INVESTIGATION OF TITANIUM BONDED GRAPHITE FOAM COMPOSITES FOR MICRO ELECTRONIC MECHANICAL SYSTEMS (MEMS) APPLICATIONS

Paul Menchhofer and Payam Bozorgi

March 8, 2016

CRADA FINAL REPORT
NFE-15-05697

Approved for Public Release. Distribution is Unlimited.
DOCUMENT AVAILABILITY


Website http://www.osti.gov/scitech/

Reports produced before January 1, 1996, may be purchased by members of the public from the following source:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone 703-605-6000 (1-800-553-6847)
TDD 703-487-4639
Fax 703-605-6900
E-mail info@ntis.gov
Website http://www.ntis.gov/help/ordermethods.aspx

Reports are available to DOE employees, DOE contractors, Energy Technology Data Exchange representatives, and International Nuclear Information System representatives from the following source:

Office of Scientific and Technical Information
PO Box 62
Oak Ridge, TN 37831
Telephone 865-576-8401
Fax 865-576-5728
E-mail reports@osti.gov
Website http://www.osti.gov/contact.html

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
INVESTIGATION OF TITANIUM BONDED GRAPHITE FOAM COMPOSITES
FOR MICRO ELECTRONIC MECHANICAL SYSTEMS (MEMS) APPLICATIONS

Authors
Paul Menchhofer
Payam Bozorgi

Date Published:
March 8, 2016

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6283
managed by
UT-BATTELLE, LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

Approved For Public Release
## CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>CONTENTS</td>
<td>iv</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>vi</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>1</td>
</tr>
<tr>
<td>1. INVESTIGATION OF TITANIUM BONDED GRAPHITE FOAM COMPOSITES FOR MICRO ELECTRONIC MECHANICAL SYSTEMS (MEMS) APPLICATIONS</td>
<td>1</td>
</tr>
<tr>
<td>1.1 BACKGROUND</td>
<td>1</td>
</tr>
<tr>
<td>1.2 TECHNICAL RESULTS</td>
<td>1</td>
</tr>
<tr>
<td>1.3 IMPACTS</td>
<td>5</td>
</tr>
<tr>
<td>1.4 CONCLUSIONS</td>
<td>5</td>
</tr>
<tr>
<td>2. PARTNER BACKGROUND</td>
<td>7</td>
</tr>
</tbody>
</table>

---

iv
LIST OF FIGURES

Table 1 Sample Compositions........................................................................................................ 2
Table 2 Thermal conductivity and immersion densities of composite samples..................................... 2
Fig. 1. Test specimens for MEMs devices along with round Flash diffusivity sample shown............... 3
Fig. 2. Thermal Conductivity (calculated) from Laser Flash Diffusivity measurements..................... 3
Fig. 3. MEMS device concept ......................................................................................................... 4
Fig. 4. Thermal ground plane in use.................................................................................................. 4
Fig. 5. ORNL Titanium/graphite composites with AM micro-channels............................................. 4
Fig. 6. Profile shows ~160 µm line thickness (desired 10 µm)............................................................. 5
ACKNOWLEDGEMENTS

This CRADA NFE-15-05697 was conducted as a Technical Collaboration project within the Oak Ridge National Laboratory (ORNL) Manufacturing Demonstration Facility (MDF) sponsored by the US Department of Energy Advanced Manufacturing Office (CPS Agreement Number 24761). Opportunities for MDF technical collaborations are listed in the announcement “Manufacturing Demonstration Facility Technology Collaborations for US Manufacturers in Advanced Manufacturing and Materials Technologies” posted at http://web.ornl.gov/sci/manufacturing/docs/FBO-ORNL-MDF-2013-2.pdf. The goal of technical collaborations is to engage industry partners to participate in short-term, collaborative projects within the Manufacturing Demonstration Facility (MDF) to assess applicability and of new energy efficient manufacturing technologies. Research sponsored by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.
ABSTRACT

PiMEMS Inc. (Santa Barbara, CA) in collaboration with ORNL investigated the use of Titanium Bonded Graphite Foam Composites (TBGC) for thermal mitigation in Micro Electronic Mechanical Systems (MEMS) applications. Also considered were potentially new additive manufacturing routes to producing novel high surface area micro features and diverse shaped heat transfer components for numerous lightweight MEMs applications.

1. INVESTIGATION OF TITANIUM BONDED GRAPHITE FOAM COMPOSITES FOR MICRO ELECTRONIC MECHANICAL SYSTEMS (MEMS) APPLICATIONS

This phase I technical collaboration project (MDF-TC-2015-074) was begun on April 30, 2015, and was completed on December 31 2015. The collaboration partner PiMEMS Inc. is a small business.

1.1 BACKGROUND

PiMEMS is a small privately held company that designs, fabricates, and packages Micro-electronic-mechanical system (MEMS) devices using titanium.

PiMEMS Inc. in collaboration with ORNL investigated the use of Titanium Bonded Graphite Foam Composites (TBGC, ORNL IP, US Patent # 09017598) for thermal mitigation in Micro Electronic Mechanical Systems (MEMS) applications. The collaboration also investigated the potential for new additive manufacturing routes for producing high surface area micro-features on titanium substrates to improve wicking in MEMs applications.

PiMEMS Inc. has a commitment to the use of titanium for MEMs devices even though titanium has poor thermal properties. This effort combined titanium (as a binder matrix material) with high thermal conductivity (ORNL developed) graphite foam powder to yield high thermal conductivity composites to be integrated within a MEMs device. The target goal for Phase I was to reach or exceed 100 W/mK (for the XY “in-plane” orientation) for the ORNL proposed composites.

1.2 TECHNICAL RESULTS

ORNL prepared specimens of titanium bonded graphite foam powder consolidated by vacuum hot-pressing and measured the resulting thermal conductivity for the three proposed compositions. The compositions studied are outlined in Table 1. Flash diffusivity was conducted (by ORNL) on coin shaped samples 13 mm dia. x 4 mm thick. (Fig. 1.) Rule of mixtures (ROM) calculations were used to determine the specific heat for the different composites. Adjustments for the relative sample densities were also figured into the calculations. The samples were machined from vertical slabs- 4 mm thick (in order to determine the XY “in-plane” performance for the three compositions).

Rectangular samples were prepared at 40 mm x 50 mm x 1mm thick as requested by PiMEMs. Additionally, three small (10-40 mm x 1mm thick) samples were shipped to PiMEMS for evaluation and testing. (Fig. 1.)
Immersion density (Archimedes’ method) was used to determine the densities of the hot-pressed samples and for comparison to their theoretical densities. As seen in Table 2, as the graphite foam content increased, the percent of theoretical density fell. Of particular interest is a corresponding reduction in the slope of the curve for improvement in thermal conductivity with increasing graphite content (Fig. 2.), with a maximum thermal conductivity of > 173 (W/mK) being measured at room temperature for TCP-3 (in air) in the XY in-plane direction.

Thermal diffusivity ($\alpha$) was measured with the Xenon flash facility located within the Thermal Physical Properties User Center at ORNL (High Temperature Materials Laboratory, HTML). The thermal conductivity ($\kappa$) was calculated using the relationship: $\kappa = \alpha\rho C_p$. A constant value for $C_p$ of 1020 J/kgK for Koppers Inc. (P-1) foam and a value for $C_p$ of 523 J/kgK for titanium metal was used for the calculation. Further processing optimization would likely improve the final composite density for the higher graphite containing composites and achieve further improvements in the final thermal conductivities.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Vol. % Foam</th>
<th>Vol. % Ti</th>
<th>[W/mK]</th>
<th>Density</th>
<th>% TD</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCP-1</td>
<td>55.00%</td>
<td>45.00%</td>
<td>147.75</td>
<td>3.22</td>
<td>98.56%</td>
</tr>
<tr>
<td>TCP-2</td>
<td>65.00%</td>
<td>35.00%</td>
<td>172.29</td>
<td>2.94</td>
<td>96.30%</td>
</tr>
<tr>
<td>TCP-3</td>
<td>75.00%</td>
<td>25.00%</td>
<td>173.45</td>
<td>2.60</td>
<td>92.25%</td>
</tr>
<tr>
<td>TCP-4</td>
<td>85.00%</td>
<td>15.00%</td>
<td>156.27</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

Table 1 Sample Compositions

<table>
<thead>
<tr>
<th>Sample</th>
<th>Vol. % Foam</th>
<th>Vol. % Ti</th>
<th>[W/mK]</th>
<th>Density</th>
<th>% TD</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCP-1</td>
<td>55.00%</td>
<td>45.00%</td>
<td>147.75</td>
<td>3.22</td>
<td>98.56%</td>
</tr>
<tr>
<td>TCP-2</td>
<td>65.00%</td>
<td>35.00%</td>
<td>172.29</td>
<td>2.94</td>
<td>96.30%</td>
</tr>
<tr>
<td>TCP-3</td>
<td>75.00%</td>
<td>25.00%</td>
<td>173.45</td>
<td>2.60</td>
<td>92.25%</td>
</tr>
<tr>
<td>TCP-4</td>
<td>85.00%</td>
<td>15.00%</td>
<td>156.27</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

Table 2 Thermal conductivity and immersion densities of composite samples
Fig. 1. Test specimens for MEMs devices along with round flash diffusivity sample shown.

Fig. 2. Thermal Conductivity (calculated) from Laser Flash Diffusivity measurements (ORNL).

ORNL also investigated additive manufacturing (laser additive manufacturing on a Renishaw laser melting system) to produce high surface area features (micro-channels) on select specimens, which were faced with commercially pure (CP) titanium. These specimens were also evaluated by PiMEMs. Unfortunately, the print resolution and resulting surface features were too large to offer
improved wicking for their MEMs devices. (Figs. 3-6.) Profiles of printed lines show ~160 µm line thickness, which exceeds the 10 µm desired target thickness.

Fig. 3. MEMS device concept.

Fig. 4. Thermal ground plane in use.

After fusion, each substrate shows some warping and delamination. (2015-08-05, Line Spacing = ~250 µm) Lines (3 layers, ~50µm ea) on TCP substrates, 200 W power

- Ti faces delaminated from the Ti/C substrate
- Substrates show end-to-end warping

Fig. 5. ORNL Titanium/graphite composites with AM micro-channels.
Thermal conductivity performance was also evaluated by PiMEMS using their in-house technique that measured the “lateral thermal conductivity” of the hot-pressed samples. The ORNL Ti-Graphite composites demonstrated an increase in the overall lateral thermal conductivity of the samples tested by a factor of 4 compared to the control sample (CP titanium).

### 1.3 IMPACTS

The potential for integrating the proposed technology in MEMs devices has been demonstrated although further improvements are recommended by PiMEMs.

The aspect ratio (width/depth) of the fabricated micro channels and mesh made by laser additive manufacturing on titanium substrates remain too large for micro fluidic and MEMS devices. If smaller channels with smaller surface roughness features (desired <10 µm features) could be demonstrated by further processing improvements, it might then be possible to utilize and integrate the proposed techniques for the manufacture of MEMs devices.

### 1.4 CONCLUSIONS

1- The applied TBGC method shows a potential to make a Ti-Graphite composite that increases the overall lateral thermal conductivity of the sample made by factor of 4 compared to CP titanium. However, the current approach needs to fabricate even thinner Ti-Graphite composites (100–300 µm) in order to be applicable for MEMS applications/devices.

2- The aspect ratio (width/depth) of the fabricated micro channels and mesh using laser additive manufacturing on titanium substrates is too large for micro fluidic and MEMS devices. Further processing improvements would be required in order to fabricate smaller features, channels and meshes.
2. PARTNER BACKGROUND

PiMEMS is a small privately held company that designs, fabricates, and packages Micro-electronic-mechanical system (MEMS) devices using titanium.

PiMEMS Inc. in collaboration with ORNL investigated the use of Titanium Bonded Graphite Foam Composites (TBGC) for thermal mitigation in Micro Electronic Mechanical Systems (MEMS) applications. (ORNL IP, US Patent # 09017598) The collaboration also investigated the potential for new additive manufacturing routes for producing high surface area micro-features on titanium substrates to improve wicking in MEMs applications.