Materials Characterization Capabilities at the High Temperature Materials Laboratory and HTML User Program Success Stories

DOE 2012 Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

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Washington, DC
May 18, 2012

Sponsored by
U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Vehicle Technologies

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The HTML User Program – Objectives & Relevance

• The HTML is a DOE Designated National User Facility. The Vehicle Technologies Program funds the operation of the HTML User Program to maintain *world-class expertise and instrumentation capabilities for materials characterization* to work with industry, universities and national laboratories to address critical technical barriers to achieving the goals of DOE’s Vehicle Technologies Program.

• The HTML User Program capabilities are also utilized to support Vehicle Technologies Program projects at ORNL in the program’s technology areas of Lightweight Materials, Propulsion Materials, Energy Storage, Power Electronics & Electric Motors, Emission Controls and Solid State Energy Conversion.
# Overview

<table>
<thead>
<tr>
<th>Timeline</th>
<th>Barriers</th>
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</thead>
</table>
| Project Start Date: 1987  
Project End Date: - | HTML user projects address cost and technical barriers in most of the Vehicle Technologies Program technology areas. |

<table>
<thead>
<tr>
<th>Partners</th>
<th>Budget</th>
</tr>
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</table>
| DuringFY2011, the HTML User Program collaborated with                     | The FY2011 budget for the HTML was $5,650,250  
• 16 companies  
• 23 universities  
• 3 national laboratories  
• 63 user projects  
• 87 researchers, 54% of them first-time users  
• 581 research days.  
• Five students earned their Ph.D. degree and two earned an M.S. degree based in part on research they conducted through the HTML User Program. |  
• $555,000 for capital equipment purchases  
• $5,095,250 for the operation of user program  
Users cost-share user projects through:  
1. direct involvement with HTML staff members during the development of the user project;  
2. funding their time and travel to the HTML  
3. costs of materials provided by the user and the research performed prior to the user project;  
4. subsequent collaboration with HTML staff members to analyze and publish the results. |
### Timeline
- Project Start Date: 1987
- Project End Date: -

### Barriers
HTML user projects address cost and technical barriers in most of the Vehicle Technologies Program technology areas.

### Partners
- During **FY2012** efforts were focused on completing existing user projects.
- No new user projects were initiated in **FY2012**. However, two projects were carried out in response to urgent calls for technical assistance by two industrial partners.
- During FY2012 a long-range plan is being developed for the future of the HTML and the HTML User Program

### Budget
The **FY2012** budget for the HTML was $910,500 (received on March 13, 2012)
<table>
<thead>
<tr>
<th>Industry</th>
<th>Universities</th>
<th>National Labs</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Applied Sciences, Inc.</td>
<td>• Alfred</td>
<td>• ORNL</td>
</tr>
<tr>
<td>• ArcelorMittal Global R&amp;D</td>
<td>• Dartmouth</td>
<td>• BNL</td>
</tr>
<tr>
<td>• Capstone Turbine Corp.</td>
<td>• Kansas State</td>
<td>• Sandia Nat. Laboratories</td>
</tr>
<tr>
<td>• Caterpillar, Inc.</td>
<td>• MIT</td>
<td></td>
</tr>
<tr>
<td>• Corning Incorporated</td>
<td>• Michigan State</td>
<td></td>
</tr>
<tr>
<td>• Cummins, Inc.</td>
<td>• Mississippi State</td>
<td></td>
</tr>
<tr>
<td>• Electron Energy Corp.</td>
<td>• New Jersey Tech</td>
<td></td>
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<tr>
<td>• Fireline TCON, Inc.</td>
<td>• North Carolina St</td>
<td></td>
</tr>
<tr>
<td>• General Motors R&amp;D</td>
<td>• No. Carolina A&amp;T</td>
<td></td>
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<tr>
<td>• Honeywell Turbo Tech.</td>
<td>• Northwestern</td>
<td></td>
</tr>
<tr>
<td>• Industrial Ceramic Solutions</td>
<td>• SUNY, Stony Brook</td>
<td></td>
</tr>
<tr>
<td>• Magnesium Elektron NA</td>
<td>• Tennessee Tech</td>
<td></td>
</tr>
<tr>
<td>• Marlow Industries, Inc.</td>
<td>• Virginia Tech</td>
<td></td>
</tr>
<tr>
<td>• Motorola, Inc.</td>
<td>• Worcester Polytechnic</td>
<td></td>
</tr>
<tr>
<td>• Toyota Research Institute</td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Turbocam</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| University of:                           | University of:                                    |                                |
|• Akron                                    | • Akron                                           |                                |
|• Dayton, Research Institute               | • Dayton, Research Institute                      |                                |
|• Florida                                  | • Florida                                         |                                |
|• Michigan                                 | • Michigan                                        |                                |
|• Missouri-St. Louis                       | • Missouri-St. Louis                              |                                |
|• South Carolina                           | • South Carolina                                  |                                |
|• Tennessee-Knoxville                      | • Tennessee-Knoxville                             |                                |
|• Utah                                     | • Tennessee-Knoxville                             |                                |
|• Virginia                                 | • Tennessee-Knoxville                             |                                |
| 2011: 16 industry, 23 university, 3 nat. lab |                                      |                                |
HTML User Program
Participating Organizations
2009-2011
Approach

The HTML is organized into 6 User Centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.

Diffraction

Materials Analysis

Mechanical Characterization

Residual Stresses

Thermography & Thermophysical Properties

Tribology Research
Approach

The HTML is organized into 6 User Centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.

The concentration of these capabilities and expertise in one location make the HTML User Program a unique national asset.
Approach: Access to the HTML

- Access to the HTML User Program is provided through a formal proposal process. Proposals are reviewed by an internal review committee and evaluated based on:
  - Technical merit
  - Relevance of the proposed research to the mission of the Vehicle Technologies Program
  - Non-competition with the private sector
  - Organizations based in the U.S.
- Research is completed within 24 months, and it involves one or more user visits to the HTML.

A user agreement (proprietary or non-proprietary) is required prior to starting a user project.
Performance Goals and Milestones

- Complete three user projects dealing with the characterization of lightweight materials, including magnesium and aluminum alloys, carbon fibers and carbon fiber-reinforced polymer matrix composites (09/11).

- Develop capabilities to perform *in situ* microstructural observations of lightweight materials, such as magnesium and aluminum alloys and complete a user project with an industrial user using these capabilities (09/11).

<table>
<thead>
<tr>
<th>Project ID</th>
<th>Organization</th>
<th>Project</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>2010-018</td>
<td>Atriax Components Inc.</td>
<td>Characterization of corrosion in heavy vehicle compressor components: Mg and Al casting and Al-MMC cylinder liner interface</td>
<td>Completed</td>
</tr>
<tr>
<td>2010-027</td>
<td>Virginia Commonwealth University</td>
<td>Characterization of lightweight materials for automotive applications</td>
<td>Completed</td>
</tr>
<tr>
<td>2010-028</td>
<td>University of Alabama-Birmingham</td>
<td>Effect of chemistry on the transformation characteristics of metastable austenite in intercritically austempered ductile iron for automotive applications</td>
<td>Completed</td>
</tr>
</tbody>
</table>
User Projects at the HTML address critical barriers to achieving the goals of DOE’s VT Program

HTML User Projects
2009-2011
Examples of User Projects

FY2011-FY2012
### User Project with Honeywell “Analysis of Residual Stresses in Turbocharger Shaft-Wheel Assembly Welds”

<table>
<thead>
<tr>
<th>Research problem</th>
<th>To characterize and quantify residual stresses in turbocharger shaft-wheel assembly weld joints.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Technical approach</td>
<td>Utilized neutron and x-ray diffraction techniques to obtain the distribution of residual stresses in the weld region.</td>
</tr>
<tr>
<td>Implications</td>
<td>Turbochargers contribute to power and fuel efficiency of internal combustion engines. Optimized manufacturing processes and component reliability are necessary for their widespread utilization.</td>
</tr>
<tr>
<td>Barriers</td>
<td>Weight and cost reductions of advanced propulsion systems</td>
</tr>
</tbody>
</table>
| Collaborators | **Honeywell Users:** Kalathur Pattabiraman and Sujoy Chaudhury  
**HTML Staff:** Cam Hubbard and Tom Watkins |

Sujoy Chaudhury mounts a test specimen at ORNL’s neutron residual stress facility.
User Project with Honeywell “Analysis of Residual Stresses in Turbocharger Shaft-Wheel Assembly Welds"

- Turbocharger wheels are attached to shafts by electron beam welding. An annealing treatment is used to relieve residual stresses, which can cause distortions and throw turbochargers out of balance.

- Residual stress maps were obtained by both neutron and X-ray diffraction around the shaft-wheel joint.
User Project with Honeywell “Analysis of Residual Stresses in Turbocharger Shaft-Wheel Assembly Welds”

• Significant changes in hoop and radial strains around the circumference of the weld.

• Hoop strain changes were also found in “stress relieved” shaft-wheel assemblies. These changes were associated with the start/stop overlap of the E-beam weld.

• As a result of this investigation, Honeywell has modified their design and manufacturing processes.
### User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers”

<table>
<thead>
<tr>
<th>Research problem</th>
<th>To understand the influence of molding processes on the structural properties of glass fiber-reinforced polymers.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Technical approach</td>
<td>Used optical and x-ray tomography to characterize glass fiber length distribution, orientation, and dispersion in polymer matrix composites. Evaluated tensile properties as a function of strain rate.</td>
</tr>
<tr>
<td>Implications</td>
<td>Understanding the influence of microstructure on material properties is critical for proper design and application of fiber-reinforced polymer to achieve the goals of DOE’s Vehicle Technologies Program.</td>
</tr>
<tr>
<td>Barriers</td>
<td>Cost. Manufacturing.</td>
</tr>
</tbody>
</table>
| Collaborators    | **Toyota User:** Umesh Ghandi  
**HTML Staff:** Vlastimil Kunc, Don Erdman |

Umesh Ghandi prepares a test specimen for high strain rate tensile testing.

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Effect of manufacturing process on fiber orientation

**Case 1:** Aligned with flow direction

**Case 2:** 45° with flow direction

**Case 3:** Transverse to flow direction

Moldflow simulation

**weak**

0.304 to 0.451

**strong**

0.598 to 0.892
User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers”
Effect of manufacturing process on fiber orientation

a) Fiber orientation

Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers

User Project with Toyota
User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers”

Effect of manufacturing process on fiber orientation

- Fiber orientation
  1. S11 stress (MPa)
  2. e11 strain (\(-\))

Random
User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers”

X-ray Radiography

Fabrication method #1

Fabrication method #2

Plates fabricated by Magna
User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers"

Orientation algorithm

Centerline coordinate

Orientation tensor

Normalized fiber orientation

CT scan and Mimics calculation were performed by Sebastian DeBoodt, Materialise
Sequence of overlapping digital microscope images is post-processed to obtain orientation for individual fibers:

• Stitching of images and image enhancement
• Fiber identification based on threshold
• Fitting of ellipse to fibers
• Generating a database of ellipse statistics

Results are averaged over a representative volume element to obtain orientation tensor.
User Project with Toyota “Influence of Molding Processes on Properties of Glass Fiber-Reinforced Polymers"

- Excellent agreement between CT scan measurements and measurements using the ellipses method.
- Autodesk® Simulation Moldflow® software was used to predict glass fiber length distribution, orientation, and dispersion in polymer matrix composites.
- The availability of robust tools for materials characterization is essential to design new or improved manufacturing methods.
Summary

• The HTML is a National User Facility that supports the missions of the Vehicle Technologies Program, by working with industry, universities and other national laboratories to develop energy-efficient technologies that will enable the U.S. to use less petroleum and reduce greenhouse gas emissions.

• The HTML User Program capabilities are also utilized to support Vehicle Technologies Program projects at ORNL in the program’s technology areas of Lightweight Materials, Propulsion Materials, Energy Storage and Thermoelectric Conversion.

• During FY2011 the HTML User Program collaborated with 42 different organizations (industry, universities, national laboratories) in the execution of 63 user projects. These projects addressed a wide range of materials technologies including lightweight materials, propulsion materials, materials for lithium-ion batteries, thermoelectric materials, catalysis, magnetic materials and materials for the manufacture of vehicular structures.
Future Work

• Funding for the HTML User Program was reduced from $5,650,250 in FY2011 to $910,500 in FY2012.

• Efforts during the rest of 2012 will be focused on completing existing user projects and on developing a long-range plan for the future of the HTML and HTML User Program.
High Temperature Materials Laboratory

Perhaps just as important as the direct support of the 21CTP is the extensive benefit to the broader research and development community that comes from the research conducted at the High Temperature Materials Laboratory (HTML). This research covers a wide range of challenging problems for which solutions require the unique instrumentation at HTML as well as the expertise of the knowledgeable DOE researchers who oversee and operate the facility. The fact that many academic researchers, as well as industry research specialists, seek collaboration with HTML speaks to the value of the facility with respect to the advancement of knowledge on many fronts.

The High Temperature Materials Laboratory, located at Oak Ridge National Laboratory, was established more than 20 years ago as a National User Facility. It was created to provide specialized, and in some cases one-of-a-kind, instruments for materials research and characterization of value not only to the 21CTP but also to other programs needing a fundamental understanding of materials properties.

Finding 3-15. The HTML continues to be a valuable resource for materials research for the 21CTP, providing specialized and in many cases unique instrumentation and professional expertise. The expertise of those who oversee the laboratory, and therefore the value of HTML to all users, is enhanced by the participation of the HTML staff themselves in the research.

Recommendation 3-10. The DOE should continue to provide 21CTP researchers and other potential users access to HTML, and it should make every effort to maintain support for HTML and to maintain the cutting-edge capability of the facility. Moreover, DOE should provide sufficient funding for HTML, and for the research specialists who oversee and operate the facility, to enable continued research collaboration with the academic community, other government laboratories, and industry. In particular, HTML support should not be reduced to a level that allows only maintenance of the equipment for paying users.