Report # N00014-94-C-0046
Data Item No. A003

The Production of Distorted 3-3 Hydrophone Composites from Reticulated Ceramics -Manufacturing Process Report

Prepared by:
Douglas Karst
Andy Norris
Truett Sweeting

Hi-Tech Ceramics
Box 788
Alfred, NY 14802

May 8, 1997

Contract # N00014-94-C-0046

Prepared for
ONR
Office of Naval Research
Washington, DC

ABSTRACT

Reticulated ceramics have been in production for about 20 years. Annually, hundreds of thousands of square feet are produced with prices ranging down to twenty cents per square inch for some materials. This work was undertaken to apply reticulate ceramic processing techniques to piezoelectric ceramic-composite production and the subsequent production, testing, and evaluation of six hydrophones arrays. This first generation composite material has resulted in an rugged distorted 3-3 piezoelectric ceramic composite with decent acoustical properties. Modeling results suggests that simple modifications result in enhanced sensitivities and acoustics. However, a tradeoff has to be made in sensitivity to maintain strength at high pressures. A manufacturing scale-up as well as suggestions for a second generation material are presented. Composite costs are projected to be under $10/square inch in moderate volumes.

CONTENTS

LIST OF FIGURES

I. SUMMARY

Hi-Tech Ceramics was sponsored by the Office of Naval Research to adapt technology developed for reticulated ceramic production to the production of piezoelectric ceramic composite material for hydrophone applications. This work was in collaboration with the New York State College of Ceramics, Matt Creedon, then Ph. D. student, and his advisor Dr. Walter A. Schulze at the New York State College of Ceramics.

Polyurethane foam was stretched at elevated temperatures and cooled in place. This foam was then cut into pieces ~ 1" x 1" x 1/4" with the stretching direction in the 1/4" direction. . After stretching, the foam was coated with piezoelectric ceramic, forming a replica of the stretched foam. A great amount of effort went into the development of the foam stretching process so as to have the proper balance between the volume percent ceramic and the optimized aspect ratio for enhanced anisotropy of the final ceramic replica. The ceramic coated foam was dried and placed in a high temperature furnace to "burn off" the foam and fire the ceramic. Then the fired ceramic foam was filled with epoxy, surface ground, cut, electroded, and poled in the 6 mm direction. Under contract, Hi-Tech was to deliver six 4"x 4" arrays. The arrays were each composed of sixteen 1" squares. The first two were 4 mm thick and the last four were made 6 mm thick as a means of increasing the sensitivity. The arrays were tested at Northurp-Grumman in Annapolis, MD.

Though the sensitivities are modest, -192 dB re 1 Volt/ micropascal, the composite material is highly robust. Both a thickness resonance and a lateral resonance is evident from the acoustic and electronic testing. Without nomalization and even with it, the nonlinearity of this material limits it to certain frequency ranges. It is felt that a high degree of consistency can be expected with scale-up and the purchase of certain items for manufacturing, as well as the implementation of certain production tolerances. Finite element modeling by Matt Creedon indicates that by dropping the elastic modulus of the epoxy and reducing the Poisson's ratio at the same time will result in much higher sensitivity with a diminished d31 and thus improved acoustics. Dr. Schulze has proposed adding microballoons to the epoxy. Hi-Tech Ceramics is anxious to produce and evaluate this second generation of reticulated piezoelectric ceramic composite. It is believed that a highly sensitive composite with linear acoustic response and output can be realized with only minor modifications.

This project concludes that reticulated ceramic processing is adaptable to hydrophone production. Moreover, the processing adapts well to low volume, custom shapes and high volume repetitive shapes. A wide range of shapes are readily fabricated with low tooling costs (plates, donuts, cylinders, etc.). The composite is rugged with properties similar to those of 1-3 composites. Tooling costs are low, allowing for fast prototyping. One limitation is a lower limit of thickness of about 2 mm, equivalent to a thickness resonance of about 1 Mhz.

II. INTRODUCTION

The following report summarizes the methods used at Hi-Tech Ceramics to utilize the production techniques of reticulated ceramics to produce piezoelectric ceramic composite material and to fabricate six prototype hydrophone arrays for the Office of Naval Research under Contract #N00014-94-C-0046. A process description is provided for the manufacture of the prototypes and ideas for scale-up are presented. Manufacturing scale-up costs assume a production volume of 1200 square feet per year. This is less than 1 % of Hi-Tech's current production rate of reticulate ceramic.

Each hydrophone array was composed of sixteen individual piezoelectric ceramic reticulate-epoxy composites of an orthorhombic shape (henceforth referred to as "coupons"). Each coupon was about 1" square by 1/4" thick. The coupons were prepared by replication of an open cell polyurethane foam with a piezoelectric ceramic. It was necessary to stretch the foam at elevated temperatures, in order to form an anisotropic structure for replication so that the final piezoelectric composite would have enhanced hydrostatic piezoelectric properties. The foam is cut to size after stretching, taking into consideration shrinkage factors. The foam was coated with piezoelectric slurry, dried and placed in a high temperature electric furnace for "burnout" of the foam and sintering of the ceramic. The piezoelectric ceramic foam replica was filled with epoxy, surface ground, cut into

coupons, and poled. The 4 x 4 hydrophone arrays were assembled from the 1" x 1" x 1/4" coupons.

The project has shown conclusively that the reticulate ceramic processing methods are applicable to the production of hydrophones and short range projectors. Moreover, the processing adapts well to low volume, custom shapes and high volume repetitive shapes. A wide range of shapes are readily produced with low tooling costs. Prototyping turn around times can be quite rapid compared to other types of processing, especially when unique shapes are sought. Finally, this report offers suggestions for an improved next generation of reticulated ceramic composite materials as well as array design improvements.

III. PROCESS DESCRIPTION

The overall process flow diagram is shown in Figure 1. The individual process steps are described below.

Cut Foam

Stretch Foam

Cut Foam

Batch and Ball Mill PZT Slurry

Coat Foam with Slurry

Fire

Impregnate Reticulate with Epoxy

Surface Grind

Electrode Composites

Pole

Test Composite Coupons

Assemble Array with Conductive Interconnects

Attach Wires

Encapsulate with Urethane

Test Array

Figure 1 Flow diagram of process description for the production of distorted 3-3 hydrophone composites from reticulated ceramics.

A. Cut Foam

An open cell polyurethane foam was used to make the prototypes with 15 ppi (pores per inch). Very large buns (8' x 4' x 4') were cut down to 4" x 4" x 12" which were then used in the foam stretching step which followed. Looking at the foam, one can see that there is some anisotropy, whereas there is a direction where the foam looks slightly more oblong than in the other two directions. This is referred to as the "football" orientation. The foam was cut such that the "footballs" were oriented in the 12" direction, which later becomes the 6 mm dimension and poling direction.

Specialized foam cutters do the foam cutting with equipment and jigs developed at Hi-Tech Ceramics. Much of the time involved was set-up time, so for prototypes, the times were longer than they would be for larger production orders.

B. Stretch Foam

The 15 ppi foam cut in the prior step to 4"x 4"x12" was oriented with the footballs parallel to the direction of stretching (long ways). Each of these small buns yielded about 20 coupons (1" x 1" x 1/4") which after losses yields about 16 coupons or one array. The length prior to stretching between the grips was 8". See Figure 2. Aluminum tape was wrapped around each end of the foam and then a clamp (part of the stretching machine) was bolted together around the aluminum tape. For the Hi-Tech

stretch apparatus the tape was essential for gripping friction at higher temperature. The bolt for the grip goes through the foam.

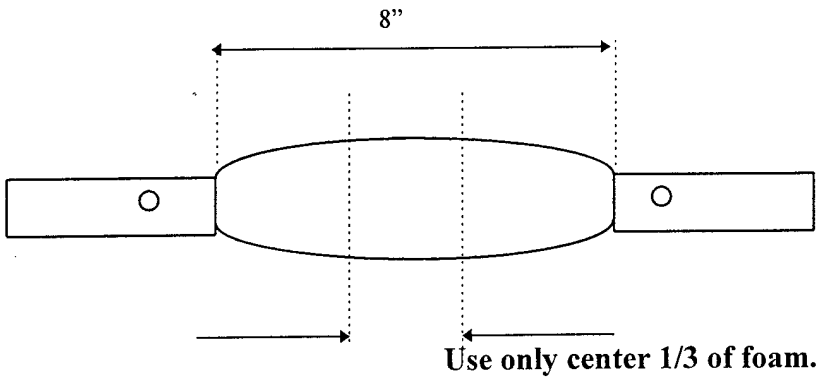

Figure 2 A 4" x 4" x 12" bun of foam as prepared for stretching with clamps.

The stretching apparatus (shown in Fig. 3) is adjustable up and down and has two pins to hold it in several different potential positions. The foam stretch machine was heated to 315 F, after which pressure is applied. The foam was then allowed to cool in this stretched condition.

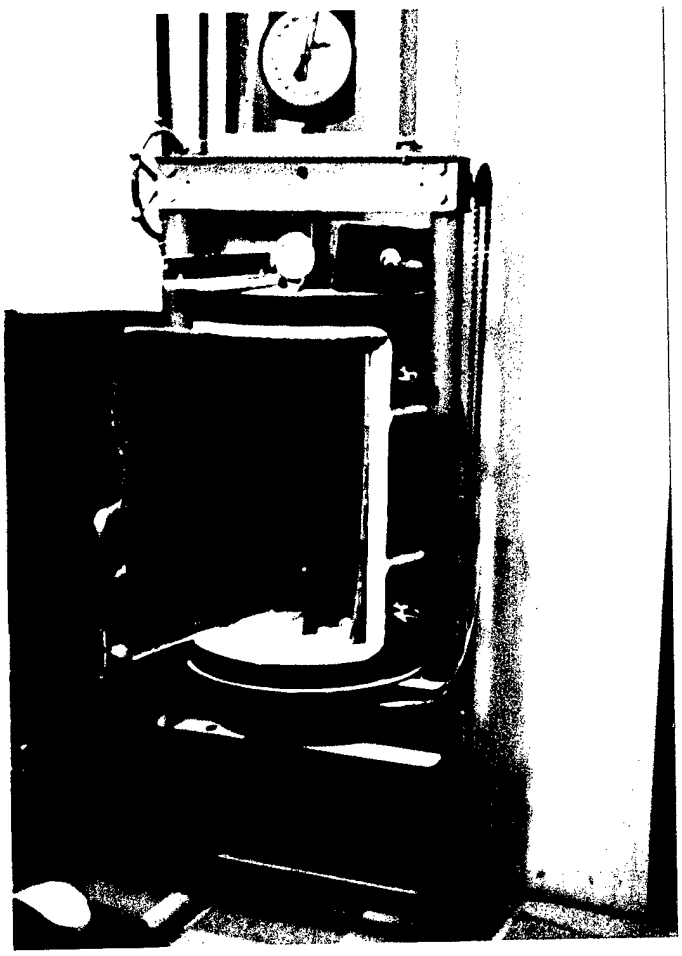

Figure 3 Foam stretch apparatus.

Each small bun will yield about 20 coupons. With the present set-up two thermal runs can be comfortably run in one 8 hour shift, netting two buns or about 40 coupons, and thus about two arrays worth per day.

C. Cut Foam

Due to firing shrinkage, machining and other factors the foam was cut large to 30 mm x 30 mm x 10 mm per coupon. It was cut such that the 10 mm

direction was parallel to the foam stretch direction. Only the center one-third of the bun (See Figure 2.) was used in order to have a maximum perpendicularly of the ceramic webs with the 30 mm x 30 mm plane.

About 25% of the original volume of foam gets used after the cutting process from the original large buns to the ~ one inch coupons. Each small bun was 1.33 board feet and yields about 20 coupons or one array, choosing the best 16.

D. Batch and Ball Mill PZT Slurry

PZT 5H piezoelectric ceramic was obtained from Morgan Matroc. Since the ceramic powder had been previously spray-dried, it was ball milled in a 1 quart plastic jar for 11 hours with zirconia ball milling media, in order to break down the agglomerates.

E. Coat Foam With Slurry

A special room was constructed at Hi-Tech Ceramics to work with the piezoceramics which houses the furnace, hood, ball mill, work tables, spinner, and spray equipment.

The foam coupons were dipped into the slurry from the ball mill, individually spun, and sprayed off lightly with air to cause an even build up on the foam. The entire set was then put into a drying oven for about an hour. This was repeated several times until the desired wet weight (7.5-8.0 grams) was reached, yielding a final fired reticulate of approximately 20 volume percent ceramic and 80 volume percent porosity. It was important to keep the wet weights of the individual coupons very close to the same weight; this minimizes the variability in capacitance and acoustical impedance from coupon to coupon.

F. Fire

A Harrop furnace with a Honeywell controller, over-temperature protection and chart recorder were used to fire the piezoelectric ceramic reticulates. See Figure 4. Two coupons were placed on an alumina setter. To avoid contamination from the setter, sacrificial PZT was placed in the bottom of the crucible. See Figure 5. Three such setters were stacked on a round alumina setter and covered with an alumina crucible. Two such crucibles were used per firing for a total of 12 coupons per firing. Lead rich atmosphere powder supplied by Morgan Matroc was added to each setter. The amount of lead rich powder added was 44% of the dry weights of the total weight of the two coupons. The furnace was capable of accommodating more crucibles, several if stacked.

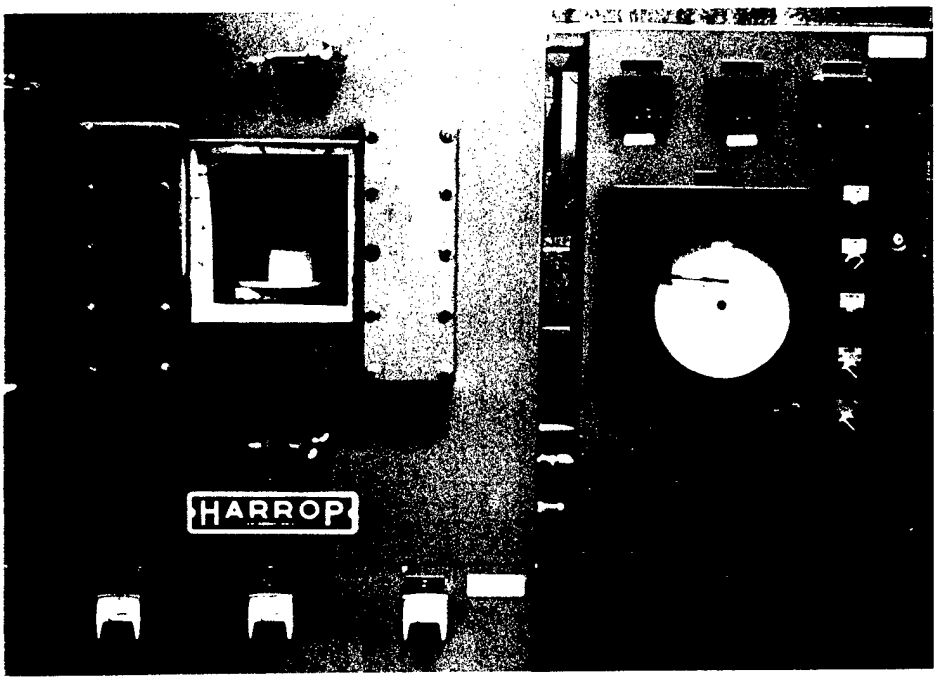

Figure 4 Furnace used to fire the reticulated piezoelectric ceramic.

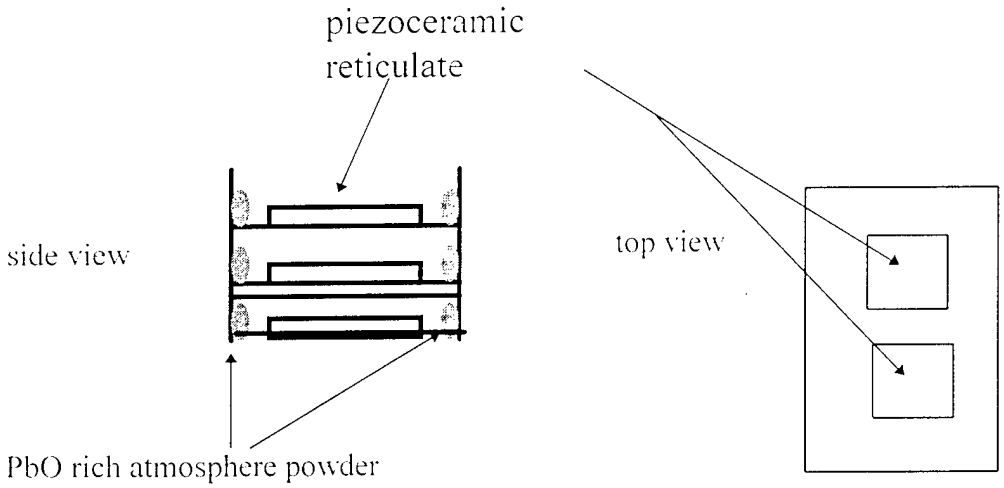

Figure 5 Setter arrangement with piezoceramic coupons and excess atmosphere powder.

The firing cycle was as follows:

6 hr to 600 F

3 hr to 932 F

1 hour hold

12.18 hr to 2372 F

1 hr hold

10 hr to 100 F

G. Impregnate Reticulate with Epoxy

An RTV silicone mold (~4" x 4" x 0.5" deep) was made to hold 16 ceramic reticulated coupons to be impregnated with epoxy. See Figure 6. The RTV silicone mold was sprayed with mold release (Dwight Products, Inc. Lyndhust, NJ) at least three times, allowing the release agent to dry each time. The coupons had to be kept clean to assure good bonding with the epoxy. The mold release was sprayed in a separate room from where the ceramic was to avoid airborne contamination of the ceramic. The 16 coupons were set in the mold 4 coupons by 4 coupons. A four part epoxy, No. 51350 Spurr Low Viscosity Embedding Epoxy (Ernest Fullam Inc. Latham, NY), was flooded in around and over the piezoelectric ceramic reticulate. The epoxy has a viscosity close to that of water, and so it was fairly easy to remove air bubbles by evacuation. The epoxy was weighed out accurately, as changes in the ratios change the cured stiffness of the epoxy. It was also mixed quite thoroughly, for at least 3 minutes. In order

to fill the mold the total amount of Spurs needed was about 100 grams. The formula used for the 6 prototype arrays is shown below.

Spurs Embedding Formula

bottle label	weight (grams)
ERL	22.5
DMAE	1.0
DER	18.0
NSA	58.5

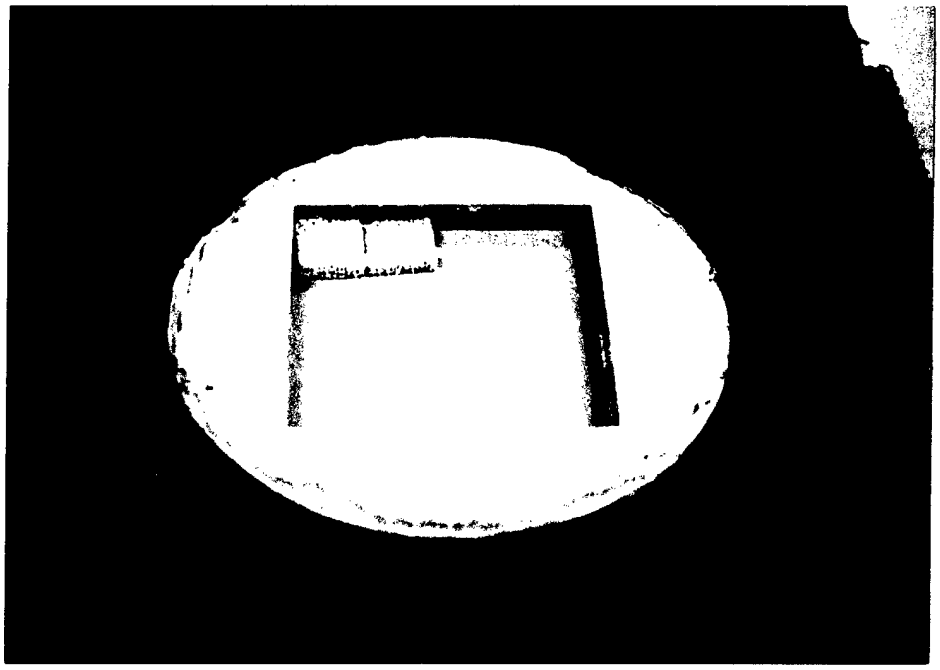

Figure 6 The RTV silicone mold for epoxy impregnation was shown with 2 of 16 reticulated piezoceramic coupons.

When the epoxy was warm, bubbles were less prevalent and easier to remove. This point was quite important. For this reason, the temperature in the room has a significant effect on the ease of processing and quality of the

composites. Air bubbles enhance voltage breakdown during poling and affect the acoustics due to variability.

A vacuum was pulled on the epoxy both after mixing (5 minutes) and after impregnating (20 minutes or until bubbles stop) the piezoelectric ceramic reticulate. Sixteen open ceramic reticulate coupons were placed in an RTV silicone mold. Epoxy was poured slowly about the ceramic while slightly tipping the mold, avoid entrapping air bubbles and causing the reticulate to be pushed up and out of place due to buoyancy. It was only necessary to cover the reticulate, discarding the excess epoxy, since too much epoxy over the reticulate, means too much cured epoxy to surface grind off later. The impregnated reticulate within the mold was carefully transported to the curing oven, taking caution not to tilt the mold, allowing air to get back into the coupons.

The epoxy cured in a minimum of 8 hours at 70 °C. It was important not to use an oven that swings up and down in temperature, causing unwanted thermal stresses and depoling problems. The oven used is shown in Figure 7. When the mold was removed from the oven it was best to let it cool and allow the reticulate-epoxy composite to shrink away from the RTV silicone mold, rather than take a chance in breaking the composite by physically bending it while pulling it from the mold. After the composite 4 x 4 was removed, the mold was cleaned with isopropyl alcohol and paper towels.

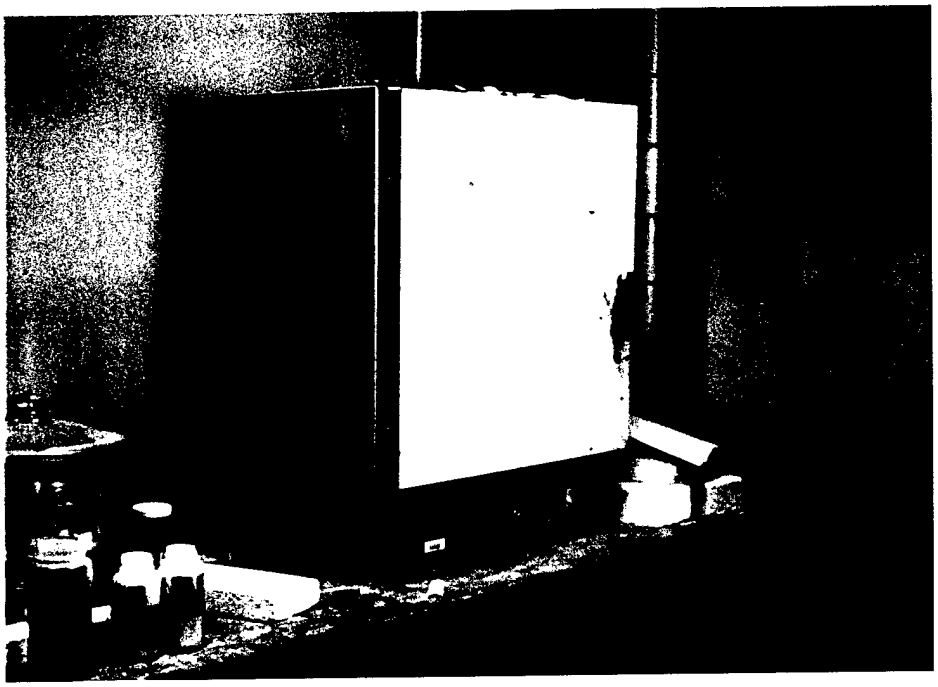

Figure 7 Oven used to cure the epoxy, conductive epoxy, and urethane encapsulation.

H. Surface Grind

Surface grinding was accomplished using a Model Felker, Bay State surface grinder made by Dresser and a new diamond coated cutting wheel (10.0" x .062" x 5/8 arbor, MD 60 N 100MP 3/16) made by Diamond Systems, Inc. of Gardena, CA. See Figure 8. Each piezoceramic composite ~ 4" x 4" x 10 mm was mounted on the surface grinder with duct tape. Cross feed rates were .080" per sweep. Before final cuts were made, the wheel was trued up

with a magnetically mounted diamond faced truing stub. Depth of cuts were limited to .010" and dropped to .005" for two cuts, and finally to .001" to .002" for the last 5 cuts. It was important not to make large depth changes, in that the piezoelectric ceramic may fracture down into the webs of the ceramic reticulate at a depth beyond the final machining depth. This would cause poling and other problems.

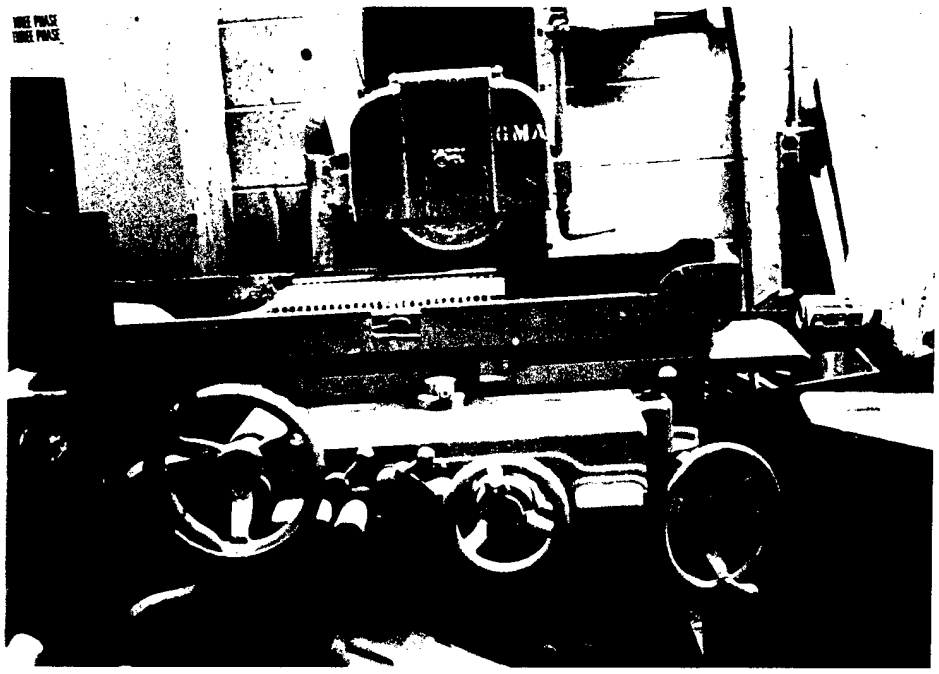

Figure 8 Surface grinder used to machine the piezoceramic-epoxy composites to their proper thickness.

The final composite thickness with silver epoxy electrode had to be under 6 mm to fit comfortably into the aluminum mold (used for urethane encapsulation) with 1 mm urethane spacers on each side. Approximately 6 hours was necessary to properly grind each 4" x 4" composite plate to a

target thickness of 5.85 to 5.90 mm and thus 7.5 hours for the 20 coupons for one array (selecting the best 16).

I. Cut

A cut-off saw manufactured by Covel (s/n 17H-5767) of Benton Harbor Michigan was used to cut the 4" x 4"x 5.85 mm piezoelectric composite plates into individual 23.75-24.00 mm (~ 1 inch) coupons. See Figure 9. Sample pieces of plastic were cut and measured to set up the saw to the right cutting dimensions. Then each composite sheet clamped to the table and cut, using water to cool the cutting surfaces.

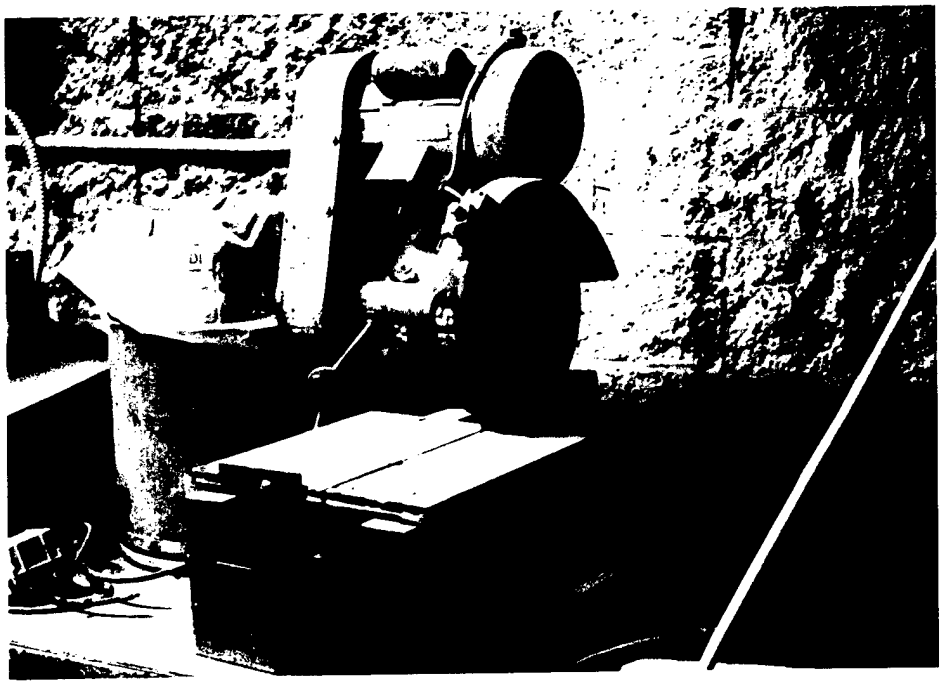

Figure 9 Cut-off saw for cutting the piezoceramic-epoxy composite sheets into coupons.

J. Electrode Composites

A two-part conductive epoxy, Ablebond 1601, Ablestik FSCM 21109, was used to electrode the piezoelectric reticulated ceramic composites. The epoxy used was obtained from:

Ablestik Electronic Materials & Adhesives
20021 Susana Road
Rancho Dominguez, CA 90221.

This epoxy cures in about 1 hour at 65-70 °C. Some of the coupons were screen printed and others were electroded by holding the coupon and spreading the conductive epoxy with a razor blade. The razor blade method was adequate for small quantities, due to the time associated with set-up and clean-up of the screen printer. The wavelength in water at 100 kHz is 15 mm so a fair amount of roughness on the surface (composite only 6 mm thick) was tolerable. The screen for screen printing was obtained from RIV Inc.; 908 Columbia Circle, P.O. Box 220, Merrimack, NH 03054. For screen printing, the viscosity was dropped slightly by adding a couple drops of isopropyl alcohol. The hold off distance was about 1 mm. The screen printer was furnished by the New York State College of Ceramics at Alfred, NY. See Figure 10.

After printing an electrode on one side, the coupons were placed in an oven to cure for an hour, and the process was repeated for the other side.

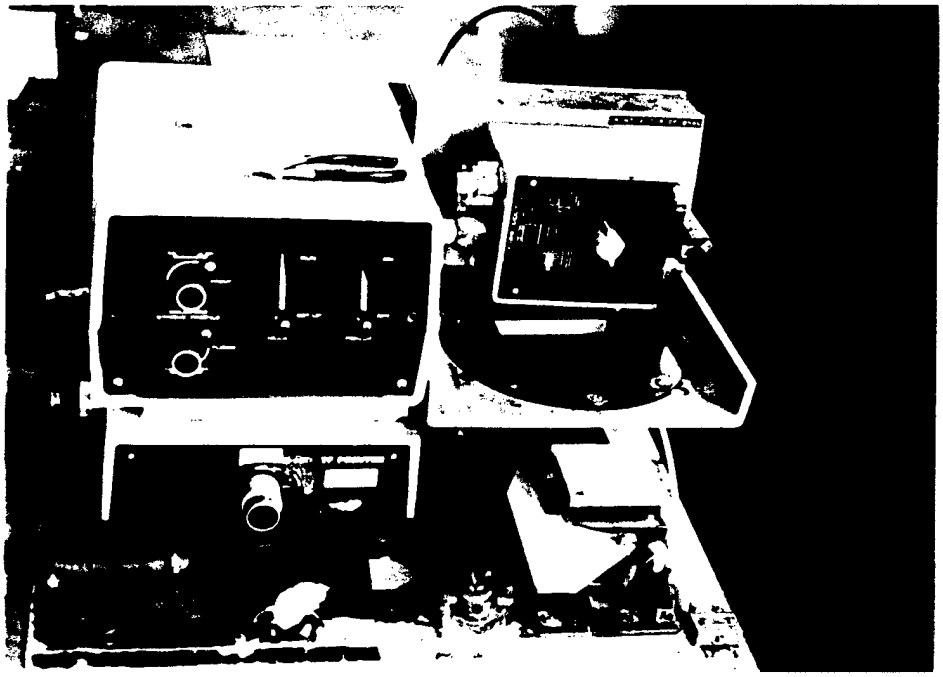

Figure 10 Screen printer used to apply conductive epoxy electrodes.

K. Pole

The composite coupons were poled at the New York State College of Ceramics, with a power supply manufactured by Spellman, High Voltage Electronic Corporation of Plainview, NY 11803, SK Series, PN SL 40PN150, Model Rev.A1, 0-40 kV/3.75 mA. See Figure 11.

Figure 11 Poling apparatus including power supply, heater, and tank filled with household cooking oil.

Due to the high voltages associated with poling, caution was taken to protect the person doing the poling. A thick electrically insulating mat was used on the floor below the apparatus and where the operator stands. The leads were securely held in place with suitable insulators and connectors.

The coupons were individually heated in cooking oil for about 5 minutes and poled at about 17 kV/cm for 5 minutes at 70 ^{0}C. The operator presets the voltage then activates it with one finger, while standing on the insulated

mat. It was found that an overall higher d_{33} (and thus d_h and g_h) could be obtained by using a slightly higher poling voltage. Higher voltages (20 kV/cm) were possible on most samples, but due to the arcing of some samples, the voltage was dropped. For the prototypes individual coupons were poled separately, but if larger quantities were being manufactured, more coupons could be poled at one time with the same or similar power supply. While one coupon was being poled, the previous one was cleaned off and had its d_{33} measured. Isopropyl alcohol was a suitable solvent for removing the oil without affecting the epoxy. The positive pole of each coupon was marked with a dot in the center of that side.

L. Test Composite Coupons

A Channel d_{33} meter was used to measure d_{33}. Standards were provided with the instrument for calibration prior to use. To assure adequate poling, d_{33} was measured and recorded on all the coupons. See Figure 12 below. At a proposed operating frequency of 100 kHz, the composite looks uniform; however some specific d_{33} readings may be low since the probes come to a point, and there may be no ceramic reticulate below that particular point. For this reason measurements were taken in five places and averaged. The average d_{33} for each coupon exceeded 230 pC/N at 100 Hz.

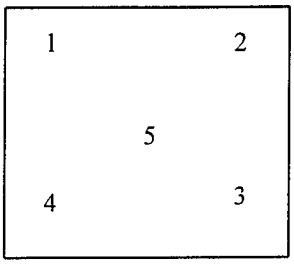

Figure 12 Face view of one piezoelectric ceramic coupon showing points where d_{33} measurements were taken.

The capacitance of each coupon was also measured. The best procedure was to fabricate large numbers of the composite coupons and record their individual capacitances. The capacitance values can then be put into a spreadsheet such as Excel and sorted from low to high or high to low. This allows the array builder to set the ceramic into the array in order of capacitance assuring that each ceramic was approximately the same capacitance as the one next to it. According to Fred Geil at Northrup Grumman, this yields an overall better wave form and acoustic performance.

The hydrostatic charge coefficient was measured in the Electroceramics Division of the New York State College of Ceramics in Alfred, NY. A photo of the set-up is shown in Figure 13. A standard was supplied by the Office of Naval Research. A stacked ring configuration driver provides a 400 Hz signal. By measuring the voltages across the standard and sample

and taking the areas of each into consideration, the hydrostatic charge coefficient, d_h, of the sample was obtained:

$$d_h = d_s \frac{A_s V_c}{A_c V_s},$$

where d_s was the hydrostatic strain coefficient of the standard, A_s was the area of the standard, A_c was the area of the composite, V_c was the voltage of the composite, V_s was the voltage of the standard. The pressure was incremented in steps: 250 psi, 500 psi, 750 psi, and 1000 psi, taking voltage measurements at each pressure. Also of interest was whether the sample was damaged in testing to 1000 psi. This was checked by rerunning the same sample to see if the same d_h values were obtained. Fred Geil suggested sending some poled coupons to an outside vendor that he knows for extensive high pressure cycling (1000 cyles) at a cost of $1200, and then retesting the coupons against control coupons that were not subjected to the cycling pressure test.

Figure 13 Set-up for measuring d_h.

M. Apply Conductive Interconnects to Array

All 16 coupons of the first array were connected in parallel. For each of the last 5 arrays, each section of 2 sets of 8 coupons were connected together in parallel, forming Section A and Section B. All 16 coupons had their positive polarity facing the same direction. A conductive adhesive copper tape, model 837, was purchased from Compac Industries, Inc, Edison, NJ 08837. An aluminum mandrel was made to bend precut (5/8" long by 1/8" wide) conductive adhesive copper tape as shown in Figure 14 below. The individual coupons were ordered in increasing capacitance adjacent to one another to minimize acoustical variations in the final array.

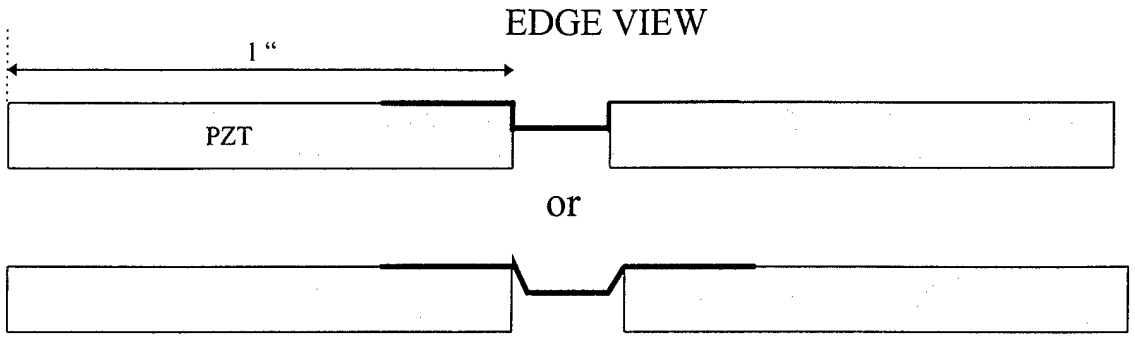

Figure 14 Edge View of the coupon showing the adhesive copper conductors taped to the coupon. This configuration gives the array a high degree of flexibility and allows the array to conform to curved surfaces.

A top view is shown in Figure 15 below, showing the staggering of the copper conductive electrode tape. This was necessary to avoid high voltage arcing. There must be adequate distance between the electrodes to prevent voltage breakdowns in the urethane encapsulation for voltages as high as 4000 volts.

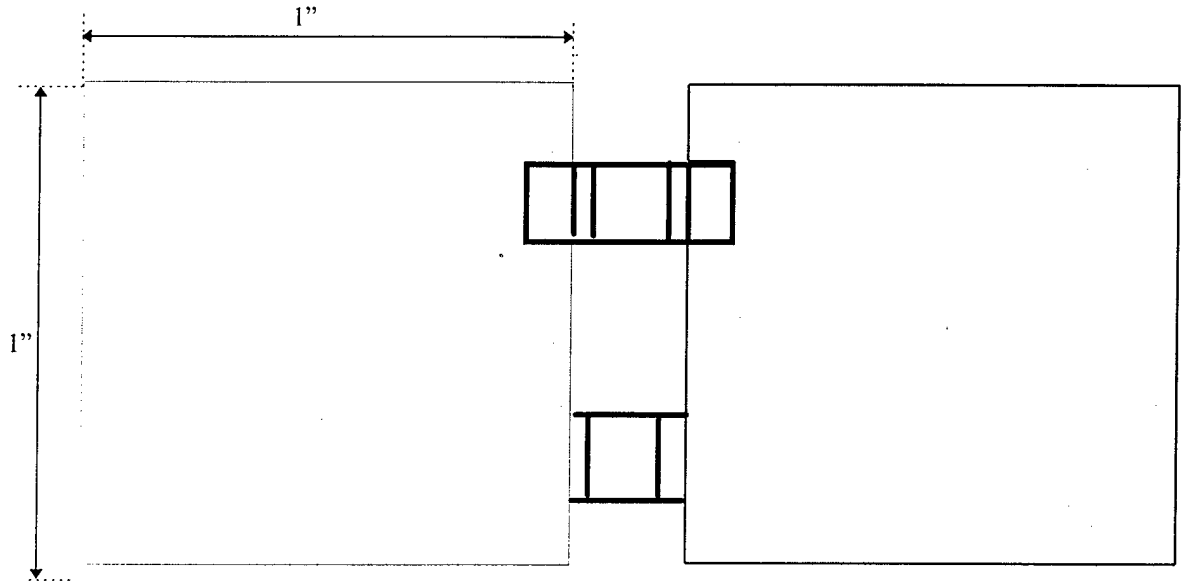

Figure 15 Top view of 2 coupons showing adhesive tape copper interconnects.

This configuration allows the array to be flexible and thus conformable to a curved surface. For this reason it was necessary for the copper to be bent at one-half the thickness of the coupon (3 mm for the 6 mm thick coupons). It was important to clean and dry the surface of the coupons with isopropyl alcohol to assure good adhesion of the conductive tape.

Two mm urethane discs (~1/16" in diameter) were used between the coupons by gluing them in with Superglue. The urethane, produced by Conap 1405 Buffalo Street, Olean, NY 14760, goes by the trade name

Conathane EN-4 Part A Urethane Prepolymer and Conathane EN-12 Part B Curative. It was prepared by mixing equal weights of both A and B. The viscosity was high so each should be preheated before mixing. The mold was preheated to help the urethane stay warm and to have a low viscosity and flow through the mold easily. Two molds were made: one to make 1 mm sheets of urethane and another for 2 mm sheets. The spacers hold the coupons parallel to each other. The spacers were made by punching out ~1/16" discs from the urethane sheets with a leather punch. Most of the coupons have two 2 mm spacers along each of their four edges, along the outside of the squares superglued 3 mm high at the center of the thickness. Some were left out to allow the urethane to flow more easily. Four 1 mm thick by 1/16" diameter urethane spacers were glued to each face of each coupon (very near to the four corners) to allow the urethane to flow over the face of the conductive electrodes. This urethane coating formed a protective layer to the electrodes. Three mm spacers were used between the two sides of the array, since there seemed to be that much space to fill, after using 2 mm spacers around the outside of the array between the array and the aluminum mold.

Air-dry conductive adhesive was painted over the copper conductive adhesive tape and out to about 1/16" beyond the tape as a precaution to assure good continuity to the silver epoxy. The air dry conductive paint was acquired from GC Electronic in Rockford, Il., catalog number 22-246. The air-dry dries in about 10 minutes.

N. Attach Wires

Ablebond 16-1 epoxy was used to attach the conductive leads. This was the same epoxy used to make the conductive electrodes. It was crucial to keep the bare lead wires within the urethane encapsulate far away as possible from one another, since the pulse voltages were potentially as high as 4000 volts. Care was taken not to stretch or step on the electrical lead wires, since doing so may change the capacitance per linear foot as specified in the catalog (here 29 pF per linear foot).

RG-174/U (Belden #8216) PVC coated coaxial cable was obtained from Anixtar, 4 Marway Circle, in Rochester, NY 14624. The coaxial wire was cut exactly to 30'. For the first array with all 16 coupons in parallel, the inner wire of the coaxial cable was connected to the positive polarity and the outer shield to the negative polarity. For the other five arrays, having a Section A and Section B, 2- 30' coaxial wires were needed. The inner conductor (positive polarity) of one wire was connected to the Section A positive polarity and the inner connector of the other wire was connected to Section B positive polarity. The outer shield of each wire was connected to the negative polarity of each Section, respectively.

The proper polarity conductors were bonded in place on one side of the array with a small drop (3-4 mm diameter) of two part conductive epoxy. Alligator clips were used to hold the conductors in place while transporting

and for the one hour oven cure at 65-70 °C. The alligator clips also grounded the array while it was in the oven. It was necessary to make sure that both sides of the array (and both Section A and B) were grounded from the positive polarity to the negative polarity. Additional alligator clips were added in other locations in case the clips fell off, avoiding depoling problems. Once the wires were connected on one side of the array and cured, it can be flipped over and the process was repeated for the other side of the array.

O. Encapsulate with Urethane

After the wires had been attached to the array the entire assembly was set into the aluminum mold which had been sprayed with mold release and allowed to dry. An inadequate amount of mold release made it very difficult to remove the array from the mold. The mold release was not sprayed in the same room where the array is, since the airborne spray may deposit itself on the array surface, hindering adhesion. Wooden stirring sticks were not used when working with the urethane since absorbed water can cause cloudiness in the urethane. The mold is shown in Figure 16.

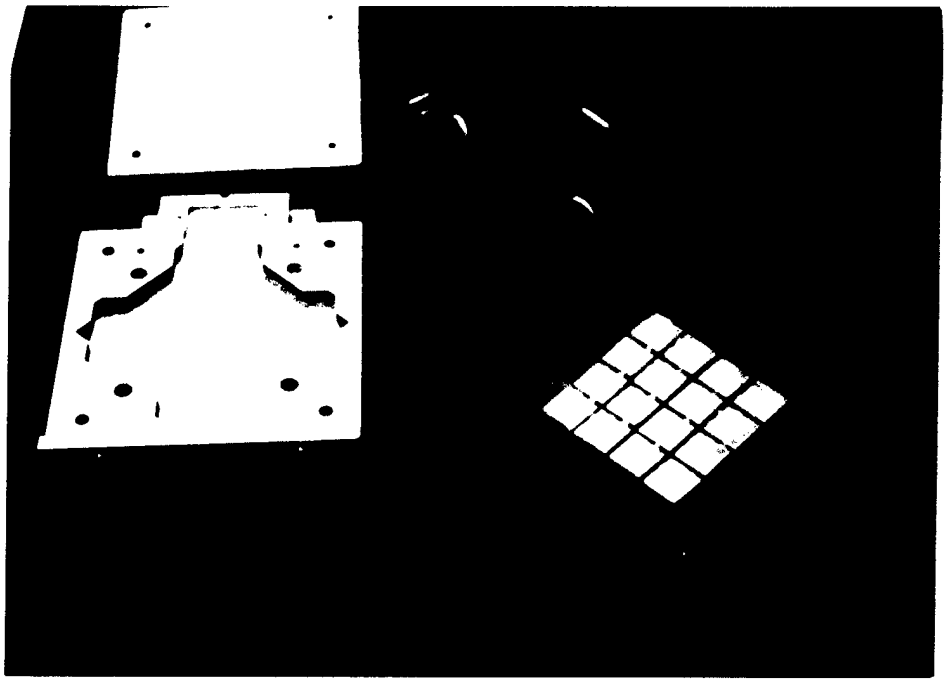

Figure 16 Mold used to encapsulate the piezoceramic composites in urethane.

The positive and negative ends of the wires of each half of the array were wrapped together to prevent depoling during curing. Morgan Matroc gives a maximum use temperature for their PZT 5H of 120 C, but some depoling effects may occur as low as 100 $^{\circ}$C. Care was taken not to use an oven that may exceed 100 $^{\circ}$C due to inadequate temperature control or isolated hot spots.

A two part urethane was used to encapsulate the array, providing a protective coating for the electrodes and some acoustical matching. The urethane, produced by Conap 1405 Buffalo Street, Olean NY, goes by the trade name Conathane EN-4 Part A Urethane Prepolymer and Conathane EN-12 Part B Curative.

Part A and Part B were preheated in an accurate temperature controlled oven to reduce their viscosity since at room temperature they were thick like

syrup. About 100 grams total was needed per array. The mold, with the array contained within it, was preheated so that the urethane did not cool when poured into the mold. These preheat times take about one-half hour at 70 °C. It was preferable to raise the temperature slightly toward 80 °C to lower the viscosity as much as possible. When the viscosity was at its lowest, air bubbles tended to come to the surface and expel themselves without the use of a vacuum or minimum vacuum time.

First the array was placed in the mold and the cover placed on the mold and this assembly was placed in the oven to be heated. Also, the urethane Part A and Part B were heated. Then the Part A and Part B were removed and mixed thoroughly for five minutes, and a vacuum was pulled on the urethane to remove air bubbles. The vacuum time must be minimized, since the urethane was cooling during the vacuum time, and the viscosity was going up. Then the mold and array were removed from the oven and stood vertically. The reservoir was filled about 2/3 way full with the urethane and the plunger was inserted. The more urethane used, the more likely all the air bubbles were flushed out. The plunger could not be removed to add more urethane, so it was imperative to fill the reservoir with plenty of urethane. The plunger was pushed slowly and steadily so as not to dislodge any of the coupons by excessive force or pulses of pressure. The plunger was pushed continually until the air bubbles stopped coming out about the electrical wires, where the urethane escapes at the top. The plunging was stopped intermittently during the plunge to remove excess urethane as it comes out around the wires and tends to fall over the top of the plunger and

down into the reservoir. The mold was tipped away from the plunger side slightly to cause the urethane to run down the opposite face; this face was previously sprayed with mold release to stop the urethane from sticking. When no more air came out of around the wires and the plunger was in the down most position, the mold was wiped clean and placed in the curing oven.

The mold was placed in an oven in the same upright position. If the urethane was still tacky to the touch, the array was not removed, since it would be damaged. The urethane takes about 24 hours to cure at 65 ^{o}C- 70 ^{o}C. When the urethane was properly cured, the excess at the top of the mold will not be tacky but rubbery. After the urethane has been properly cured, the mold and array within it was removed from the oven to cool. It takes about 30-45 minutes for the mold to cool enough to be handled. The more it was allowed to cool, the more rigid and less tacky the urethane became, making it easier to remove the array without damaging it.

P. Test Array

The array was visually inspected for air bubbles that may have remained trapped and repairs were made if necessary by cutting open the bubbles and making more urethane, filling the void, and perhaps placing it back in the mold. When the proper procedure was followed, there were no air bubbles

and no need for repairs. The impedance of Section A and Section B were measured and compared. A check was made by adding the total capacitance of the individual coupons that was measured prior to building the array and by subtracting out 29 pC per foot for the wire. An HP 3585A spectrum analyzer with a tracking generator was used to view the overall frequency spectrum of the arrays, including resonances. An HP 4192A Impedance Analyzer was used for comparison; National Instruments Labview software was used to obtain plots of impedance vs. frequency.

The arrays were tested acoustically by Fred Geil at Northrup Grumman in Baltimore in their large redwood acoustic testing tank, using a standard supplied to them by the Navy.

IV. PROCESS SCALE-UP

Scale-up is proposed for an area equal to 1200 square feet as an approximation for one attack submarine. It is expected to take one year. Instead of making one inch squares x 1/2" thick composite material, 3" x 4" x 1/2" composite plates are considered, as it lowers the lateral resonant frequency and makes them easier to produce. The "Cutting" step and the "Conductive Interconnect" step are reduced. At these volumes Hi-Tech Ceramics narrows its role to ceramic composite fabrication, leaving the wire attachment and probably the encapsulation to the systems manufacturer.

Using 1200 square feet as a target as well as one year (50 weeks) and planning on selecting 89 percent of the 3" x 4" plates, 320 plates per week and thus 64 plates per day minimum would need to be fabricated.

A. Cut Foam

This process is the same as for the prototypes, except that all the foam for 1200 square feet can be cut at one time. This saves a great deal of set-up time.

B. Stretch Foam

Since it is desirable to cut as much foam as possible at one time in the next step, it is of interest to stretch as much foam as possible, as fast as possible. Either several foam stretch machines (5-10) will have to be built, or different ones which are much longer will be necessary.

C. Cut Foam

The same basic process is used as with the prototypes, except that the foam cutters cut to approximately 3" x 4" x 1/4". The time per area is greatly reduced by having larger pieces and by cutting large quantities at one time and reducing the total set-up time.

D. Batch and Ball Mill PZT Slurry

Large jars of PZT can be batched at one time and ball milled. Milling times can be reduced to mixing times if a slightly different powder is purchased. The actual time that a person spends weighing out the batch is minimal.

E. Coat Foam With Slurry

A large amount of foam can be coated per day by one person. Since the foam is dipped several times, another drier would be purchased to keep up with the load. Additional electric and/or gas service to supply the dryer would be needed as well.

F. Fire

The inside cavity of the Harrop furnace is 18" deep by 10" wide x 12" high with a thermal couple protruding into it from the back near the roof of the cavity. If the entire furnace was used 360 parts could be fired per firing. Allowing for the crucible and setters, etc. and if the furnace is fired three times per week on the average, no extra furnace will be needed. Allowing for furnace maintenance and lags in production, it is advisable to purchase another furnace of the same size.

G. Impregnate Reticulate with Epoxy

From a project management standpoint, the daily capacity might be confortably set to be twice the 64 parts per day planned. About 150 RTV silicone molds would allow for increased production and allow the

previously used molds to be cleaned during vacuum time, etc.. A stable flat table and space is necessary as well as two vacuum pumps and two bell jars.

H. Surface Grind

A more suitable surface grinder (potentially a Blanchard) and fixture would be purchased allowing much faster machining of the composite material. By tight control of the foam cutting and impregnating steps, the amount of surface grinding would be minimized. It is expected that by choosing a suitable grinder and fixture, and with tight control over previous steps, that the grinding time per side of each part be on the order of a couple minutes per side. At 64 parts per day, there is 6.0 minutes available per part. If necessary, a second shift could be run.

I. Electrode Composites

The same two-part conductive epoxy type material is planned for the scale-up operation. An alternate approach is to apply electrodes by copper plating and sputtering gold alloy. This second approach would not be done in house. If the first approach is used, the parts will be screen printed and cured in the same ovens used to cure the impregnated epoxy from an earlier step.

J. Pole

Though 64 parts would be needed to be poled per day, the time per poling is 5 minutes, and about 5 minutes to heat up and remove, for a total of 10 minutes per part. At this rate about 40 parts could be done individually per shift, with the same type of power supply and poling set-up used for the prototypes. Since the power needed is quite minimal, in that the parts are insulators and thus nonconducting, it is probable that two or three parts could be poled simultaneously. This could be tested by measuring d_{33}.

If a high power commercial type poling power supply was purchased, 64 parts could be poled at one time. A larger tank would be required as well as sample holders.

K. Test Composite Coupons

A Channel d_{33} meter can be used to measure d_{33}. They would be tested individually as with the prototypes. Again, they can be tested in several spots and averaged. Again the capacitance would also be measured and recorded, allowing similar capacitance parts to be adjacent to one another.

The Hydrostatic Charge Coefficient set-up in the Electroceramics Division of the New York State College of Ceramics in Alfred, NY is being modified

to handle larger area parts such as these. This was described in the Prototype Section. This type of testing is fairly slow and so only selected parts representative of the group can be chosen, unless more modifications are made to the system, such as computer data collection.

L. Lead Attachment

Leads will have to be either epoxied (epoxy conductor) or soldered (copper plate/gold sputter) to the parts prior to encapsulation.

M. Encapsulate with Urethane

It might be desirable for the systems contract company to encapsulate many parts together after attaching the wires. Hi-Tech Ceramics is not privy to these processes. If the composite material is encapsulated at Hi-Tech Ceramics, Inc. either the details would be needed or each part could be encapsulated as the prototypes were. In the latter case, no less than 64 similar molds would be needed. If the process requires temperature, adequate oven space would be required.

N. Test

Quality control people would be needed to monitor variability and quality issues. Weight, d_{33}, capacitance, impedance, and d_h, could be measured internally with the purchase of certain pieces of equipment.

Acoustical testing would have to be performed at the systems contracting facility. Some of the parts would be sent out for long term repetitive pressure cycling to spot potential application damage.

V. ECONOMICS

The costs vary considerably depending on scale of manufacture, because of the costs associated with initial start-up costs. A certain amount of equipment has been purchased, related to processing of the six prototypes, however, developments during the prototyping and associated design modifications lead to particular changes in the modes, and since the initial project was one of proof-of-concept, certain equipment necessary for a high quality product were not purchased as of yet, in particular proper cutting and grinding equipment and tooling. To produce 1200 square feet per year it is estimated that seventeen full time employees would be needed, including one production/quality supervisor and one engineer.

At 1200 square feet per year the following cost estimate has been compiled (Table I and Table II). It is expected that some of the costs can be reduced by purchasing used equipment, renting, or other cost savings measures.

Table I Equipment costs for scale up.

EQUIPMENT COSTS

EQUIPMENT	DOLLARS
foam stretch machines	10000
jar mill and media	300
dryer	1500
furnace, controller, recorder	35000
shipping, installation	4000
150 RTV silicone molds (20 ea.)	3000
table	100
2 vacuum pumps	700
2 vacuum jars	180
rotary surface grinder	80000
poling power supply	20000
poling tank	300
poling heater	500
Chanel d33 meter	1800
Hewlett-Packard 4192A, impedance analyzer	22000
EQUIPMENT TOTAL	179380

Table II Operating Expenses for Scale-up.

OPERATING EXPENSES

EXPENSE	
Direct Labor	458518
Fringe Benefits (38.5%)	176529
Fixed Plant Overhead (58.4%)	267775
Direct Materials	238030
Other Direct Costs:	
Outside Testing	12000
Travel	4000
consultant fees	10000
hazardous waste removal	10000
other	5000
Subtotal	1181852
General and Administrative (12%)	141822
Total Costs	1323674
Fixed Fee Earned (12%)	158841
Total Amounts Claimed	$ 1482515

VI. CONCLUSIONS

This project has shown conclusively that reticulate ceramic processing methods are applicable to the production of hydrophones and short range projectors. The assembly of composite coupons into flexible, conformable arrays has been demonstrated. Without modifying the material and subtracting out the losses in the wire, sensitivities were -192 dB re 1 volt/micropascal at 100 kHz.

Hi-Tech Ceramics already manufactures hundreds of thousands of square feet of reticulate material per year. The ease of cutting and shaping the foam allows many shapes to be made: squares, rectangles, cylinders, hollow cylinders, dics, donuts, ... Since the precursor is easy to cut to shape, prototyping is relatively rapid. The cost is inexpensive compared to other piezoelectric composites and are projected to be ~ $10/ square inch in moderate volumes. Hi-Tech Ceramics would most probably limit their involvement to the production of the composite material.

Scale-up for large area orders is quite simple, since the basic process is the same. For large area orders the variability diminishes and results in a superior product. Larger production rates also affords more selectivity and less variability within composite sections. At the same time smaller volumes naturally yield higher variability.

Some basic testing of the composite material can be made at Hi-Tech Ceramics. The acoustics of the material would be tested externally. Perhaps, airbox testing could be set-up in house. A one thousand low/high pressure hydraulic cycle test has been suggested and would also be performed externally.

Minor changes in the manufacturing process are expected to result in significant improvements in the sensitivity and linearity of the composite. Hi-Tech Ceramics looks forward to its involvement in the development of this next generation of improved piezoelectric composite material.