February 27, 1997

Wallace Smith
Office of Naval Research
Code 332 (SMITH) Room 504
North Quincy Street
Arlington, VA 22217-5660

Dear Wally:

Enclosed is our fifth six month report (1/96-6/96) on the production of Distorted 3-3 Hydrophone Composites from Reticulated Ceramics Contract #N00014-94-C-0046. This report covers contact activity during the first of our two six month no cost extensions to this program.

The most significant progress during this period was the completion of our first full sized (4"x4") conformable 3-3 composite array and its delivery to Fred Geil of Northrup Grumman for testing. The array manufacture was a joint effort between Hi-Tech and Alfred University (Matt Creedon and Walter Schulze) with the next five arrays to be produced entirely by Hi-Tech Ceramics. We are still dependent on Alfred University for their poling and testing equipment and are seeking their input on additional design refinements for the next arrays. Matt Creedon has finished up his PhD dissertation based on these 3-3 composites and has successfully defended.

We have received confirmation of our contract extension through 12/96 and expect to be finished with all the contract deliverables, six arrays and the accompanying manufacturing reports, at that time.

Sincerely,

D. Andrew Norris
Development Engineer

c. DCMO Buffalo
   Director, Naval Research Laboratory
   Defense Technical Information Center
   Walter Schulze, NYSCC at AU

enclosure
PHASE I - PERFORMANCE OPTIMIZATION

PROGRESS:

Coupon manufacturing has been exclusively 15ppi, 3:1 distorted foam processed to 15-20% density. All samples during this report period were processed and fired at HTC and fabricated into composites at Alfred University. A group of 20+ composites were made and characterized. Composite properties obtained have been reported by Matt Creedon and Walter Schulze of the New York State College of Ceramic at Alfred University during a presentation at the 1996 American Ceramic Society Convention in Indianapolis and in poster summaries by both Hi-Tech and Alfred University at the 1996 ONR Transducer Materials and Transducers Workshop at Penn State University. Table I summarizes the characteristics and properties for the 3:3 composite batch from which 16 were chosen for the array.

ACTION:

Processing of 15ppi, 3:1 PZT-5H reticulate will continue for use in array assembly. If time and resources permit, epoxy modifications may be investigated as a means of increasing the composite’s hydrostatic dielectric response. These modifications would be made based on the results of modelling done on the 3:3 structure by Matt Creedon as part of his thesis work.

PHASE II - PANEL ASSEMBLY

PROGRESS:

A specific procedure and timeline was followed after the completion of the second trial array for producing our first deliverable array which was shown at the Penn State meeting. Adjustments made from the trial array were: larger composites (25x25x4mm) and a larger urethane encapsulation mold (100x100x6mm).

Twenty five PZT reticulate with 3:1 aspect ratio were fired at Hi-Tech Ceramics in two firings. All of these reticulate were infiltrated with Spurr’s epoxy (soft recipe) in groups of up to 16. After curing, the infiltrated sheets were ground on the faces to reach a thickness of 4mm for the composites. The individual composites were then cut from the sheet and were 23x23x4mm. The face grinding exposed the PZT reticulate and provided contact points for silver epoxy to be painted on the composite faces. Each composite was then poled at 25kV for 5 minutes at 70 C. After poling, a complete characterization of each composite was made. Table I summarizes these results.

The $d_{33}$ figure of merit was used as the criteria to select the 16 composites from the lot to be assembled in the array. All 16 composites had a $d_{33}$ greater than 1000. The composite interconnects and array wiring was done the same as for the trial array described in the previous six month report. Encapsulating the array in urethane with the
bottom fill mold, shown in Figure 1, produced a 108x108x6mm conformable array. Figure 2 is a photograph of the array.

This first array and the accompanying composite characterization table was reviewed with Wally Smith of ONR and Fred Geil of Northrup Grumman at the 1996 ONR Transducer Materials and Transducers Workshop at Penn State. Fred's initial comment was that maximizing voltage response is more critical in his applications than the $d_{33}$ we've used for selection. He would also like to see the array wired in two halves with longer twisted pair shielded wire and have some area made on the array for mechanical attachment during testing. Fred took the first array with him for testing and a letter was sent to Wally Smith confirming this was the first of our deliverable arrays. Air box testing results from Fred on our array are summarized in Table II. We are still awaiting the tank testing results.

ACTION ITEMS:

The next array assembled will incorporate as many of Fred's suggestions as possible based on his hydrophone experience and any testing results he obtains on the first array. One immediate change will be to increase the composite thickness from 4 to 6mm to increase the voltage output. Design changes to the encapsulation mold will incorporate this and also the "ears" desired for mechanically gripping the array.

PHASE III - MANUFACTURING ASSESSMENT

PROGRESS:

All PZT firing for the arrays is being done at Hi-Tech facilities. The technique uses 99.5 Alumina Hi-Tech fabricated setters to support the PZT reticulate and atmosphere powder on PZT tape (supplied by Morgan Matroc) during firing. This set-up is then covered with a dense 99.8 Alumina crucible to limit lead loss during firing.

ACTION ITEMS:

The manufacturing report will be written over the next several months as more composites are made and a specific assembly process developed for the deliverable arrays.
PHASE IV - PROGRAM MANAGEMENT

PROGRESS:

Group meetings between Hi-Tech Ceramics and Alfred University were held on an as needed basis to review progress and set directions. Much of the communication was on a more informal basis by telephone and laboratory visits between the two facilities by researchers. Copies of the group meeting minutes between 1/96 and 6/96 are contained in Appendix A.
<table>
<thead>
<tr>
<th>Characteristic/Property</th>
<th>Average</th>
<th>Std. Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fired Bulk Density of PZT5H Reticulate</td>
<td>1.24 g/cm³</td>
<td>(0.10)</td>
</tr>
<tr>
<td>Apparent Density of PZT5H Reticulate</td>
<td>7.57 g/cm³</td>
<td>(0.03)</td>
</tr>
<tr>
<td>Composite Density</td>
<td>2.22 g/cm³</td>
<td>(0.09)</td>
</tr>
<tr>
<td>Volume % PZT</td>
<td>15.86%</td>
<td>(1.13)</td>
</tr>
<tr>
<td>Dielectric Constant</td>
<td>224</td>
<td>(25)</td>
</tr>
<tr>
<td>Average $d_{33}$</td>
<td>196</td>
<td>(24)</td>
</tr>
<tr>
<td>Average $d_h$</td>
<td>46</td>
<td>(4.2)</td>
</tr>
<tr>
<td>Average $g_h$</td>
<td>23</td>
<td>(0.16)</td>
</tr>
<tr>
<td>Average $d_h g_h$</td>
<td>1067</td>
<td>(149)</td>
</tr>
</tbody>
</table>

Note: Total 25 pieces, approximately 25x25x4mm
Table II. Initial Results on First Deliverable Array

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sensitivity in air box</td>
<td>-201dB</td>
</tr>
<tr>
<td>Capacitance</td>
<td>3500pF</td>
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<tr>
<td>tan d</td>
<td>.02-.03</td>
</tr>
<tr>
<td>Thickness Resonance</td>
<td>235kHz</td>
</tr>
<tr>
<td>Lateral Resonance</td>
<td>32kHz</td>
</tr>
</tbody>
</table>

Note: Compared to pzr, about 10dB worse in sensitivity, about 3x better in capacitance
Figure 1. Photograph of Urethane Encapsulation Mold.

Figure 2. Photograph of Distorted 3-3 Hydrophone Composite Array made from PZT-5H Reticulated Ceramics.
APPENDIX A

Meeting minutes Jan-Jun '96
ONR Contract Meeting Minutes
1-24-96

Attendees: Matt Creedon, Andy Norris, Truett Sweeting, Walter Schulze

This meeting was held to see the mini array of 9 composites assembled by Matt and to set some short term goals to reach prior to the Penn State ONR meeting in March.

Walter Schulze began the meeting by expressing his frustration over our current state of progress since the start of the contract. He felt HTC’s efforts have been less than what had been needed to reach the original goals of the contract. The mini array assembled by Matt has a modest $d_h$ of 29 and excellent flexibility, though with additional effort and samples Walter feels that greater performance may have been achieved after these 2 years. Over riding priorities at Hi-Tech have displaced ONR contact efforts and Truett proposed some written short term goals that would help focus our efforts toward the deliverables and a positive showing at the Penn State Meeting.

A timeline has been made (see attached) for processing a full size deliverable array of 3:3 reticulate PZT/epoxy composites for display at the Penn State Meeting. Numerous tasks need to be completed and will begin immediately. The reticulate will be 15ppi, 3:1 stretched ratio foam with fired size of approx. 25 x 25 x 5mm. These will be processed and fired at HTC. Matt will direct the impregnating, electroding and assembling of the composites into the array. The final array will be 100 x 100mm containing 16, 3:3 PZT/epoxy composites.
ONR Contract Meeting Minutes
5-21-96

ATTENDEES: Matt Creedon, Andy Norris, Trucet Sweeting, Walter Schulze

This meeting was held to discuss and set some summer goals to be accomplished prior to the ISAF meeting in August and Matt’s thesis completion in September.

With Matt’s pending completion in September his priorities will be the modeling of our composite, optimizing the poling of reticulate PZT 5H, testing samples with modified impregnation epoxy and testing mini arrays with modified hinge material. The modifications will be done to alter the stiffness to possibly gain some initial sensitivity. HTC needs to supply fired 11ppi 3:1 distorted samples for these tests ASAP. Matt has 11 samples he can begin working with.

Our first array which was given to Fred Geil for tank testing at Northrup Grumman will be tested the week of 5/27. Another meeting will be held June 4th between HTC and AU to discuss the results. Our next array will have some modifications from the previous one based on discussion with Fred and his results. The initial modifications will be to wire the array in two halves instead of one with longer lead wires, cast or attach “ears” for holding the array, and increasing the thickness of the 3:3 composite from 4 to 6mm. There may be additional changes after testing is completed. This second array will be processed to be completed the first week of July. HTC will need to have the urethane mold altered to cast the thicker composite.

ACTION ITEMS:
Next meeting June 4 at 11:00am at HTC
HTC to process and supply samples to Matt for testing
HTC to process reticulate for next array and oversee its production with Matt’s assistance

CC LAURIE
DOUG