CHARACTERIZATION OF ORGANO-FUNCTIONALIZED SILANES FOR CONTROLLED VAPOR DEPOSITION ONTO S-2 GLASS® FABRICS

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Introduction

- S-2 Glass® fiber reinforced epoxies are used for military composite armor
- Fiber/matrix interface has been shown to improve composite properties, including tensile and penetration resistance
- Organo-functionalized silanes are used in the glass fiber sizing formulation and likely contribute to the formation of a strong fiber/matrix interface
- A fiber-scale chemical vapor deposition (CVD) method has shown promising results on improving the interface
- Transition to fabric level deposition requires further process development

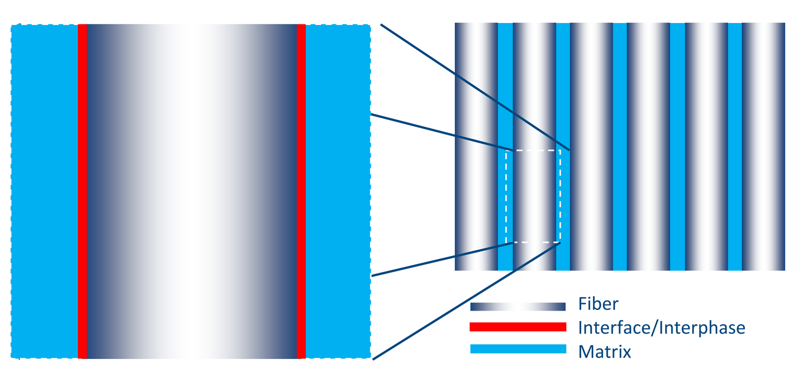
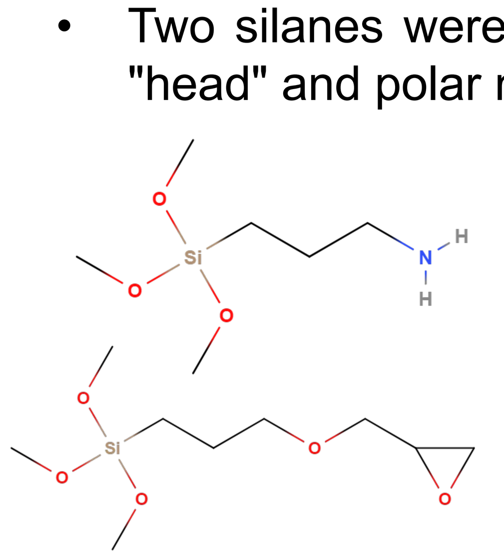


Figure 1. Fiber/Matrix interphase schematic

Chemical Vapor Deposition

- Two silanes were chosen for their non-polar silicon "head" and polar matrix compatible "tail"



3-aminopropyltrimethoxysilane (APS)

3-glycidoxypropyltrimethoxysilane (GPS)

Figure 2. Silane structure[1]

- Fiber tows were held in an isolated chamber, in which the silanes were introduced under vacuum

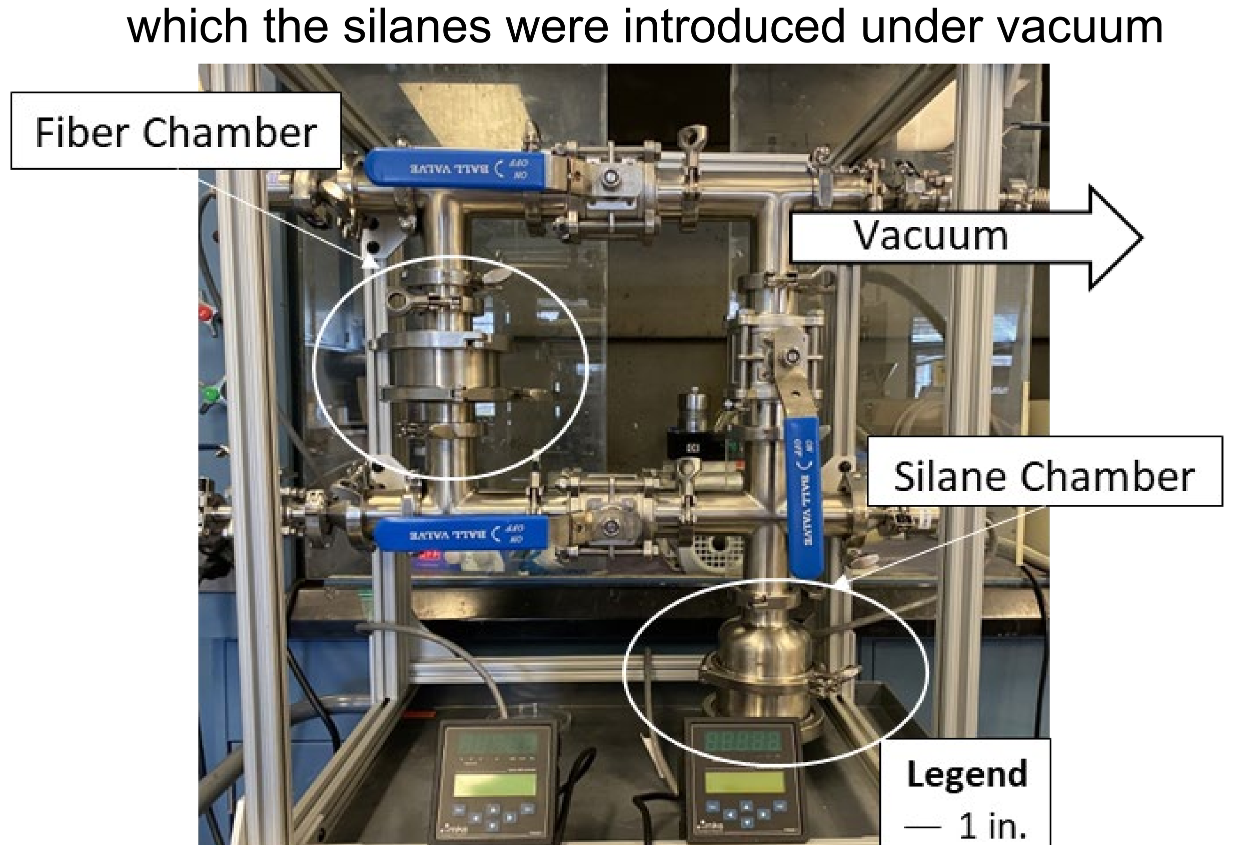


Figure 3. Reactor Setup

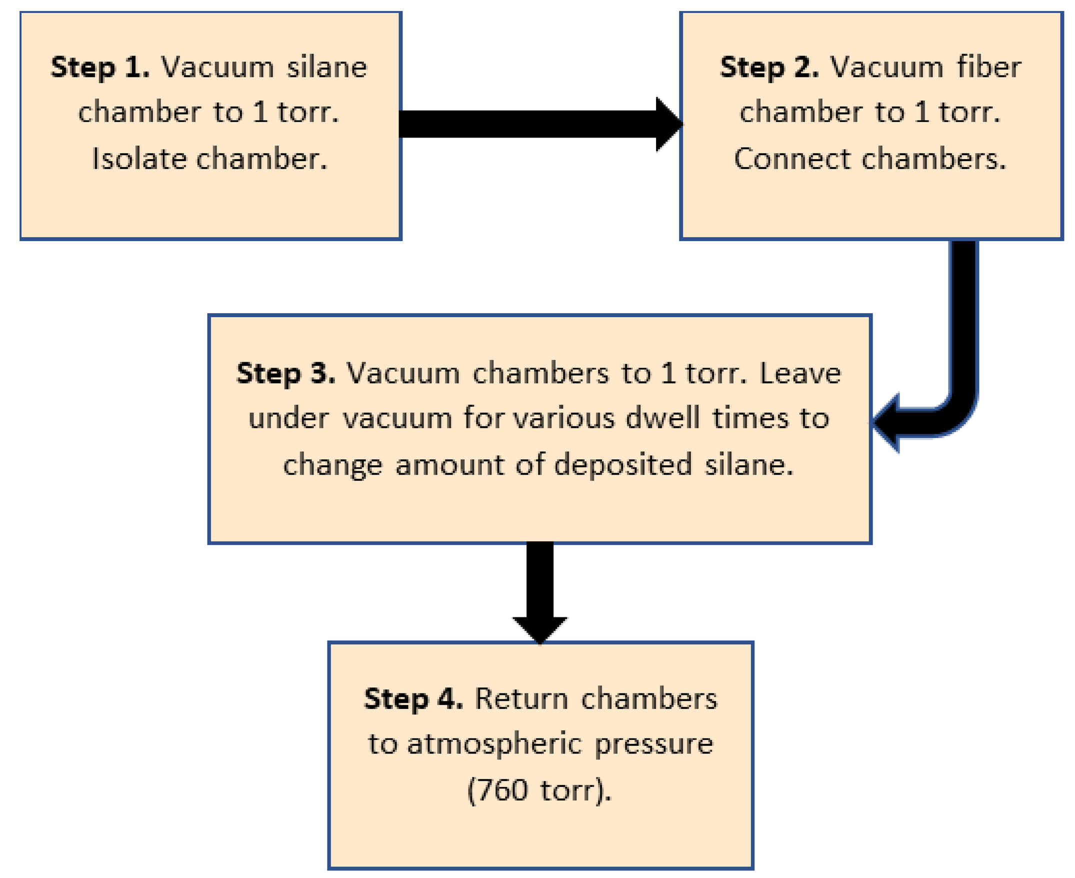


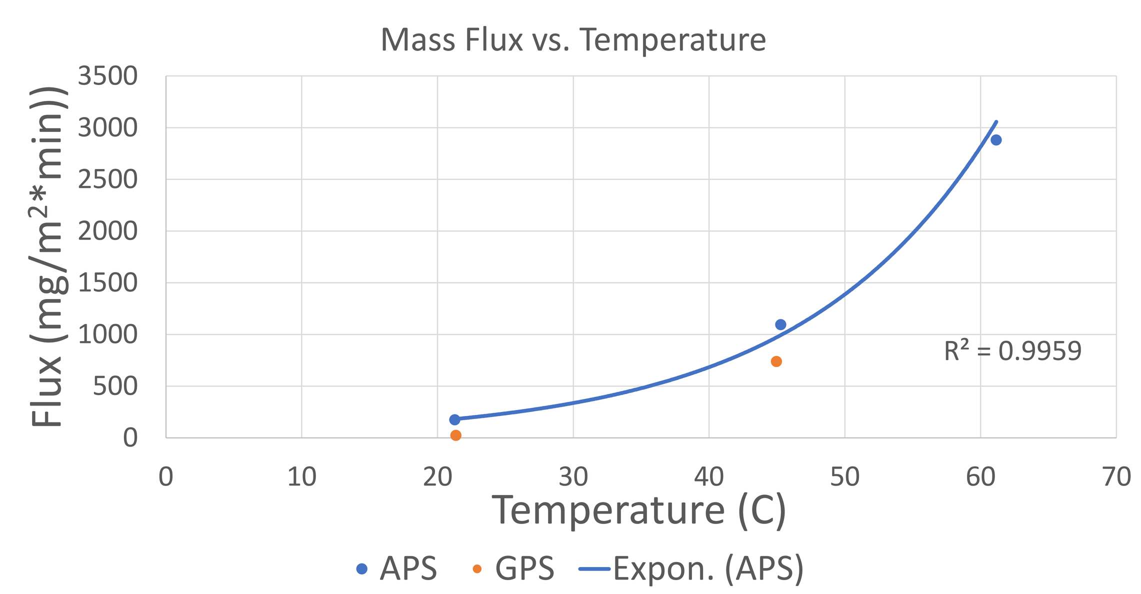
Figure 4. Process flowchart

Thermal Characterization

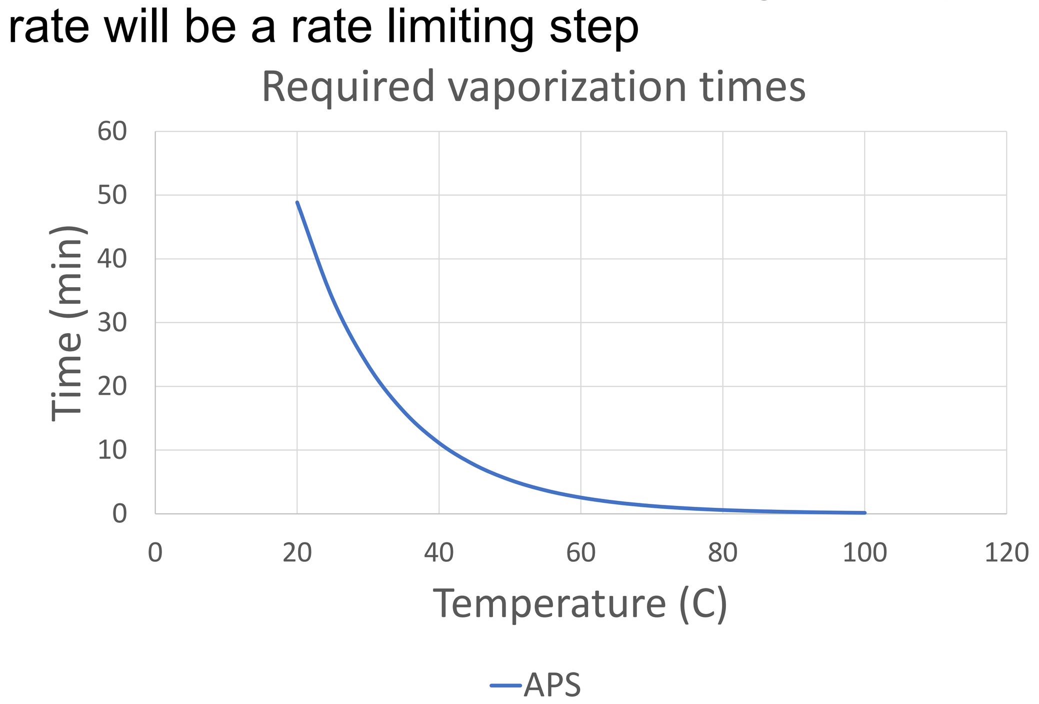
- Concentration of a low pressure system can be modeled by the Ideal Gas Law

$$PV = nRT \rightarrow P/_{RT} = n/_{V} = C$$

- Limited published data[2] allows for the estimate of the vapor pressure as a function of temperature for silanes of interest for the processing temperatures of interest
- Isothermal TGA experiments were conducted to determine the mass flux at different temperatures

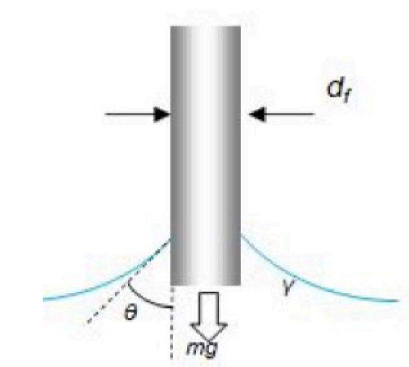


- With flux and we can establish the time required for the vaporization of silane, showing the vaporization rate will be a rate limiting step



Contact Angle/Interface Measurements

- Changes in surface chemistry were measured through dynamic contact angle analysis
- Contact Angle, the angle that a fiber makes when submerged into a liquid, is measured using a modified Wilhelmy plate equation



$$\theta = \cos^{-1}\left(\frac{m \cdot g}{\pi\, d_f \gamma}\right)$$

d_f: fiber diameter

m: mass

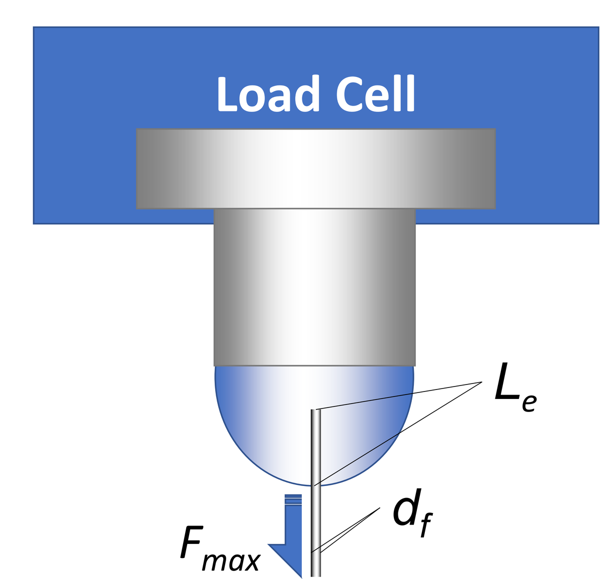
g: gravitational constant

θ: contact angle

γ: surface tension

Figure 5. Modified Wilhelmy Plate Equation

- Effectiveness of the deposited silane coating as an interface was measured using the fiber pullout test



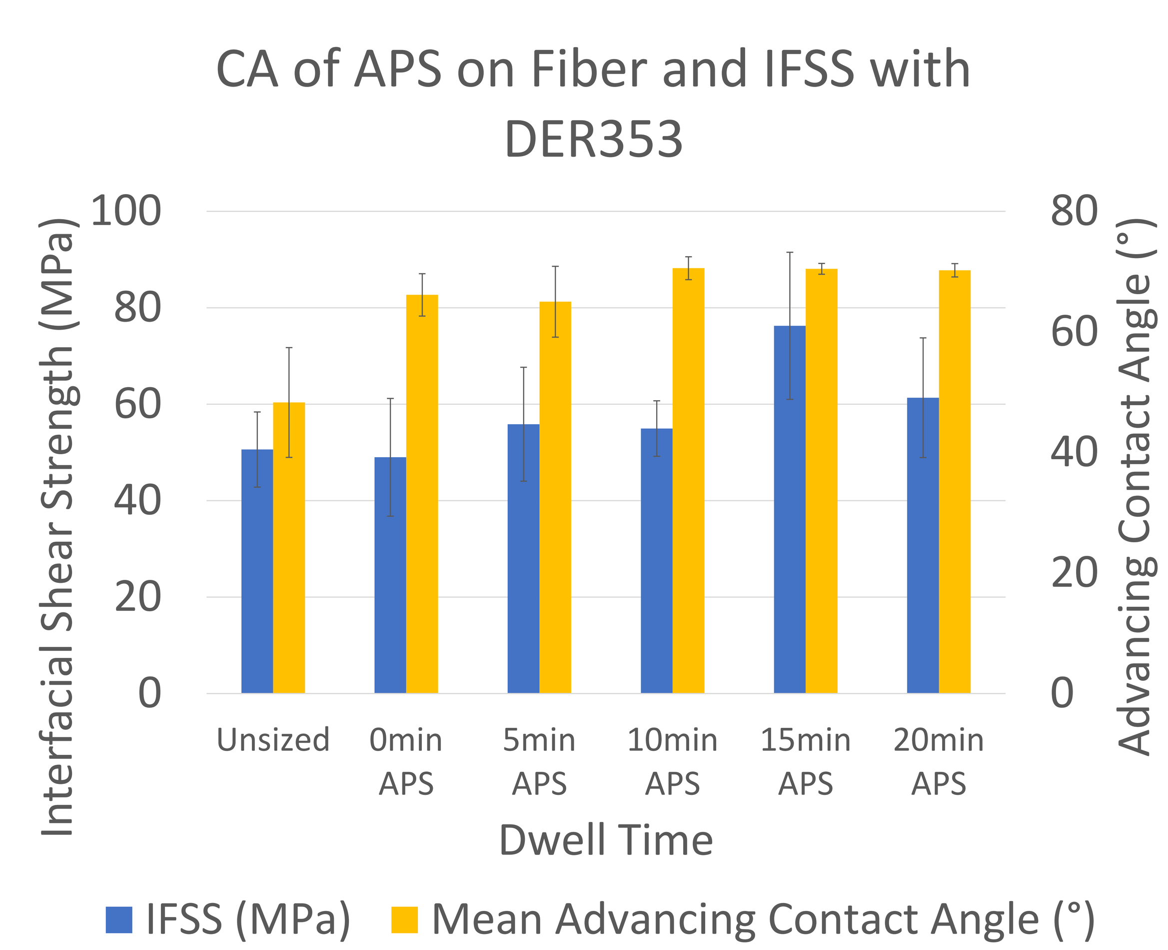
$$IFSS = \frac{F_{max}}{Area} = \frac{F_{max}}{\pi d_f L_e}$$

F_{max}: max force

d_f: fiber diameter

L_e: embedded length

Figure 6. Pullout Schematic[3]



- These data show an increase in CA for all treated fibers compared to unsized, verifying success of the deposition process due to differing surface chemistry
- The 15min treated fibers show an increase in IFSS compared to unsized, suggesting it is optimal

Conclusions/Moving Forward

- We have established essential parameters for the CVD process; notably the times necessary for each step
- Statistical difference in contact angle measurements for silane-treated fibers vs. unsized confirms the effectiveness of the CVD process
- CA and IFSS measurements imply a 15-minute deposition time is optimal—though length of time to vaporized must be evaluated
- The new goals are to 1) study elevated temperature CVD and 2) to scale up the process—i.e., working with sheets of fabric vs. single fibers

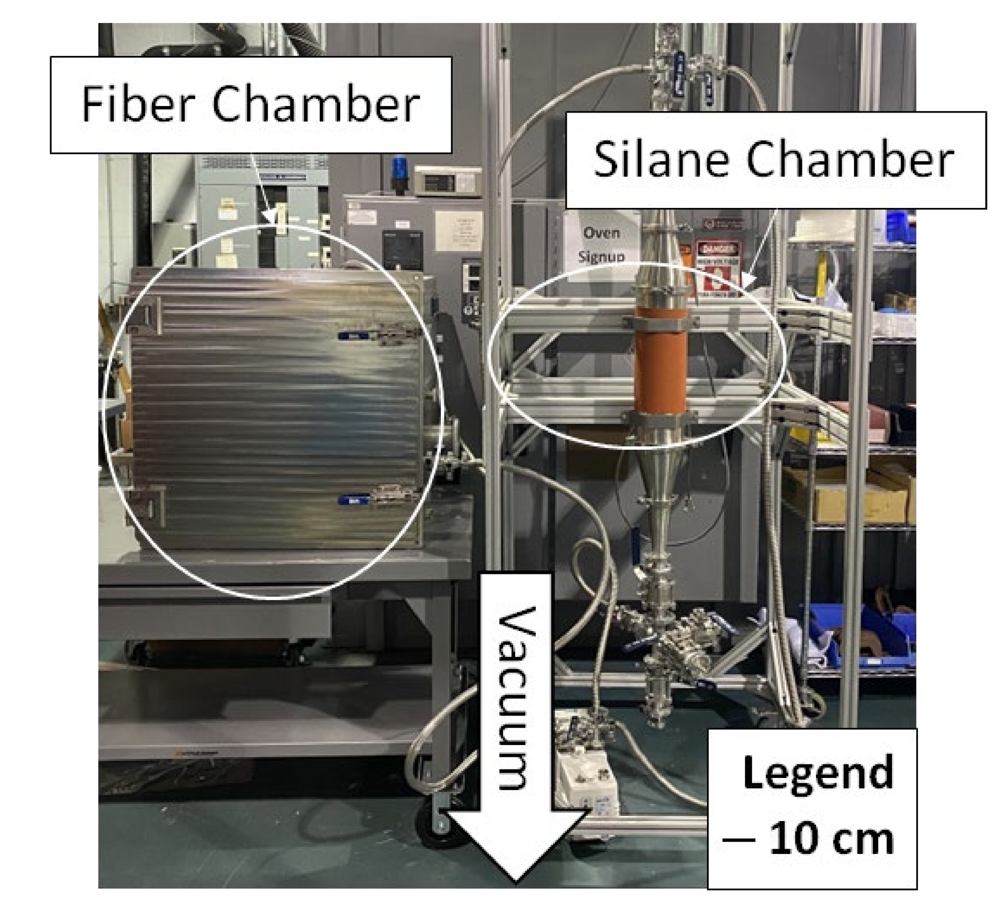


Figure 7. Preliminary Upscale Reactor

References

1) https://molview.org/?cid=87217987

2) https://www.shinetsusilicone-global.com/catalog/pdf/SilaneCouplingAgents_e.pdf

3) https://pdfs.semanticscholar.org/c0c4/6fcf6a55b7be0956a49c41388565170d08f1.pdf

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