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A Comprehensive Evaluation of the Performance and Materials Chemistry of a Silicone-Based Replicating Compound

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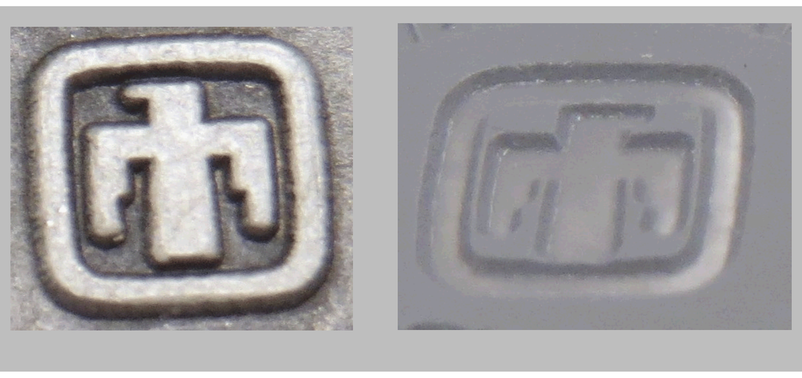
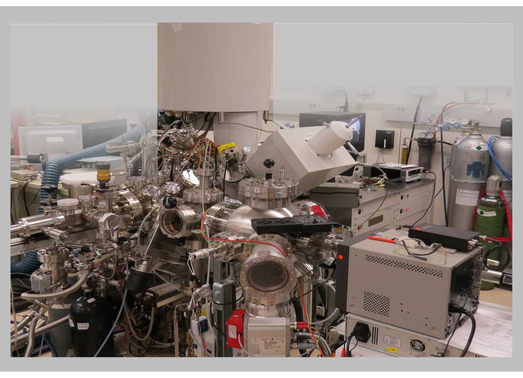
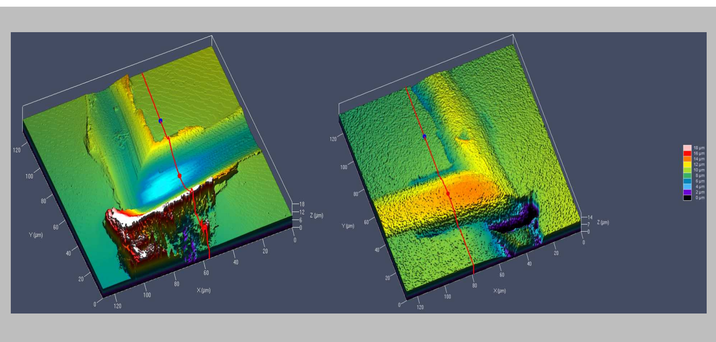
Sandia National Laboratories

Exceptional service in the national interest



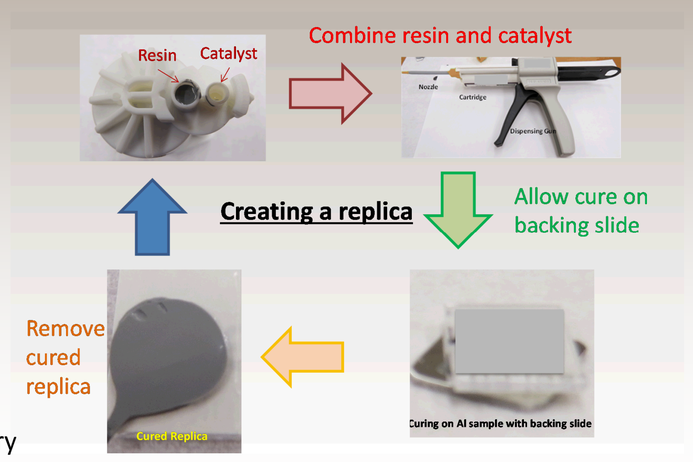




Abstract: The objective of this project was to characterize the performance and chemistry of a silicone-based replicating compound. Some silicone replicating compounds are useful for critical inspection of surface features. Common applications include examining micro-cracks, surface pitting, scratching, and other surface defects. Claimed resolution is 0.1µm. Materials characterization techniques – FTIR, XPS, ToF-SIMS, AFM, and Confocal Microscopy – were used to verify the claimed specifications as well as look as side-effects (surface contamination) that may be left behind after the cured compound is removed from a surface.

Background

- High resolution, fast curing replicating compounds can be used to create negative replicas of physical defects on surfaces.
- The negative replicas can be used for further analysis of the defects.
- Are these replicas accurate? Is there any residue left behind on a surface after use, and, if there is, can the contamination be cleaned off?



Evaluation Techniques:

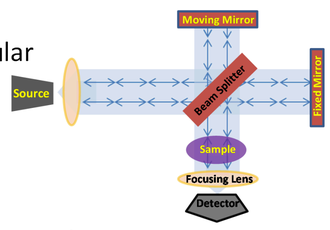
- *FTIR*: Fourier Transform Infrared Spectroscopy
- *XPS*: X-ray Photoelectron Spectroscopy
- *ToF-SIMS*: Time-of-Flight Secondary Ion Mass Spectrometry
- *AFM*: Atomic Force Microscopy
- *Confocal Microscopy*

Evaluation Techniques

Composition and Residue Analysis

FTIR:

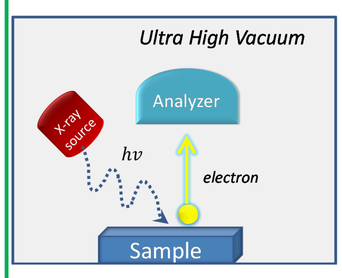
- Uses **infrared light** and **Fourier Transform** to convert raw data into a spectrum
- IR light causes molecular transitions in the **rotational** or **vibrational states**.
- Useful for identifying molecular species

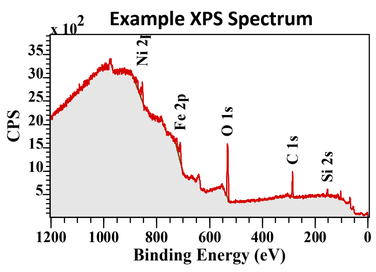


XPS:

- Ultra-high vacuum technique for evaluating composition, chemistry, and electronic structure.
- Sample is subjected to **X-ray illumination**.
- **X-ray Energy > Binding Energy of Electrons**
  Then electrons will be emitted via **photoelectric effect**.
- Electrons can be detected and sorted according to their kinetic energy.

$$KE = h\nu - BE - \Phi$$

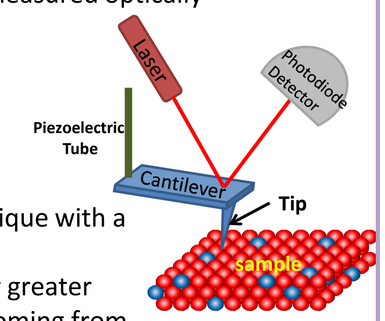




Composition and Residue Analysis

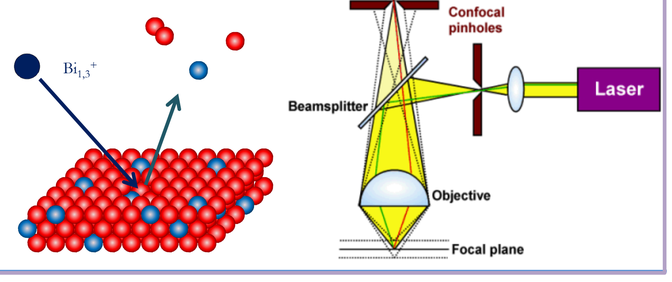
Atomic Force Microscopy (AFM)

- Angstrom resolution
- Stylus cantilever controlled by a **piezoelectric tube** is scanned across a surface.
- **Atomic forces** between the stylus and surface move the tip and are measured optically providing topography.



Confocal Microscopy:

- An optical imaging technique with a **very small aperture**
- Small aperture allows for greater resolution as only light coming from the sample that is in focus enters the optical system.
- 2D&3D images can be obtained by scanning the light source (typically a laser) across the surface of the sample.

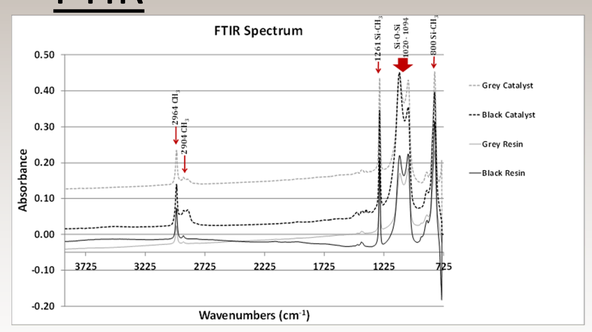


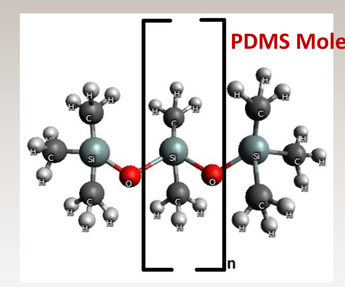
ToF-SIMS

- Ultra-high vacuum technique which involves the bombardment of a surface with an **energetic beam of ions**.
- Time-of-flight of the ion is measured, ions with lower mass-to-charge (m/z) ratio reach the detector faster.

Results

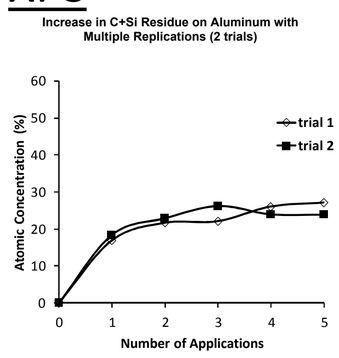
FTIR

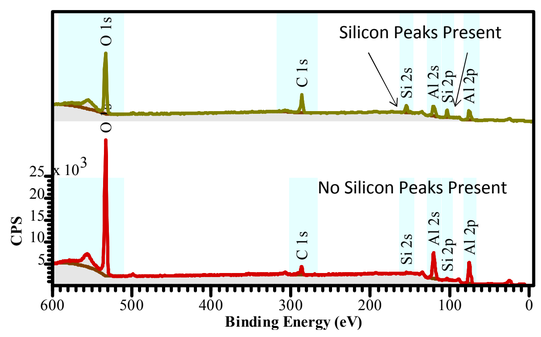




- **Primary component is silicone, most common silicone is PDMS**
- **Slight differences in the grey and black resins:**
- **Black contains carbon filler, Grey contains TiO_2 (rutile)**

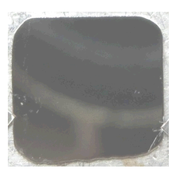
XPS



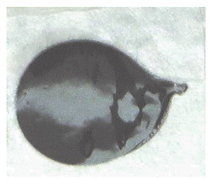


- **Replicating compound leaves a silicone residue which is seen by XPS.**
- **Residue builds with multiple applications**
- **Even after surface is cleaned by solvents there is still C+Si residue**

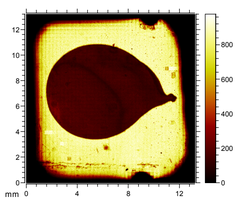
ToF-SIMS



Aluminum after 1 application



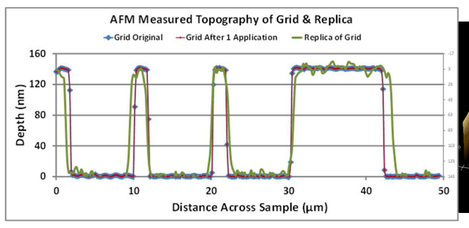
Silicone Replica



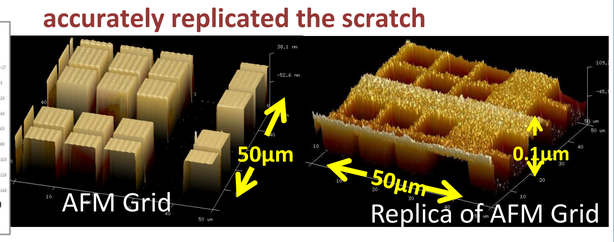
Tof-SIMS image of aluminum after 1 application

- **ToF-SIMS detects residue even though there is no residue optically visible**
- **Residue left behind is primarily silicone**

AFM & Confocal



- **Measurements showed the compound accurately replicated the scratch**



- **The replicating compound accurately replicated the topography of the grid**
- **There is no remaining residue measurable by AFM**

Conclusion

The work done for this project resulted in successful characterization of a silicone based replicating compound.
FTIR data showed composition differences between the different colors.
XPS and ToF-Sims data showed the replicating compound leaves behind a thin silicone residue when the replica is peeled off the surface.
AFM and Confocal Microscopy data showed the compound does accurately replicates surfaces down to the claimed resolution of 0.1µm.
This project was a great learning experience and an introduction to capabilities of several common materials characterization techniques.

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