INVESTIGATING THE STRUCTURE OF CVD DEPOSITED AMINO SILANE ON SILICA SUBSTRATE VIA HIGH RESOLUTION CHARACTERIZATION METHODS

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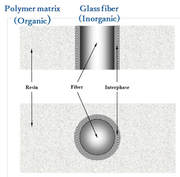
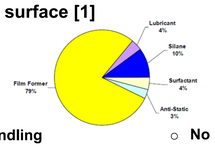
Introduction



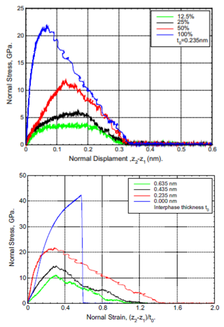
- Interphase between the fiber reinforcement and the matrix plays a key role in effectively distributing load within a fiber reinforced polymer composite
- Traditionally sizing packages are multifunctional and are applied to a fiber surface [1]



ADVANTAGES
- Ease of handling
- Prevent fiber damage
- Enhances Compatibility
- Reduces Moisture Degradation

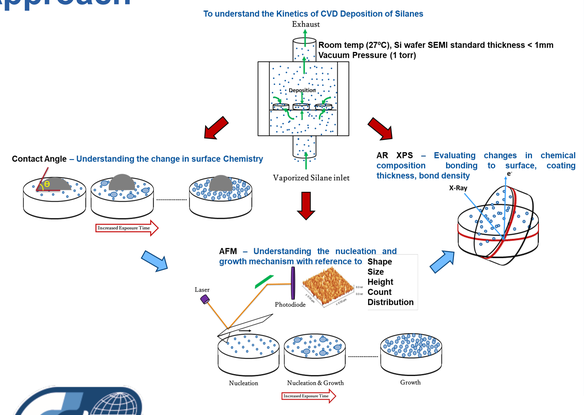
LIMITATIONS
- No Control over coating thickness and uniformity
- Sizing layer morphology not well characterized

- An adhesion promoter like organofunctionalized silanes is often a part of these sizing packages, and they can help achieve desirable interphase properties.
- Molecular dynamics (MD) simulation studies have shown that these glass-silane-epoxy interphase strength and energy absorption is highly dependent on bond density, interphase-coating thickness, and strain rate (monolayer and fully saturated bond density resulted in highest interphase strength) [2].

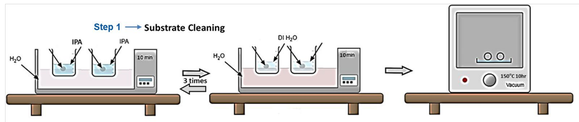


- Chemical vapor deposition (CVD) may be well suited approach since it can create a uniform silane coating with control of the thickness range as low as a monolayer.

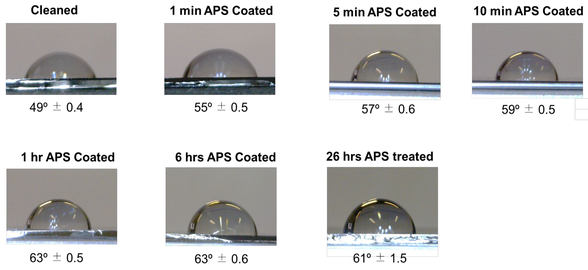
Approach

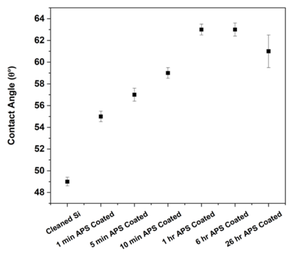


Methodology



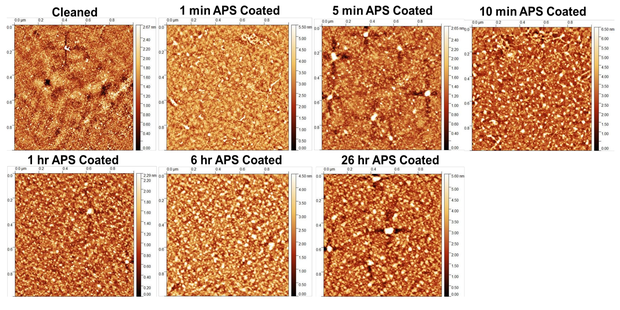
Contact Angle to Understand Change in Surface Chemistry



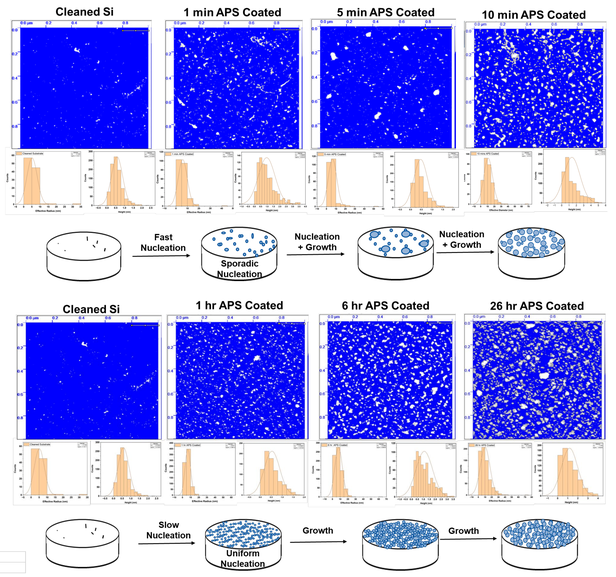


- Contact angle increased 7-10° from 1 min to 10 min as compared to clean substrate
- For deposition time 1 hr to 26 hr the contact angle saturated with an increase of almost 14°

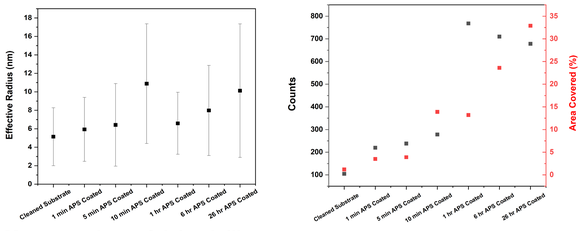
AFM Images Showing Silane Deposition on Si Substrate for Different Exposure Time



AFM Images Analyzed through ImageJ

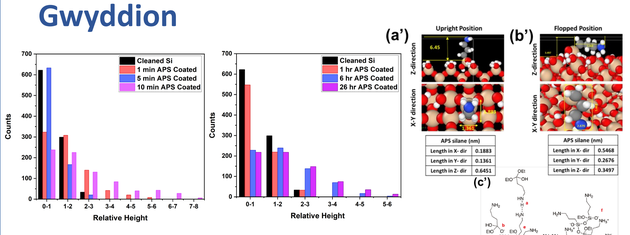


Effective Radius, Counts and Area Covered as a Function of Exposure time



- More number of island like structure appeared with longer exposure times which seems to saturate post 1 hour
- The increase in effective radius were observed for increase in exposure time indicating growth

Height Distribution Analyzed through Gwyddion

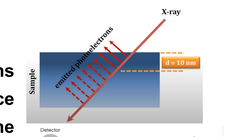


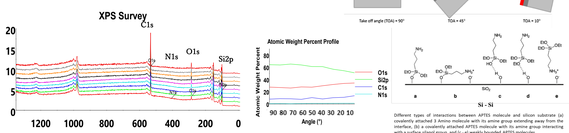
	Cleaned Si	1 min APS Coated	5 min APS Coated	10 min APS Coated	1 hr APS Coated	6 hr APS Coated	26 hr APS Coated
Avg. Height (nm)	0.5761	0.9678	0.4715	1.3975	0.5366	1.0845	1.1795
Std. Dv.	0.319	0.654	0.302	1.102	0.352	0.695	0.803

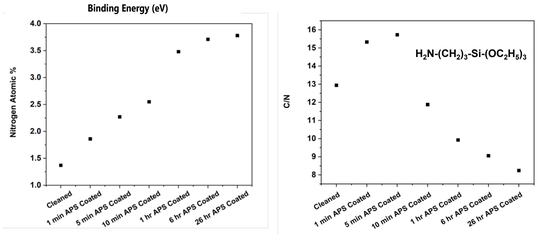
Silane molecules incorporated into the layer via (a) hydrogen bonding, (b) electrostatic attraction, (c) covalent bonding with the substrate, and (d) horizontal and (e) vertical polymerization with neighboring silanes; (f) oligomeric/polymeric silanes can also react/interact with functionalities present at the interface.

XPS

XPS analyzes material between depths of 1 and 10 nm, Since XPS is a surface technique, the orientation of the material affects the spectrum collected.







- With APS coating there is an overall increase in N atomic%
- The C/N ratio is ~ 9.5-8.5, approximating APS silane

Conclusions

- Coating the silicon substrate with APS decreases the free surface energy thus increasing the CA by 7 to 10° for 1min-10 min and saturates to ~14° at and above 1 hr.
- AFM studies confirmed the sporadic distribution at low time intervals and uniform distribution at higher time intervals. The number of nucleation sites increases at low time interval along with effective diameter. While the number of nucleation sites saturate and only growth happens post 1 hr.
- XPS studies showed a steady increase in Nitrogen atomic % on silicon substrate with almost 2 fold increase after 1 hr deposition, post which it saturates. C/N ratio was seen to decrease down to ~9 which verifies that most of the signals comes from coating.

Path Forward

- High resolution AFM will be done to understand silane orientations in island like structures
- To calculate the thickness and bond density of the silane deposited as a function of time

References

[1] Gao X (2006) Tailored interphase structure for improved strength and energy absorption of composites. Ph.D. Thesis, University of Delaware, USA

[2] Journal Mater Sci (2017) 52:12981–12998

Acknowledgements

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