C. Structural Reliability of Lightweight Glazing Alternatives

Principal Investigator: Moe Khaleel
Pacific Northwest National Laboratory
P.O. Box 999/K7-36
Richland, WA 99352
(509) 375-2438; fax: (509) 375-6605; e-mail: moe.khaleel@pnl.gov

PPG Project Manager: Jim Valimont
(412) 820-8731; fax: (412) 820-8630; e-mail: jvalimont@ppg.com

Technology Development Manager: Joseph Carpenter
(202) 586-1022; fax: (202) 586-1600; e-mail: joseph.carpenter@ee.doe.gov
Field Technical Manager: Philip S. Sklad
(865) 574-5069; fax: (865) 576-4963; e-mail: skladps@ornl.gov

---

Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objective

- Optimize glazing systems for cars of the future by decreasing sound transmission while maintaining structural rigidity.
  - Reduce sound transmitted through sidelights by 6 decibel (db) by determining the effect of sidelight shape on sound transmittance, investigating sound-dampening materials for operating sidelight glazing, and quantifying the influence of alternate materials (e.g., laminated glass, bi-layer glass) on sound attenuation.
  - Maintain the level of structural integrity while reducing glass thickness by validating the structural rigidity model for various types of resins used in fixed glazing systems and by expanding/combining existing models to test various alternative constructions and glazing systems for side, rear, and roof windows.
- Reduce vehicle weight through alternate or thinner vision panels and/or by reducing the heat load transmitted through the glass. The goal is to improve the fuel economy of a car by requiring 30% less glass weight.
- Reduce side, rear, and (potentially) roof window glass weight while maintaining acoustics and minimizing price increases.
  - Consider several material options, including laminated glass, cast-in-place, pseudo-laminated glass and bi-layer materials.
  - Enhance fuel economy from reduced solar-energy transmission.
  - Reduce solar-energy transmission through glass by using absorbing substrates and reflective coatings.
  - Quantify gas-mileage improvement opportunities from improvement in solar-energy transmission.

Accomplishments

- Completed fabrication of newly-designed tooling for complex automotive side window shapes and laminates with new interlayer materials.
- Demonstrated feasibility for scaled-up production using resin injection method for large automotive side window laminations.
- Demonstrated strength requirements for 252 g ball impact from 9.14 m.
- Fabricated 30 test windows with new injection method for humidity and ball-drop testing of interlayer material.
- Reduced humidity delamination.

- Conducted initial investigation into surface treatments and edge sealing.
- Demonstrated feasibility of nanocomposites.
- Completed the investigation of edges sealants for further reduction of humidity affects.
- Lamination of large, curved, glass side windows.
- Lamination of soft and hard inclusions in interlayer mix for measuring sound transmission loss differences.
- Correlated modeling efforts to the experimental results from the soft- and hard-inclusion test results.

Future Direction

- Investigate the use of nanoparticles for mechanical property enhancements.
- Investigate non-symmetric laminates for weight reduction.
- Investigate multilayer interlayers for improved acoustical properties and weight reduction.
- Investigate ultra-thin glass layers for weight reduction.

Introduction

This project is a cooperative research and development agreement (CRADA) between the U.S. Department of Energy, Pacific Northwest National Laboratory (PNNL), Visteon Automotive Systems (Glass Division), and PPG, Inc. It started in June 2002, and is expected to be completed in 2006.

The project will evaluate designs for optimized glazing systems to be used in cars of the future and will work to help achieve the goals of FreedomCAR. The primary objective of the project is to reduce vehicle weight, improve fuel economy, and reduce vehicle emissions. However, to achieve these goals, it is necessary to consider the needs for high levels of structural reliability, competitive manufacturing costs, and passenger comfort from the standpoints of minimal acoustical noise levels and controlled interior temperatures. Energy savings will come from reducing weight by using thinner glazing; prior studies at PNNL have shown a potential for 30% reductions in weight from thinner glazing. Energy savings will also come from reducing interior heat loads that, in turn, will reduce the demand for air conditioning. The evaluation of alternative glazing concepts will also seek means to improve acoustical characteristics that will minimize interior noise levels while maintaining glazing at minimal thickness and weight levels.

Lightweight Window Manufacturing

New lightweight window samples were fabricated using 2.3-mm glass plies (a conventional automotive glass ply has a thickness of between 2.4 and 2.6 mm. The new lightweight samples were formed and laminated at PNNL using the newly developed resin injection technique.

New lightweight laminated side glass was fabricated using 2.3-mm plies. The window glass samples that were constructed are 2.3-mm glass, 0.76-mm polymer interlayer, and 2.3-mm glass.

During FY 2005, researchers completed the tool built for demonstration of large side windows with curved surfaces, as shown in Figure 1. The studies conducted demonstrated flow behavior, time to fill, evaluation of void formations, and visual observation for laminate distortion. The material used for the study is the current material that has

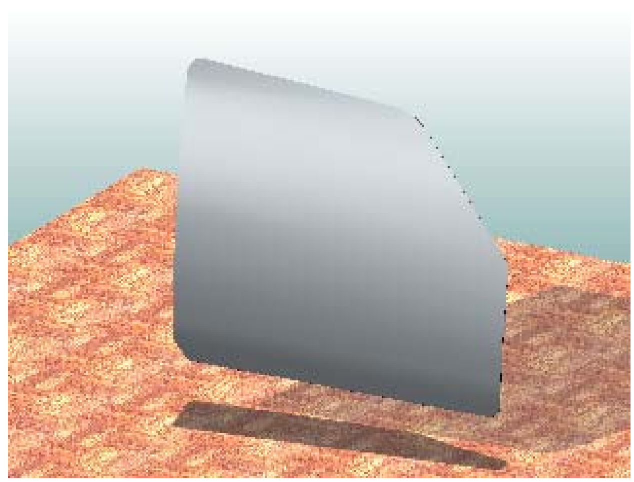


Figure 1. Typical large side window with curved surfaces.

undergone SAE Z26.1 testing with the 12-inch by 12-inch test panels.

Tool Construction

The construction material for the tool build was polymer/fiberglass composite. The matrix was a vinyl ester resin system with a foam core for increased rigidity. The master pattern was created from a current laminated, curved glass window that utilizes the polyvinylbutyral interlayer material. Prior work with this laminated glass provided the availability of thin glass for laminations.

Figure 2 shows the construction of the actual tooling. The outer gray perimeter material is the dam that constrains the resin material to the edge of the glass. The two inner o-rings are seals for vacuum chucking the glass to the mold and resin damming to keep resin out from under the glass.

The two halves are clamped together once the glass layers are chucked up to the mold surface. The resin can then be injected in from one corner and vented out at the other.

Figure 2. Test fixtures for injection process validation.

Flow Behavior and Fill Time

The large-tool-flow experimental setups were first checked for flow behavior and then to determine the time-to-fill. The flow-experiment setup had a diagonal flow length of 1.04 m (41 inches) and an area of approximately 4200 cm. The tool was tilted so there was a 2 cm elevation change from inlet to outlet. This allowed resin to flow evenly through the window and to prevent air from being trapped in the curved shape. The total fill volume was 1.7 liters of resin with 1.8-mm-thick glass laminates and the calculated volume of the window laminate for 5.3-mm-thickness is approximately 720 cm^3. The difference between the total fill volume and calculated volume would be the difference of the fill lines, inlet and outlet reservoirs, and larger separations in the two outer glass surfaces than anticipated.

The resin material had a viscosity of 500 cps and the initial flow test was done with 10 inches of head pressure. The time to fill was 30 minutes. Future testing will be accomplished with resin injection equipment and pressures as high as 1 bar.

Laminate Testing

Laminate testing has been done by both PNNL and PPG. The testing covers a wide variety of tests as shown in Table 1. Tests performed followed the ANSI/SAE Z26.1 criteria for laminated side lights. Although several chemistries have passed most of the required tests, the humidity and impact tests continue to be elusive. Furthermore, material optimization for sound transmission loss is also being investigated. PPG continues to support the project with total light transmission, UV and IR stability, boiling water, humidity, and thermal cycling.

Table 1. Tests performed on laminated glass.

ANSI/SAE Z26.1-1996	PNNL Testing	PPG Testing
1) Light Stability		Pass
2) Luminous Transmittance	Pass	Pass
3) Humidity Test	Pass Rnd 1 Fail Rnd 2	Reduced Signif. Rnd 1 Fail Rnd 2
4) Boil Test	Pass	Pass
5) Impact, Dart (9.14m)		Pass
6) Impact, Ball (9.14m)	Fail Rnd 1 Pass Rnd 2	Fail Rnd 1 No test on Rnd 2 currently
9) Temperature -40°C to 70°C	NT	NT

Humidity Testing

The elusive humidity test has been the most difficult test to pass. One formulation passed, but failed the ball-drop test. Most of the effort put forth has been toward passing this test. The delicate balance of trying to pass one test often upsets several others. However, we have gotten the entire set of tests covered with the exception of the humidity testing. The changes needed to pass the humidity test have had significant changes in the impact testing.

A measurement technique of establishing some type of standard for comparing humidity results is loosely in use. The environmental stability number (ESN) is a number that can be used to compare results from one set of results to another, Table 2. The results must be used with caution. An edge delamination with an ESN of 100 can be different from one sample to the next. For example, 100% of the perimeter could be delaminated in 1/16th of an inch that would give an ESN number of 100, but the same could be true for a small corner delamination that has a depth of ¼ of an inch. It is important to note whether the delamination is equal on all sides or whether it is spotty around the perimeter. See Figure 3 for examples of how the number is determined.

To reduce humidity failure, a couple of approaches have been investigated. Changes to resin chemistry, edge sealing, and surface treatments to the glass are a few of the approaches that have been tested.

The most significant effect was the change in resin chemistry. A 5% change in the resin ratio changed the effect humidity had on delamination by quite some amount. However, this 5% change also reduced the impact properties enough and the lamination failed impact testing. An alternative catalyst is currently being investigated for improved impact properties. The ESN number has been as low as 100. Starting numbers were typically around 2000 prior to resin modification.

Table 1. ESN criteria.

ESN=Pct 1+Pct 2x4+Pct 3x9+Pct 4x16+Pct 5x25	
Percent of perimeter at 1/16th depth	length of delamination less than 1/16th depth/ perimeter
Percent of perimeter at 1/8th depth	length of delamination less than 1/8th depth/ perimeter
Percent of perimeter at 3/16th depth	length of delamination less than 3/16th depth/ perimeter
Percent of perimeter at 1/4th depth	length of delamination less than 1/4th depth/ perimeter
Percent of perimeter greater than 1/4th depth	length of delamination less than 1/4th greater than depth/ perimeter
Highest possible ESN = 2500	

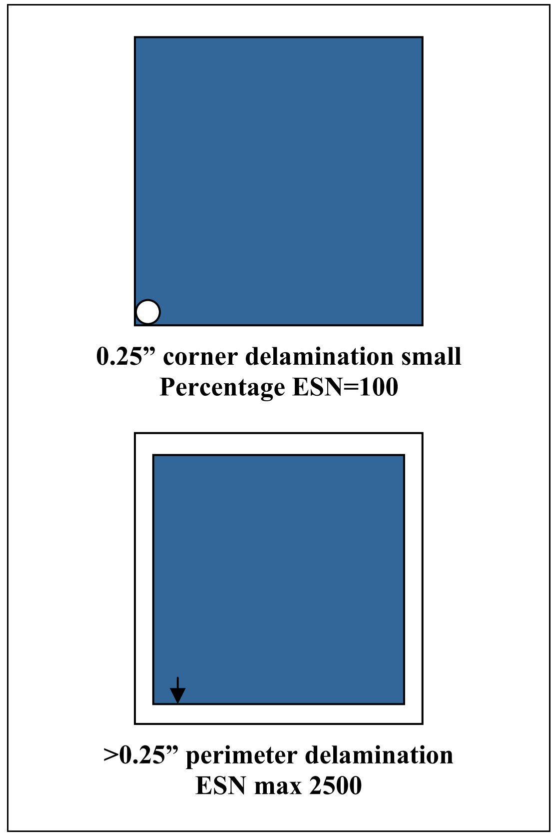


Figure 3. Measurement process for determining ESN number.

An edge-sealing approach was used with UV-curable edge sealants. The sealants were toughened methylacrylates typically used in sealing edges of heads-up display systems. Edge surfaces were prepared by cleaning with the traditional glass-cleaning process used prior to laminating the glass. The other method used was to sand the edges of the glass and then clean. The sealants were then added to the cleaned edges. The initial sealant testing had failed with high ESN numbers over 1500. A second round of testing with better cleaning of the edges and a different set of sealants to try has decreased the ESN number down to the double digits (Table 3).

Table 3. Humidity results with modifications to resin and edge sealing.

Sample	ESN
Standard Mix	2400
5% Resin change	1667
10% Resin change	52
15% Resin change	26
1% Nanoclay	467
Edge Seal #61	16
Edge Seal #68	0
Edge Seal #81	5

A different set of experiments completed the use of self-assembled monolayers. The functionality for the first set of experiments was an isocyanate surface treatment. Past experiments on thinner glass with a different catalyst yielded excellent results. However, the current system did not yield the same results and there are glass panels currently being treated with different functionality for future testing. There was an ESN of zero on past experiments with the previous catalyst and ratio change, the newer catalyst has been less successful.

The last set of experiments used nanoclay materials that serve as a vapor barrier. The ESN number in the I.28E nanoclay resulted in a 467 ESN number while the other less compatible nanoclay I.30E had an ESN of 1152.

Impact Testing

The impact testing for sidelites requires the resistance of a 252 g (0.5 lb) ball passing through or breaking the laminate into large pieces. The laminate must hold together and resist the ball passing through the laminate. There has been great success with this test. However, when optimization for the humidity tests began, the drop test showed the sensitivity of the elongation and toughness.

Figure 4 illustrates how the laminates resist the ball impact. The panel must resist fracturing into several large pieces and restrict the ball from passing through. All of the current panels have passed this test with the standard resin mix. Resin mixes that have been altered to pass the humidity test begin to fail at 5% resin change.

Figure 4. Impact-tested plate with 252 g ball at 9.14 m.

Alternative Laminates for Testing

In Figure 5 there are three different cases of experiments that were performed for process and materials evaluation. The fourth case for comparison is a monolithic glass case.

Case 1 is the baseline comparison of an unfilled resin system. It had several different resin ratios, catalysts, different polymeric materials and cure schedules. Case 1 is what has been reported in past reports and most of this report.

Case 2 is the baseline resin system that is filled with particulates of both hard and soft inclusions of nano-sized materials. There were found to be some definite advantages with these inclusions for the optical transmission and the previously discussed humidity testing.

Case 3 is a multilayer laminate that was processed. These can be made using several different

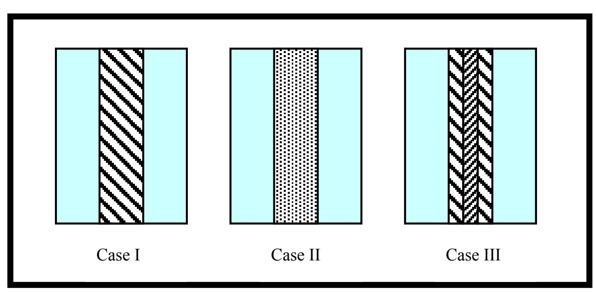


Figure 5. Illustrations of different case studies for alternative laminate possibilities.

combinations of resin blends, filled materials, and bonding agents. These were looked at for the sound transmission as well as impact and humidity tests.

Nanoparticulate Filler Experiments

The technical motivation for case 2 is to leverage the PNNL compounding and resin injection molding of materials for lightweight glazing for automotive side glass and to integrate these advanced composites technologies with surface chemistries for controlling sound, light transmission, and humidity resistance. The approach utilizes the recently-developed polymer system coupled with newly-developed nano-scale particles. This is a further enhancement of the processing and compounding of nano-scale particles for increasing the thermal stability, hardness, elastic modulus, and tensile strength through proper dispersion techniques and unique surface chemistries. An increase in mechanical properties will provide alternative design parameters and unique sound and light filtering. Electrical properties such as conductivity on certain particulates systems could also be utilized that could possibly be used for conducting interlayers for defrosting and other desirable uses.

The inclusion of organic fillers and polymers for commercial applications has been used for many years and are primarily aimed at cost reduction and stiffness improvement of the polymer composite system. A typical loading of greater than 20% by weight of micron-sized particles is generally required to bring realization of the above-stated positive effects. Polymer matrix properties such as processability, appearance, density and aging performance are compromised in systems with particle loadings greater than 20%. Therefore, composites with improved performance and low particle contents are highly desired. Tailored, nano-scale, particle-filled-polymer composites would enable new opportunities within the automotive glazing market for lightweighting of automotive side glass and, furthermore, increase service efficiencies and long-term reliability.

Polymer nanocomposites have drawn considerable attention during the last decade. The most typical fabrication method for polymer nanocomposites is the sol-gel method and it appears to be quite promising. Nanoparticulate are initially dispersed and subsequently mixed with the polymer gel at the molecular or near-molecular level.

The research effort included the following. (1) Evaluation of surface modifications of nanoparticulates for resin compatibility and control of desired properties, Figure 6; (2) Evaluation of soft and hard inclusions as well as plates vs. spheres and their effect on acoustical and optical properties. Figure 7 illustrates the difference between the neat resin and the platelet filled resin; (3) Development of mixing procedures for particulate materials with new resins through a static or ultrasonic mixer and injecting into a usable form for testing; (4) Thermal analysis cross-linking changes and microscopy of mixtures for dispersion; (5) Fabrication of test samples; (6) Testing for mechanical, physical and optical property characterization.

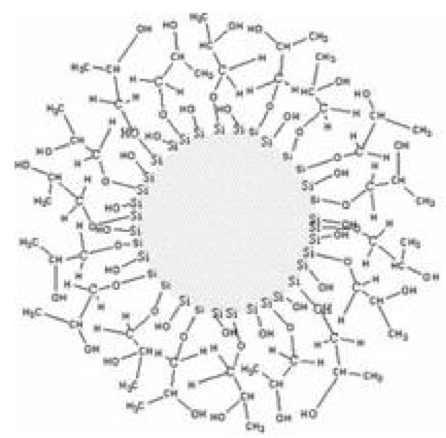
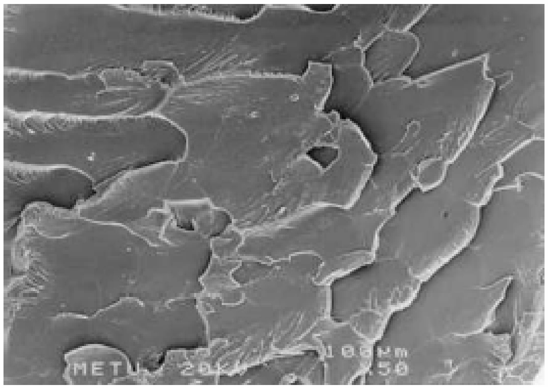


Figure 6. Modified surface of precipitated silica. (http://www.hubermaterials.com)

UV vis data were taken for different concentrations of filler and with different mixing methods. There were also two different surface treatments for testing of compatibility with the filler and resin.

Figure 8 illustrates the effects on transmittance and concentration of particles. With a 1% concentration of particles, one can have a small change on the visible spectrum and modest change to the UV and IR spectrums. The change to the IR spectrum can have a great effect on the heat loading on the interior of a vehicle.

Case three has great potential for using a wide variety of different modulus materials that could be used for sound trapping. This construction also lends



SEM image of neat epoxy

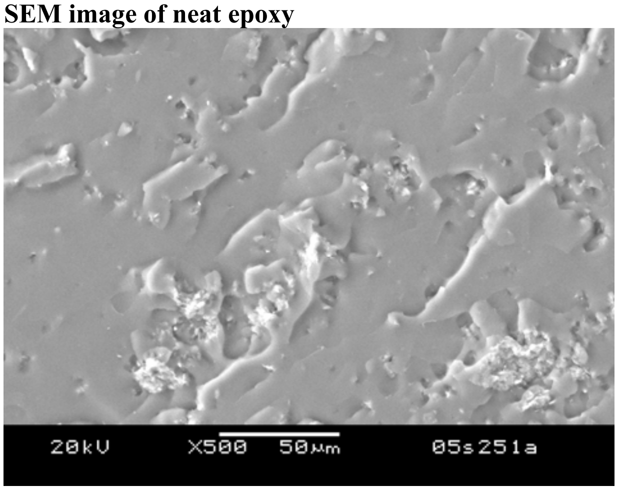


Figure 7. SEM image of cured 1% Nanocor 1.30/proprietary epoxy blend nanocomposite.

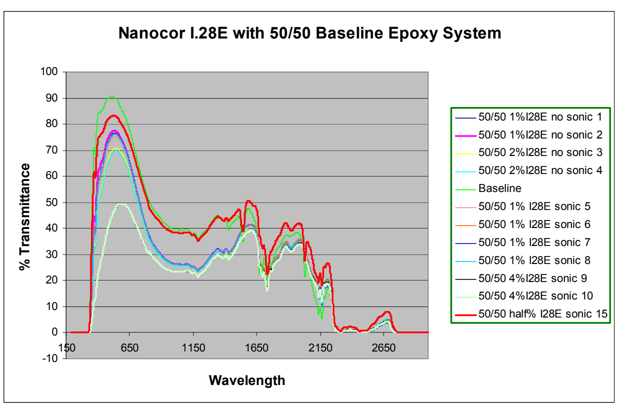


Figure 8. Full Spectrum of different concentrations of nano-particulates and the effect on the spectrum.

itself to alternative design options and adhesion alternatives. Humidity testing did not pass on the initial testing; however, the original goal was to see if it was possible to produce a multilayer laminate.

Sound Measurements and Modeling

Measurements of noise reduction (in decibels) of samples of laminated glass have been made in accordance to SAE J1400, in third-octave bands between 125 Hz and 8 kHz. The samples were mounted between the reverberant chamber and the semi-anechoic chamber. The measured sound transmission losses (STL) were compared with finite-element-determined STL predictions as well as the approximations using elastic-wave-propagation theory through laminated media. Optimized material and geometric parameters for sound insulation were studied using a factorial design method.

Future Work

Future work will focus on laminations of soft and hard inclusions as well as multilayer inter-layers for acoustics and possibly humidity reduction. Specimens will be fabricated for acoustics and material properties testing.

Modeling will be validated from the experimental data on the hard and soft inclusions in the interlayer. Efforts in modeling the sound performance of side lights through optimal multi-layer materials and dispersed particles will continue.