Use of Secondary Ion Mass Spectrometry (SIMS) in the Analysis of Alumina-Boria-Silica Ceramic

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Outline

• Introduction to Surface Analysis Techniques
  ♦ XPS, ToF-SIMS (Static SIMS) and Dynamic SIMS (Depth Profiling)
• Background - Previous Surface Analysis of Nextel 312 Fabric
• Analysis of NH₃ Treated Nextel® 312 Film Samples or “Flakes”
  ♦ Quantitative (Dynamic) Depth Profiles
  ♦ Identification of BN Using ToF-SIMS High Mass Resolution
  ♦ Reactions With H₂ Only - No Ammonia
• Comparison of NH₃ Treated Nextel® 312 Films and Fabric
  ♦ XPS, ToF-SIMS and Dynamic SIMS Depth Profiling
• Surface Model - BN *In-situ* Deposition
• Summary
Earlier Analysis of Nextel® 312 Weave Sample 8329-35B

XPS Analysis

ToF-SIMS Depth Profile
SIMS Analysis of Baseline Film

ToF-SIMS Spectrum

Dynamic SIMS Depth Profile

Al$_2$O$_3$ - 61.9%

SiO$_2$ - 24.3%

B$_2$O$_3$ - 13.7%
SIMS Dynamic Depth Profiling Analysis
Nextel® 312 Film - 1225C

Raw Data

Normailzed, Processd Data
ToF-SIMS Identification of BN Treated Nextel® 312 Films High Resolution Spectra

![Graph 1: Intensity (arbitrary units) vs. AMU showing peaks at 24.000 and 24.015 AMU labeled as $^{12} \text{C}_2^-$ and $^{10} \text{BN}^-$, respectively.]

![Graph 2: Intensity (arbitrary units) vs. AMU showing a peak at 27.005 AMU labeled as $^{11} \text{BO}^-$, indicating the presence of BO species.]

Ammonium borohydride ($\text{NH}_3\cdot\text{BH}_3$) is a reducing agent that reduces the number of defects in the BN films. This has been demonstrated through high-resolution TOF-SIMS analysis of Nextel® 312 films treated with ammonium borohydride.
High Mass Resolution ToF-SIMS Spectra

Treated Nextel® 312 Film

![Graph showing mass resolution spectra with peaks for $^{10}$BN$^-$, $^{11}$BN$^-$, $^{10}$BO$^-$, $^{11}$BO$^-$, and CN$^-$ at AMU 24.015, 25.015, 26.010, and 27.005 respectively.]
High Mass Resolution ToF-SIMS Spectra
Treated Nextel® 312 Film

Intensity (arbitrary units)

Intensity (arbitrary units)

41.005 $^{11}\text{BNO}^-$

(40.985) $\text{AlN}^-$

42.005 $^{10}\text{BO}_2^-$

42.980 $^{10}\text{BO}_2^-$

43.000 $^{11}\text{BO}_2^-$

AMU

AMU
SIMS Analysis - Hydrogen Only
Nextel® 312 Film - 1225C

Dynamic SIMS Profile

ToF-SIMS Spectra

Surface

Lightly Etched

Al

Na

Si

B

Al

Si

Depth (Å)

Weight %

Intensity (arbitrary units)

AMU
XPS Analysis of Nextel® 312 Weave Samples

XPS Survey Scan

XPS Atomic Ratios

Nextel 312 Weave - 1100oC

AF-10 - Lot 6-009 Run #46
High Mass Resolution ToF-SIMS Spectra
Weave Sample - Run #46

- $26.980$ (Al$^+$)
- $27.020$ (C$_2$H$_3^+$)
- $27.975$ (Si$^+$)
- $28.030$ (C$_2$H$_4^+$)
- $29.040$ (C$_2$H$_5^+$)

AMU

Intensity (arbitrary units)
Comparison Between Weave and Films
ToF-SIMS Analysis

![Graph comparing the ToF-SIMS analysis of Weave and Film. The graph shows the intensity versus AMU for both samples. The Weave sample has a peak at 11B+ and a peak at 10B+. The Film sample has a peak at 11B+ and a peak at Al+. The graph also indicates that the Weave sample was run #46 and the Film sample was lightly etched at 1250C.](image)
Dynamic SIMS Depth Profiling Analysis

Nextel® 312 Film - 1250C

Nextel® 312 Weave - 1250C
Possible Nitridation Mechanism

Gas Flow

NH$_3$

In-diffusion

~ 20 nm

B$_2$O$_3$ and SiO

Out-diffusion

H$_2$O

BN-Rich
Layer

Nextel Fiber

Reaction:

2NH$_3$ + B$_2$O$_3$ → 2 BN + 3 H$_2$O
SUMMARY

• XPS, ToF-SIMS and Dynamic SIMS Profiling Provided a Clear Picture of the BN Distribution
• ToF-SIMS positively Identified BN
• Weave and Film Samples Exhibited Different Diffusional Profiles