Laser Interference-based Surface Treatments of Carbon Fiber Polymer Composites and Aluminum for Enhanced Bonding

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Global Automotive Lightweight Materials
Detroit, MI
August 23-25, 2016
Collaborators

- Claus Daniel (optical setup, facility installation, laser-interference)
- C. David Warren (adhesive bonding, laser ablation of carbon fiber polymer composites),
- Jian Chen (optical setup),
- Don Ermand (LabView, Mechanical testing),
- John Henry (profilometry),
- Harry Meyer III (XPS – surface chemistry),
- Clayton M. Greer; Alexandra Hackett, Curtis Frederick (Optical micrographs, 3-12 months assignments UT-Knoxville).

Tim Skszek (Cosma Eng.), Mary M. Caruso (3M), and James Staagaard (Plasan)
Vehicles Are Multi-Materials Assemblies

Multi-Material Systems

• Future vehicles will increase the use of mixed material systems to deliver lightweighting solutions needed to maximize vehicle performance and efficiency (p. 6, Materials Technical Team Roadmap)

Lightweighting, Joining and Assembly.

• High-volume, high-yield joining technologies for lightweight and dissimilar materials needs further improvement. 2.5-3*

• Joining methods must be rapid, affordable, repeatable, and reliable and must provide at least the level of safety that currently exists in production automobiles. 2.5-4*

Successful Al-CF joining will enable an increase in multi-material use in automotive and consequently lead to significant weight reduction.

* VT Program, Multi-Year Program Plan 2011-2015, Dec 2010, pp. 1.0-2, 2.5-3, 2.5-4.
High-Volume, High-Yield Technologies Are Needed for Joining Dissimilar Materials

Current surface preparation for adhesive joining technologies are:

• Labor intensive
• Operator dependent:
  Surface preparation via grit blasting or sanding
• Low-volume
• Ecological expensive
  (Solvent Cleaning)

Lasers offer a possibility for adhesive joining: mature technology for surface cleaning and surface preparation

http://www.lasercleanall.com/
Traditional one-laser-beam rastering induces structures limited to the beam size.

<table>
<thead>
<tr>
<th>Method</th>
<th>Traditional laser</th>
</tr>
</thead>
<tbody>
<tr>
<td>Structure</td>
<td>1 dimple per spot, or 1 feature per line</td>
</tr>
<tr>
<td>Sizes</td>
<td>0.5-2 mm diameter, 10 micron depth</td>
</tr>
</tbody>
</table>

Large beam size:  
- **High**-productivity,  
- **Low**-concentration of laser-induced structures

Small beam size:  
- **Low**-productivity,  
- **High**-concentration of laser-induced structures
Interference technique

Interference of multiple wave yields alternating high & low energy variation on subscale beam size:
- Constructive interference of intensifies power,
- Destructive interference minimizes power,

\[ d = \frac{\lambda}{2 \sin(\alpha / 2)} \]

- Light in both beams must be temporally and spatially coherent in the region where interference fringes are to be obtained.
- Polarization properties of the two beams must be compatible.
- The irradiances of the two beams must be close in magnitude.

Schematic from Ekinci (SPIE) 10.1117/2.1201306.004958
Paradigm shift: more structuring at the same productivity

<table>
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<tr>
<th>Method</th>
<th>Traditional laser</th>
<th>Laser-interference</th>
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<tbody>
<tr>
<td>Structure</td>
<td>1 dimple per spot</td>
<td>200-20,000 pits (wells) per spot</td>
</tr>
<tr>
<td>Sizes</td>
<td>500µm dia, 10 µm depth</td>
<td>1µm dia, &lt;500 nm depth</td>
</tr>
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</table>

Features of the structured surface morphology:
- Undulation spacing: 0.5 – 50 µm,
- Density: 200-20,000/cm,
- Feature size: 1- 500nm,
- Structured area: 0.27cm²/shot,
- Velocity: 10,000 lines at a time, 79 million dots at a time, up to 162 cm²/min.
Laser interference patterns for polished steel

Unprocessed

Laser processed

Vertical lines indicate “ridges and valleys” produced by the two-beam laser-interference
Actual setup of the laser-interference processing

- Q-switched Nd:YAG laser system with an harmonic generator enabling the selection of one very sharp wavelengths of 1064, 532, 355, or 266nm.
- Pulse duration 10ns (heating and cooling rates above $10^{12}$K/s, frequency = 10Hz)
What is laser interference good for?
Pin-on-disk tribometer tests showed an increase in the oil film lifetime of 14X compared to that of an unpatterned reference.

Pin-on-disk tribometer tests showed an increase in the oil film lifetime of 130X compared to that of an unpatterned reference

Typical surface profiles obtained with laser-interference for lubrication applications

Friction coefficient was decreased by using laser-interference structured surfaces by 4.7X (lubricated) and 2X (dry friction).

Laser interference as a surface treatment for joining
## Surface preparation procedures

<table>
<thead>
<tr>
<th>Joint Type</th>
<th>Preparation of CFPC</th>
<th>Preparation of Al</th>
<th>Open time</th>
<th>Joining</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>• Abraded using Scotch-Brite™,</td>
<td>• Abraded using Scotch-Brite™,</td>
<td>None</td>
<td>Bonding right after preparation</td>
</tr>
<tr>
<td></td>
<td>• Ultrasonically cleaned using ethanol for 5 min.</td>
<td>• Ultrasonically cleaned using ethanol for 5 min.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laser structuring</td>
<td>• Laser-interference structuring</td>
<td>• Laser-interference structuring</td>
<td>3-14 days (in plastic cases)</td>
<td>*air-off with compressed air prior to bonding</td>
</tr>
<tr>
<td></td>
<td>• Stored in plastic cases</td>
<td>• Stored in plastic cases</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Prior to joining, the laser-structured samples were air-off with compressed air: (a) No ethanol cleaning (b) No removal of the oxide layer on the Al surface.
The use of a fixture ensured consistency of the joining process

The joint geometry (overlap of 1x1 in., i.e., 25.4x25.4 mm) was controlled by appropriate fixturing (ASTM D5868):

- glass beads were used to ensure an adhesive bondline thickness of 0.01 in (0.25 mm).
- a fixturing pressure was allowed.

3M provided 3 different adhesives: Urethane, Acrylic and Epoxy.
All baseline joints failed by fracture of the adhesive layer (cohesion failure)

Baseline joints were prepared without “open time”
Al and composite specimens were prepared without involving laser structuring:
• abraded using Scotch-Brite™, and
• ultrasonically cleaned using ethanol.

Clean fracture surfaces indicate poor adhesive adherence.
Laser-interference structuring was conducted on as-received specimens, without any additional surface preparation.

**CFPC:**
- mold release
- rolling marks
- contaminants

**Al:**
- rolling channels
- microcracks
- lubricant film
After laser-ablation, the adhesive-composite interface is non-planar and fiber reinforced.
For 6mm laser spot size, the quality of carbon fiber structuring increases with number of pulses per spot.

- 2 pulses per spot
- 4 pulses per spot
- 8 pulses per spot

Pulse fluence: 1.24 J/cm²
For 6mm laser spot, the melting area reduces with decreasing number of pulses.

Surface melting indicated by:
- drop-like network pattern (12 pulses per spot)
- web-like network structure (melting) (8 pulses per spot)

Pulse fluence 1.17 J/cm²

6 pulses per spot: network patterning at a finer length-scale than LHS and RHS (more structuring, less melting)

Structuring areas increase.
The surface roughness increased from 226 nm for the as-received surface to 392 nm for the laser-interference structuring surface.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Ra [µm]</th>
<th>Rq [µm]</th>
<th>Rt [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>untreated</td>
<td>0.226</td>
<td>0.286</td>
<td>3.21</td>
</tr>
<tr>
<td>Laser treated</td>
<td>0.391</td>
<td>0.493</td>
<td>4.68</td>
</tr>
</tbody>
</table>

Optical surface profile
10 pulses/spot

Striations normal to rolling direction were produced by laser-interference structuring

λ = 355 nm, pulse fluence 1.2 J/cm²
Laser-Structured Joints are more Ductile, Indicating an Enhanced Bonding of Adhesive to Both Al and CFPC

Displacement @ maximum load is a direct measure of the energy absorbed by the joint during the shear-lap test.

Laser structured joints can absorb approx. 2.5X more energy than baseline joints. The energy absorbed during the tensile test was 26.3 and 66.8 Joules for the baseline joint and laser-structured joint, respectively.

Data obtained for adhesive DP810 provided by 3M.
X-ray photoelectron spectroscopy was used to investigate the Ar-ion depth profile for Al specimens in the as-received and laser-structured conditions. Laser-interference is very effective in cleaning the surface, eliminating the need for solvent cleaning.
Shear-lap strength for laser-interference structured – by rastering and spot by spot for all adhesives

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>% increase by raster</th>
<th>% increase Spot-by-spot</th>
</tr>
</thead>
<tbody>
<tr>
<td>810</td>
<td>12.7-14.8</td>
<td>16.3</td>
</tr>
<tr>
<td>460</td>
<td>12.8</td>
<td>8.2-12.8</td>
</tr>
<tr>
<td>620</td>
<td>35.3</td>
<td>25.4</td>
</tr>
</tbody>
</table>
Baseline joints: 620 adhesive
Clean fracture surfaces indicate poor adhesive adherence
Baseline = No laser structuring

Laser-structured joints: 620 adhesive
Both surfaces have residual adhesive

Baseline joints: 460 adhesive
Adhesive – Al Interface Failure

Laser-structured joints: 460 - adhesive
Top Ply of Composite Delaminated

Failure mode changed due to laser-structuring
Evaluated Double Lap Shear for Laser-interference structured samples, [0.25 mm, 6mm beam, 810 Adhesive]

Laser-structured samples showed a significant increase in ductility.

Data obtained with
- 0.25 mm bondline,
- 6mm beam size,
- 810 adhesive.

50% of the samples failed in the bulk of the aluminum away from the joint.
Corrosion Test ASTM B117 was performed by Cosma

- Bondline thickness had a minimal effect on SLS of laser-interference samples.
- Shear lap strength of thin bondline samples degraded the most.
- For laser-interference samples, the thick bondline provided superior corrosion resistance.
Publications


Technical Summary - Laser-structured joints are more ductile, indicating an enhanced bonding of adhesive to both Al and CFPC

- Laser-interference process can effectively:
  - ablate the resin (on top surface of CFPC) without excessive CF damage,
  - Structure the Al surface, increasing its roughness,
- Failure mode changed due to laser-structuring changed to “failure in the composite” from the “cohesive failure in the adhesive” for baseline joints.
- Shear lap strength, maximum load, and displacement @ max. load for joints made with laser-interference structured surfaces were increased by approximately 14.8%, 16%, and 100%, respectively over those measured for the baseline joints.
- Joints made with laser-structured surfaces can absorb approximately 150% more energy than the baseline joints.
- For laser-interference samples, the thick bondline provided superior corrosion resistance.

All results obtained without empirical, labor-intensive surface preparation methods that are incompatible with automation.
Objective met: Develop a Breakthrough Joining Technology for Joining Carbon Fiber Polymer Composites (CFPC) and Aluminum (Al) Components

Goals of laser-structured CFPC and Al:
- Elimination of empirical, labor-intensive surface preparation (sanding and inherent process variability)
- Eliminates solvent cleaning by removal of mold releases and surface contaminates
- Provides a larger, non-planar contact area – by sub-scale beam size laser-interference structuring
- Yields a fiber reinforced adhesive/composite interface by removing the resin rich surface layer
- Increases Joint ductility by 2X (Crash Energy Absorbance)

**Speed and Cost**

“To use or not to use (direct laser interference patterning), that is the question” - Lasagni et al. (2015)

<table>
<thead>
<tr>
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<th>1-st Lab setup</th>
<th>2-nd Generation</th>
<th>Roll-to-roll processing</th>
</tr>
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<tbody>
<tr>
<td>Achieved Max. fabrication speed</td>
<td>$0.1 \text{ m}^2/\text{min}$ (metals) (25 W IR system)</td>
<td>$0.70 \text{ m}^2/\text{min}$ (PC-polymer) (180 W IR-system)</td>
<td>$0.17 \text{ m}^2/\text{min}$ (TCO) (6 W UV system)</td>
</tr>
<tr>
<td>Estimated max. fabrication speed</td>
<td>$2-5 \text{ m}^2/\text{min}$ (metals) (&gt;600 W IR system)</td>
<td></td>
<td>$1-2 \text{ m}^2/\text{min}$ (TCO) (50 W UV system)</td>
</tr>
<tr>
<td>*Estimated fabrication cost</td>
<td></td>
<td>$1.31 \text{ €/m}^2$</td>
<td>$3.08 \text{ €/m}^2$</td>
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</table>

*Lasagni et al. (2015) one operator working 52 weeks, 39 hours a day with 100% overhead, equipment cost distributed in 3 years at maximum achievable fabrication speed. (Source: *Proc. SPIE* 9351, Laser-based Micro- and Nanoprocessing IX, 935115 (March 12, 2015); doi:10.1117/12.2081976).
Acknowledgments

This research was conducted at UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy for the project “Laser-Assisted Joining Process for Aluminum and Carbon Fiber Components” and has been funded by the Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Office, Lightweight Materials Program.

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