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VitriEdge: Repairable & Durable Vitrimer Coatings for Wind Turbine Blade Leading Edges

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Summary: The primary goal of this Level 1b incubator project was to explore the use of vitrimer coatings for repair of leading-edge erosion on end-of-life wind turbine blade surfaces, beyond coating strength of adhesion which has previously been demonstrated in the Level 1a project. Uniform vitrimer coatings (thickness: 400 μm) were applied to two end-of-life wind turbine blades for flexural, fatigue, and laminate tensile testing where the addition of the coating did not produce any statistical variation in tensile properties with minor drops in flexural strength for some laminate formulations. However, a <2% variation in storage modulus was measured for laminate structures (*i.e.*, blade samples with vitrimer coatings) across 100,000 flexural cycles and upon laminate tensile failure, the vitrimer coatings displayed no visible signs of delamination. In addition, three methods to heal vitrimer coating damage was displayed: oven heating, addition of hot water, and a forced convection heat gun. All three heating and healing mechanisms demonstrated significant healing with scratch depths decreasing between 79-91% at healing times ranging between 1-min and 10-minutes. Finally, a water jet machine was used to simulate rain erosion for both the blade surfaces and vitrimer-coated blade surfaces where the diameter and depth of the damage was recorded as a function of exposure time, water pressure, height of exposure, and angle of exposure. Of interest, while the vitrimer coating did not significantly lessen the damage experienced during rain erosion, the addition of vitrimer composite coatings (5 wt.% mica addition) did result in a crack-resistant, durable coating capable of self-healing behavior and in all cases the angle of rain exposure was the most critical parameter explored. It is crucial to continue exploring this space where vitrimer coatings are of interest for both their self-healing properties and potential use as reversible adhesives.

Project Task: The objective of this VitriEdge Level 1b project was to explore two next steps for vitrimer coatings on wind turbine blades for leading edge erosion protection: multiple in-use healing mechanisms and coating robustness characterization.

Healing demonstrations from Level 1a explored the use of uniform blade heating (*i.e.*, ovens) to demonstrate healing; these methodologies were

chosen to ensure variable control between multiple coating thicknesses and materials but are highly improbable for in-field repair. However, by exploring more simplistic and convenient methods for healing in the first objective, demonstrations using hot water and a heat gun would promote industrial

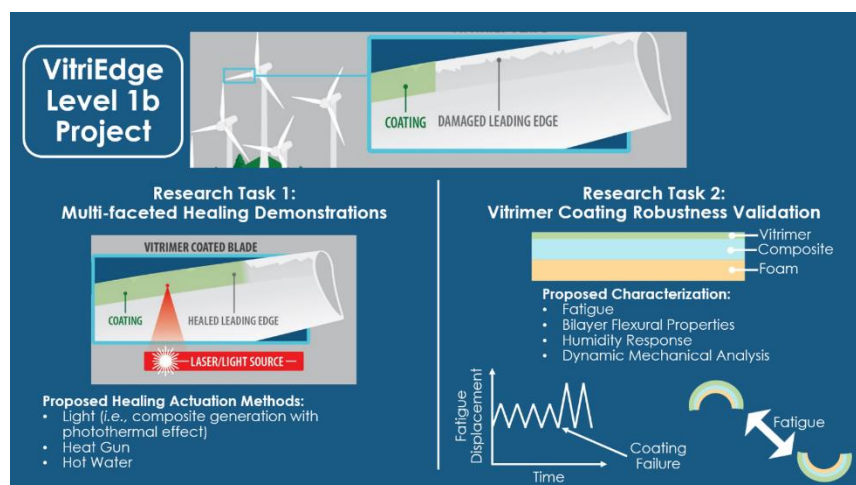


Figure 1. Summary of the proposed Level 1b research tasks.

engagement and protocols to increase operator safety. The second objective provides additional quantitative validation for vitrimer coatings as robust wind turbine blade coatings by considering the flexural and fatigue requirements of wind turbine blades. These additional validations are the necessary next step to engage with industrial partners for material incorporation into manufacturing. The task required for this project is the following: A minimum of 2 healing mechanisms will be explored for vitrimer coatings applied to end-of-life wind turbine blades where the percent of healing will be quantified; the mechanical performance of the vitrimer coating on the blade surface will be explored (*e.g.*, fatigue or flexural) to determine coating robustness beyond tensile strength. **Figure 1** displays the proposed goals of this Level 1b incubator project.

Methods: The primary means of evaluating these vitrimer coatings was to generate uniformly thick coatings ($\sim 400\ \mu\text{m}$ thickness) across the surface of 2 end-of-life wind turbine blades and test the laminate properties. In this case, the material properties of interest were tensile strength, flexural strength, and flexural fatigue of the laminates in addition to the healing abilities of the coating when manually scratched and healed. The coating was applied via a doctor blade, where the vitrimer material was pour cast onto the surface of the blades and spread to ensure uniform thicknesses prior to being cured at room temperature for 1 hour followed by a heated cured at $100\ ^\circ\text{C}$ for 2 hours (**Figure 2**). The doctor blade implemented in all cases was a GLTL Four-sided wet film coating applicator at the $400\ \mu\text{m}$ film thickness. In keeping with the Level 1a VitriEdge project, the vitrimer coating applied to the wind turbine blade surface was Mallinda's Vitrimax T130 and two end-of-life wind turbine blades implemented in this study were GE37 and Clipper blade materials (**Figure 2**).

Tensile samples were cut according to ASTM D638 Type V specimen dimensions and tensile tested on an MTS Criterion Model 45 tensile tester affixed with a 1 klb_f load cell and a 0.3-inch extensometer at a rate of 1 mm/min. Flexural and fatigue samples were cut to sample dimensions of 60 mm x 10 mm (*l* x *w*) where flexural testing was performed according to ASTM D790. Fatigue testing was performed using a TA Instruments Universal Series DMA 850 with the dual cantilever fixture; initially strain and frequency sweeps were used to determine maximum flexural ranges without damaging the machine. Flexural fatigue was performed for strains of 0.05% at a frequency of 5 Hz for 100,000 cycles of flexural testing.

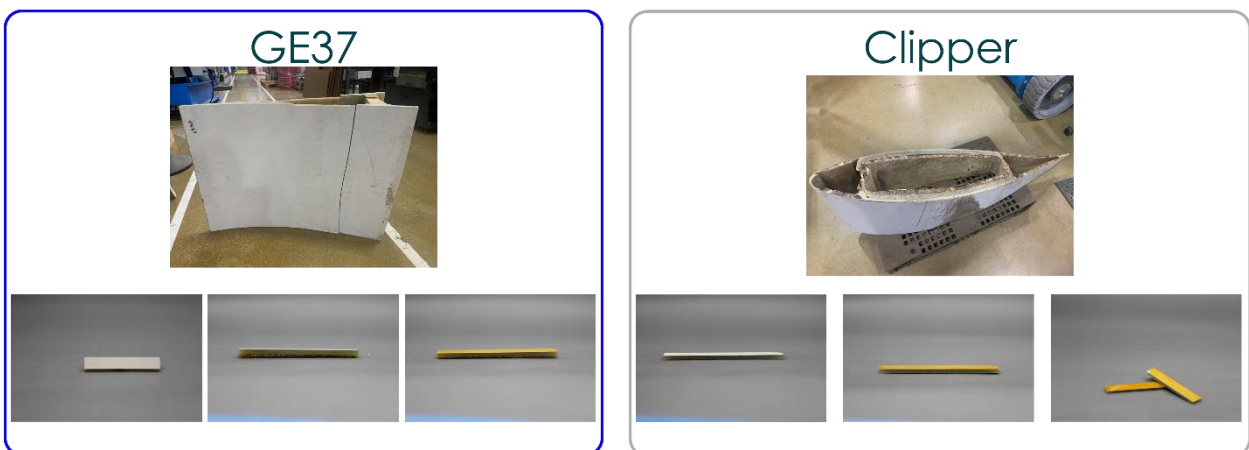


Figure 2. Images of the wind turbine blade samples with and without vitrimer coatings.

Healing experiments were conducted in the same fashion as those performed in the Level 1a VitriEdge project. Vitrimer coatings were manually scratched with the edge of a razor blade and the surface profile

was measured with a Mahr MarSurf PS10 handheld surface profilometer. Once the surface damage was measured, the sample was heated to at least the T_g (~ 96 °C according to previous Differential Scanning Calorimetry from the Level 1a project) where dynamic covalent bond exchange reactions are free to take place, and the sample was smoothed by hand. A second surface profilometry scan was performed to determine the decrease in the scratch depth due to the healing trial.

In addition to healing, it was of interest to explore the ability of these coatings to mitigate or withstand rain erosion conditions to mimic real-world conditions. However, due to the high cost, low equipment availability, and long wait times, traditional rain

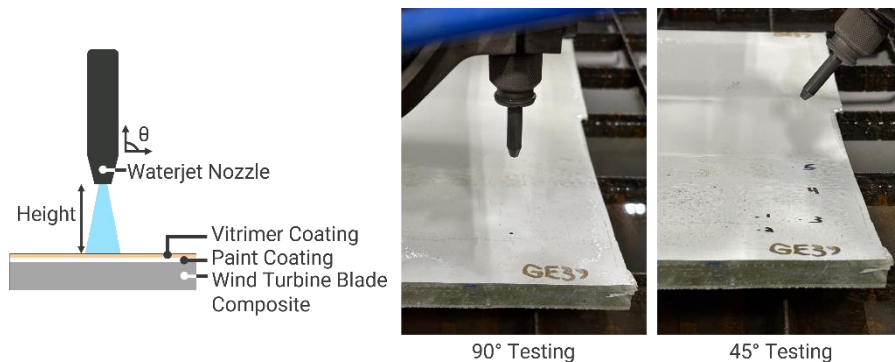


Figure 3. Schematic of the water jet setup used to simulate rain erosion testing.

erosion testing through off-site facilities (*e.g.*, UDRI) were not explored. Instead, a new protocol was developed herein to explore the use of a water jet machine as a facile method to mimic rain erosion testing. The varied parameters included exposure time (1-7 min), water pressure (500-1,000 psi), height of rain exposure (2-6 inches), and angle of rain exposure (0° & 45°) as shown in **Figure 3**.

Key Findings: The neat vitrimer resin was found to exhibit a tensile strength of 43.4 MPa, where the GE37 and Clipper blades were found to exhibit tensile strengths of 62.0 MPa and 294.2 MPa, respectively (**Figure 4**). The lower tensile properties of the vitrimer resin are not of concern, as the underlying glass fiber composite is designed to be the primary load bearing component of the laminate structure. Furthermore, by incorporating the vitrimer coating onto the GE37 and Clipper wind blade structures, the tensile properties were almost comparable to neat blade with a demonstrated 3.3% and 1.9% increase, respectively which is not found to be statistically significant based on a 95% confidence level. This lack of impact to the tensile strength is expected as the coating should not impact the primary load bearing capabilities of the underlying glass fiber composite. Regarding the Young's modulus, by incorporating the vitrimer coating onto the GE37 and Clipper wind blade structures, the stiffness exhibited a 17.2% and a 20.2% decrease, respectively. In this case, the variation in stiffness was not found to be statistically significant for the GE37 laminates but was significant for the Clipper laminates based on a 95% confidence level. This decrease likely has to do with the fact that the less stiff vitrimer coating was incorporated into the modulus calculation as part of the overall sample cross-sectional area, while it does not contribute to the load bearing of the material. All samples were found to have extremely low strain at break values, indicative of the highly brittle nature of the laminates.

The significantly higher tensile properties of the Clipper blade in comparison to the GE37 blade likely has to do with a slightly thicker blade structure being tested, where a larger portion of the sample was glass fiber composite. In this case, the absolute value of the blade tensile properties was not of primary interest, rather the change in tensile performance with coating addition was being explored.

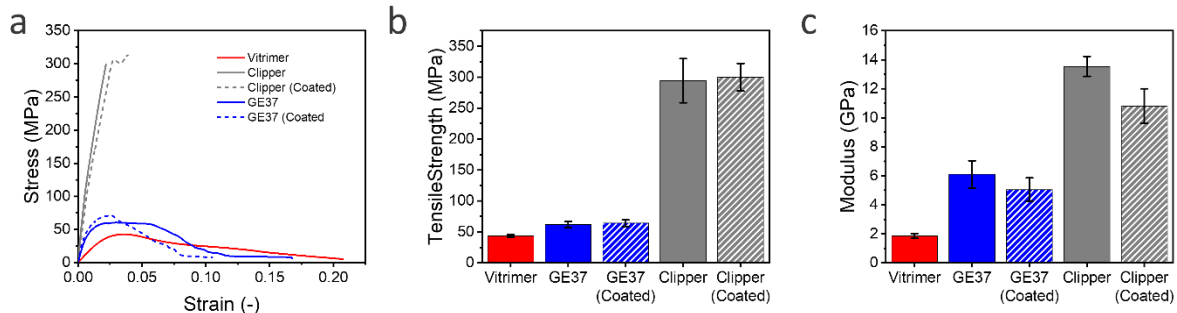


Figure 4. Tensile properties of wind turbine blade samples with and without vitrimer coatings.

While the tensile properties were of interest, it was also notable to look at the post-break samples to determine the robustness of the vitrimer coating and its adhesion to the tested samples. It is clear from **Figure 5** (white arrows) that the primary mode of tensile failure for all laminates was the breakage of the glass fiber composite. In the case of both the GE37 and Clipper laminates, the samples experienced a brittle fracture where the vitrimer coating do not show any visible signs of delamination despite tensile failure (**Figure 5**, red arrows). It is also of interest that the vitrimer samples show visible signs of necking and ductile failure, as expected for these materials and demonstrated in the tensile graph (**Figure 4a**).

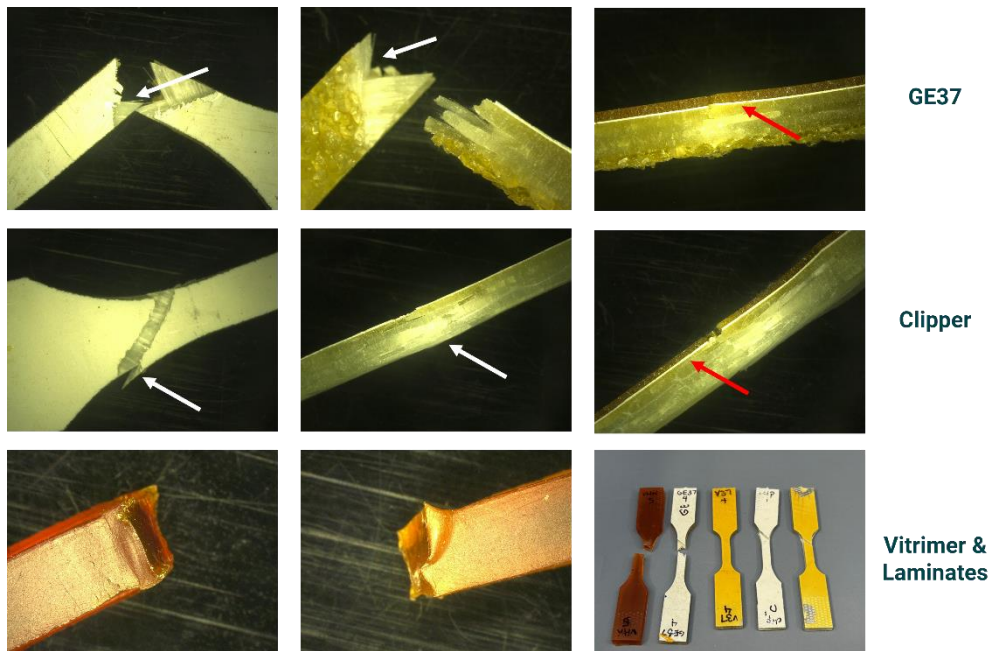


Figure 5. Images of the tensile fractured wind turbine blade samples with and without vitrimer coatings where glass fiber fracture is indicated with white arrows and lack of vitrimer delamination is indicated with red arrows.

It is expected that wind turbine blades will likely see a variety of stresses throughout their lifetime and while in use; for example, it is conceivable that the blades will experience tensile, compressive, flexural, and torsional loads while rotating. To better understand the robustness of these laminates beyond tensile properties, flexural testing was performed on both the blades and vitrimer-coated blades as seen in **Figure 6**. Similar to the tensile results, there was no statistically significant variation in the flexural modulus for the GE37 samples, with moderate decreases for the Clipper samples while both blades experienced variations in flexural strength. For example, the blades experienced a 39.1% increase and 39.7% decrease

in flexural strength when the vitrimer coating was applied for the GE37 and Clipper blades, respectively. The blades also experienced an insignificant 10.1% increase and significant 33.6% decrease in flexural modulus when the vitrimer coating was applied for the GE37 and Clipper blades, respectively. These variations were likely due to the fact that flexural testing is highly influenced by any variations in overall sample thickness. As seen in **Figure 2**, there are some nonuniformities in the coating as it was applied by hand using a doctor blade. Of interest, is the fact that the blade flexural behavior was clearly dominated by the glass fiber composite as there was no statistically significant variation in strength or modulus for the laminates when the surface (*i.e.*, paint or vitrimer coating) was flexed under tension or compression mode (*cf.* **Figure 6c** and **6d**). This indicates that the primary feature is the flexural robustness of the underlying blade composite, rather than the relatively thin coating being applied.

In addition to flexural properties to failure, it was also of interest to explore the flexural fatigue of both laminate structures (**Figure 7**). In this case, both the wind turbine blades and the vitrimer coated blades were tested under flexural load at 5 Hz for 100,000 cycles. In both cases, the addition of the 400 μm thickness vitrimer coating decreases the storage modulus of the overall laminate; this decrease is expected given the lower mechanical performance of the vitrimer material in comparison to the underlying glass

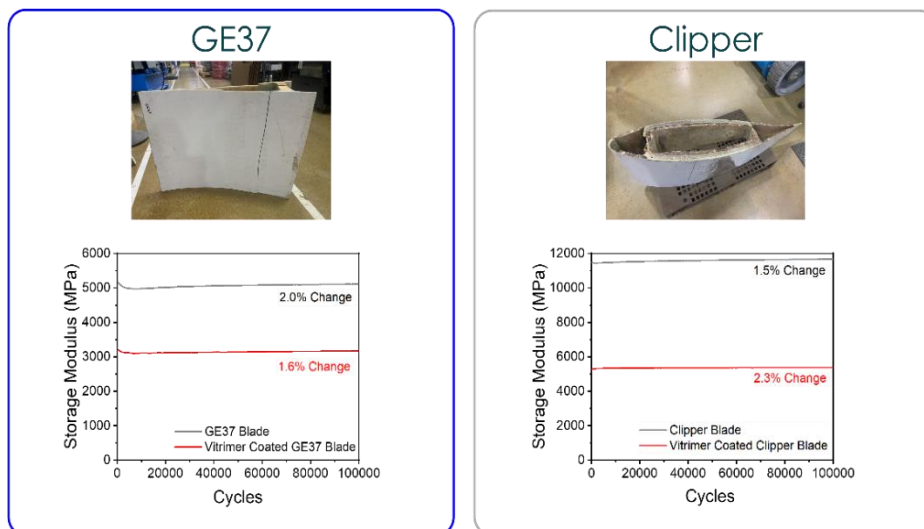


Figure 7. Flexural fatigue of the wind turbine blade samples with and without vitrimer coatings.

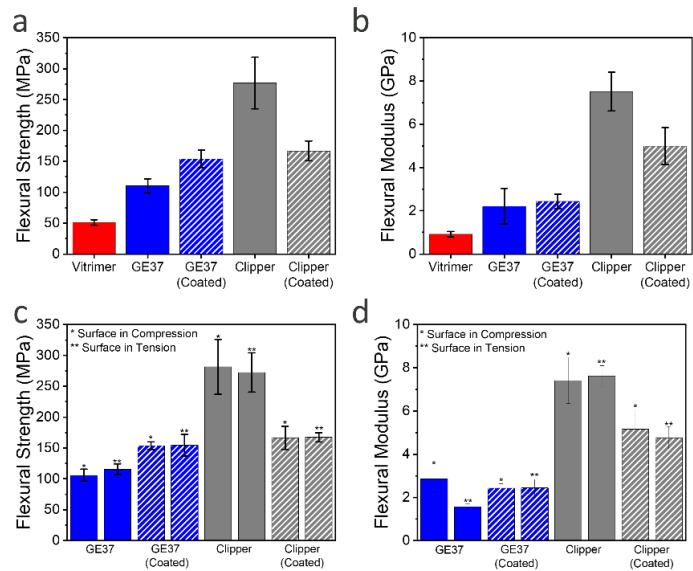


Figure 6. Average flexural strength (a) and flexural modulus (b) for all laminates. Flexural strength (c) and flexural modulus (d) for all laminates with distinction between when the coating/paint is in tension vs compression.

fiber composite and the fact that the cross-sectional area accounts for the thin coating which is not load-bearing. However, over the course of 100,000 flexural cycles, the GE37 sample and laminate both demonstrates a $\leq 2\%$ variation in storage modulus, while the Clipper sample and laminate demonstrate a slightly higher 1.5-2.3% variation in storage

modulus. The variations in storage modulus are likely due to minor cracking within the sample, which is not of critical concern given the very minimal observed drop in properties.

In addition to the mechanical properties of the laminates, the other research task of interest was the use of multiple healing mechanisms to repair vitrimer damage where an oven, boiling water, and heat gun were all explored. As demonstrated in the Level 1a VitriEdge project, a scratch was manually made on a vitrimer coating on a wind blade surface where the scratched sample was heated in an oven for 10-minutes and the scratch was manually

smoothed to demonstrate healing. This previous demonstration allowed for an 84% decrease in scratch depth. However, it is unlikely that an entire wind blade sample could, in practice, be heated to facilitate coating healing. To accommodate more “in-field” healing demonstrations that we would hope to see if this material was adopted by industry, we wanted to explore the use of hot water and a heat gun to facilitate coating healing (**Figure 8**).

When implementing water-based heating, a scratched, vitrimer coated wind blade laminate was placed into a container of boiling water for 1-minute and 5-minutes prior to manual smoothing where they exhibited a 79% and 91% decrease in scratch depth, respectively. It is likely that allowing the sample to remain in the heated water for longer periods of time allowed for temperature equilibration; however, the sample heated for only 1-minute still exhibited an impressive 79% decrease in scratch depth with an 80% faster healing demonstration. Finally, it is worth noting that we postulate there would be minimal difference between either pouring the hot water over the sample surface (closer to in-field operation) as compared to submerging the sample in the hot water. The samples were submerged in water due to safety concerns.

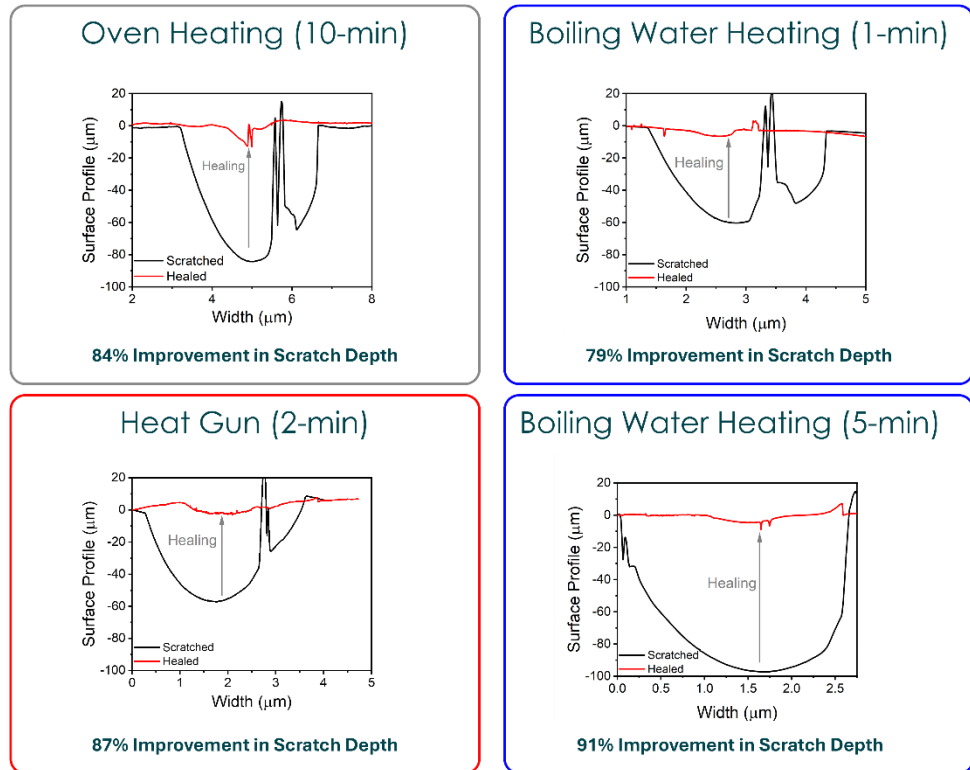


Figure 8. Images of the wind turbine blade samples with and without vitrimer coatings. The black line signifies the surface profile of the scratched sample while the red line signifies the surface profile of the healed sample.

When implementing a heat gun, the scratched sample was placed about 10-15 cm away from the nozzle of the heat gun which was set to a rating of 5.5 out of 12. The sample heated from room temperature ($\sim 25^{\circ}\text{C}$) to $\sim 100^{\circ}\text{C}$ in approximately 2 minutes prior to being manually smoothed. In this case, the healing demonstration allowed for an impressive 87% decrease in the scratch depth. It should be noted that regardless of healing mechanism it is unlikely to ever achieve a true 100% decrease in scratch depth due to the expected surface scarring that is likely to occur. The ability to heal these coatings in a matter of minutes with simplistic methods (*e.g.*, water and heated air) represents a significant step towards industrial adoption.

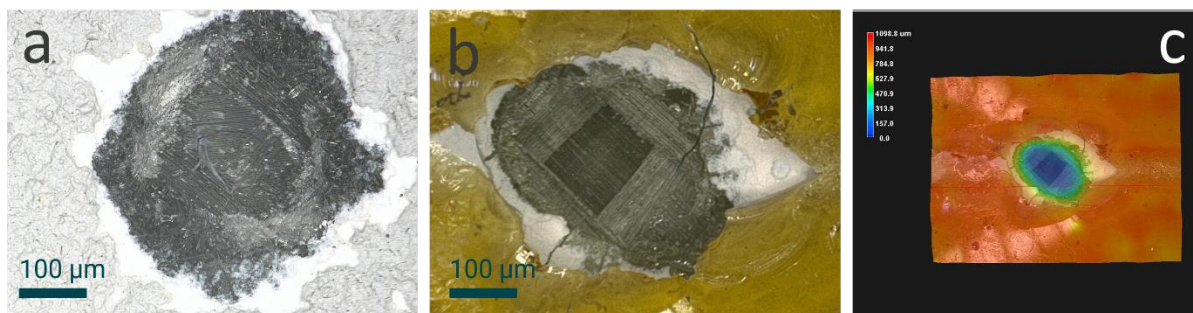


Figure 9. Optical microscopy for rain erosion testing on a neat wind blade surface (a) and a vitrimer-coated wind blade surface (b). (c) A heat map shows the depth profile of a representative damage well.

The final task for this VitriEdge Level 1b project was to explore the durability of the vitrimer coatings under a simulated rain erosion test. Due to the high cost and low availability of traditional rain erosion testing at external facilities, the team developed a novel protocol to explore rain erosion testing using a water jet machine, as depicted in **Figure 3**. In this case, four parameters (time of exposure, water pressure, angle of exposure, and height of exposure) were varied for both neat wind blade surfaces with manufacturer's paint exposed and vitrimer-coated blade surfaces with the vitrimer coating exposed. Examples of the damage inflicted during testing can be seen in **Figure 9a** and **9b** for blades and vitrimer-coated blades, respectively; in addition, a heat map seen in **Figure 9c** demonstrates the height of the damage experienced by the samples. The damage initially shows pitting as seen in the grey paint damage of **Figure 9a** and then continues to erode the surface away in the form of composite depth penetration, as seen by the black areas of fiber damage. It was important to first understand the damage to the neat wind blades as a function of these testing parameters prior to determining variations seen for the vitrimer-coated samples. For this set of tests, the GE37 blades were used for all testing to maintain consistency.

To quantify the damage, both well depth and well diameter of the damaged area was measured via optical microscopy with composite images taken by compiling micrographs at a range of depths and laser-based surface profilometry techniques. The results for both the blades (grey) and vitrimer-coated blades (red) can be seen in **Figure 10**. It was initially postulated that increasing the exposure time would increase both well depth and diameter, increasing the water pressure would increase the well depth, and angle of exposure would decrease the well depth while increasing the well diameter. As can be seen in **Figure 10**, increasing exposure time increased the damage well depth for the vitrimer-coated blade but no statistically significant change was observed for any other tests. This indicates that most of the damage inflicted on the blade was done within the first minute of exposure; interestingly, the fact that damage continues to occur with time for the vitrimer-coated blade indicates some level of increased resistance

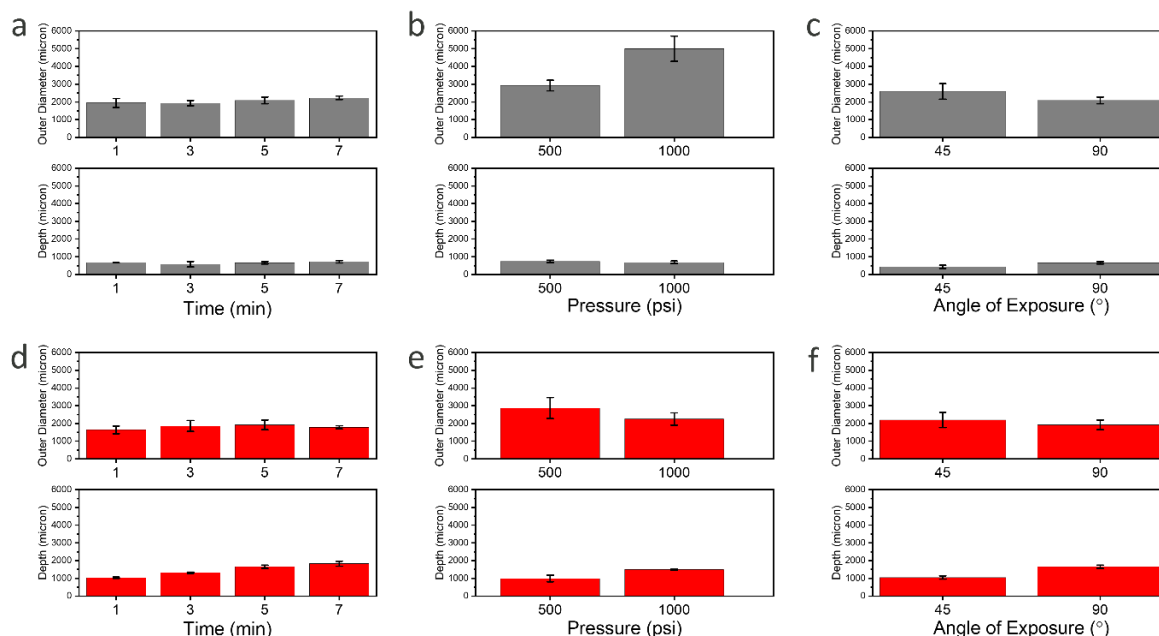


Figure 10. Outer diameter and well depth are shown for neat wind blades (a-c) and vitrimer-coated wind blade surfaces (d-f) for variations in time, pressure, and angle of exposure.

with the coating addition. Increasing water pressure during exposure resulted in an increase in the damage well depth for the vitrimer-coated blade and an increase in the damage well diameter for the neat blade surface. The inconsistency in the variations with water pressure are likely attributed to the water jet machine itself. Essentially, this machine is designed to operate around a 60,000 psi rating which is an order of magnitude greater than the highest water pressure value tested here. It is worth noting that the accuracy of the pressure reading tested here indicates a somewhat unreliable pressure being employed. The angle of water exposure was found to be the most significant parameter varied throughout these tests. Herein, it was found that increasing the angle of exposure resulted in no change in the damage well outer diameter and an increase in the damage well depth for all cases. It is unlikely that rain would impact the surface of a wind turbine blade at a perfect right angle; the fact that the damage is minimized when the angle of impact is $< 90^\circ$ is promising for this technology to move forward. The height of exposure was found to exhibit no changes in the damage response and was not reported herein.

Finally, a vitrimer composite coating was applied to the surface of the wind turbine blades for rain erosion testing where the damage well depth and diameter were measured, as seen in **Figure 11**. The vitrimer composite contained 0.5 wt% mica filler. It was found that both coatings performed comparably in terms of mitigating the damage well diameter, while the composite outperformed the neat vitrimer coating in terms of the damage well depth. This is likely due to the increase in toughness for vitrimer composites compared to their neat vitrimer counterparts and warrants further study.

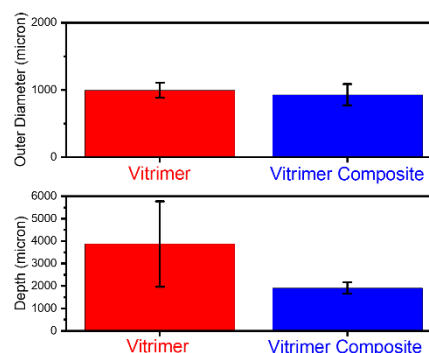


Figure 11. Variations in well diameter and depth are shown comparing a 200 μm thick vitrimer coating (red) and vitrimer composite coating (blue).

Final Product: The final project deliverables between the Level 1a and Level 1b VitriEdge project include a final report for both phases, a filed provisional patent application (Record ID 81958565: *Healable Coatings to Promote Repair of Leading Edge Erosion in Wind Energy*), and a peer reviewed journal publication currently under review at the journal *Wind Energy* which was submitted in July 2024.

Challenges Encountered & Lessons Learned: A number of challenges were encountered and overcome throughout the VitriEdge Level 1b incubator project, where the majority of these challenges were centered around the ability to simulate rain erosion testing for the vitrimer-coated wind turbine blades. For example, access to external rain erosion testing facilities is highly limited in the U.S. with issues around either gaining timely access to testing (*i.e.*, several month wait times) or due to high testing costs (*i.e.*, several thousand USD for a small number of tests). In addition, the ability to test at low pressures on the water jet was difficult as the test parameters implemented pressures 1-2 orders of magnitude below its normal operating pressure level. Finally, the ability to quantify the damage required the use of an automatic microscope with surface profilometer capabilities which was facilitated by ORNL's Center for Nanophase Materials Science (CNMS) and was prone to human error; for example, deciding where on the micrograph to count at the outer diameter was subjective when measuring damage that was not perfectly circular in nature.

A number of lessons were learned as well throughout this Level 1b project with indications for future research directions moving forward. For example, the promising results for increased durability of vitrimer composite coatings shown in **Figure 11** indicate that further study is required to explore composites as a means to increase overall coating toughness. It is also worth noting that a key feature of this project was to explore vitrimer use in wind energy for strategic applications, where their higher cost compared to traditional thermosets could be financially rationalized. While vitrimers are showing great promise as wind blade coatings, it is worth considering them in other strategic material applications for wind energy such as reversible adhesives. By manufacturing wind turbine blades with reversible adhesives, there is great promise for blade repair, blade repurposing, or remanufacturing in the future.