial investigation, some 66 specimens were manufactured with a similar geometry and a variety of oxide dimensions to look for correlation between analysis yield and specimen size. In Figure 1, the protective nickel cap is visible at the specimen apex with the bright 12 nm oxide observable 100 nm below. A narrow shank angle geometry was chosen due to its reproducibility. This allowed for predictable analysis evolution near and through the primary region of interest (the 12 nm SiO_2). Based on these 66 analysis attempts, a number of statistically significant conclusions were drawn for this sample type: first, yield through the poly-silicon is very high. In fact, additional data not reported here puts the overall yield for this region near 95%. Specimens rarely fail here regardless of analysis conditions. Second, DR does affect yield through the oxide. Lowering the DR from 0.3% to 0.1% increases yield from 24% to 82% which is statistically significant at better than a 95% confidence level. Third, specimens fail much more often through the low-to-high field Si/SiO_2 interface than the high-to-low field SiO_2/Si interface. In fact, every specimen that survived through the top high-to-low interface continued analysis into the substrate. Fourth, the physical size of the oxide interface does not affect yield. This is illustrated in Figure 2.

References:

[1] E.A. Marquis *et al.*, Current Opinion in Solid State and Materials Science **17** (2103), p. 217.
[2] D.G. Brandon, Field Ion Microscopy, Eds. J. Hren, S. Ranganathan (Plenum, 1968) p. 64.
[3] A. Portavoce *et al.*, Defect and Diffusion Forum **289-292** (2009), p. 329.
[4] A. Portavoce *et al.*, Diffusion and Defect Data **264** (2007), p. 33.

Table 1. Analysis Yield through Various Regions of the Standard Structure

Region	Successes	Attempts[a,b]	Region Yield
1) Nickel Cap/Poly Si Interface	43	61	70%
2) Poly Si	42	48	88%
3) Si/SiOx Interface DR = 0.1%	14	17	82%
Si/SiOx Interface DR = 0.3%	6	25	24%
4) SiOx/Si Interface	20	20	100%

[a]Total Number of Specimens = 66

[b]Excluded from Ni Cap Evaluation (too few ions) = 5

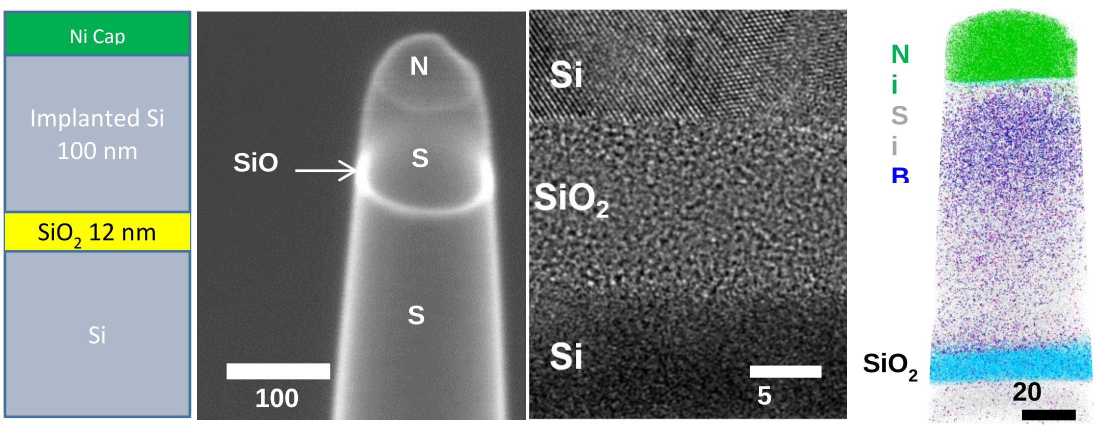

Figure 1. A description of the standard structure (far left), SEM image of a typical APT specimen before analysis, TEM of the low yielding SiO_2 region of the structure, and atom map revealing the phosphorus doping and boron implant (far right).

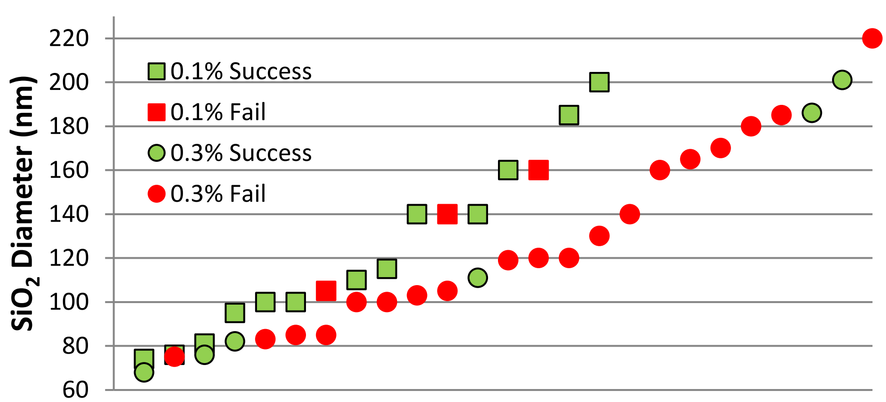

Figure 2. Ordered plot of successful analysis yield through the SiO_2 as a function of specimen diameter. Acquisition was attempted with ion detection rates of either 0.3% (circles) or 0.1% (squares).