ADVANCED CERAMETRICS INCORPORATED

1997 - OUR FIFTIETH YEAR

ADVANCED MATERIALS TECHNOLOGY COMPANY

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Dr. Smith:

Enclosed please find the quarterly progress report for the Phase II STTR, Contract # N00014-95-C-0426, "Proposal to Develop Multifuctional Composites for Sensor and Actuator Applications". As always, we appreciate your continuing support of our PZT fiber program.

Sincerely,

Jonathan D. French
Director of Research

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Quarterly Report - Period of 23 April through 23 July, 1997.

Office of Naval Research Phase II STTR: "A Proposal to Develop Multifunctional Composites for Sensors and Actuator Applications", Contract # N00014-95-C-0426.

Work progress over the last quarter is broken down into 4 areas: spinning, sintering, ultrasonic transducer prototypes and hydrophone prototypes. Projected progress for the next quarter is also given in each section.

1. Spinning Status

Batch size is presently 500 g of PZT powder. Scale-up to 1000 g and 1500 g is planned for the next 3-6 months of the program, as larger batch sizes lead to lower production costs. Powders spun to date include PZT-5H, EC76, BM532, EC64, BM740, and BM400. All have made good quality green fiber. The alternate starting powder sources and compositions are presently being certified for fiber spinning and composite prototype production. EDO Corporation's EC76 and Sensor Technologies, Limited's BM532 are being evaluated as alternate powders to the Morgan-Matroc PZT-5H powder. ACI has also obtained a 1 kilogram sample of TRS Ceramics, Inc. "fine-grained"5H-type powder. Spinning trials on the TRS fine-grain material will begin in the next quarter of the program. Chemical analysis showed a leeching of strontium from the PZT-5H material, and is likely the source of the slightly lower electromechanical properties of the fiber composites compared to the bulk material (specifically d_{33} and dielectric constant). Alternate PZT material formulations, such as PZT-4 type (EC64 and BM400 powders) and PZT-7 type (BM740), are also under spinning certification for ultrasonic transducer production. The lower dielectric constant of these other materials helps provide the impedance match for higher frequency transducers. Chemical analysis of the powders and fiber made from EC76 and EC64 before and after sintering indicate that changes in composition as a result of the spinning process were much smaller for these powders than those observed in spinning the PZT-5H powder.

2. Sintering Status

Sintering studies at Rutgers University have produced the optimal sintering conditions for PZT-5H green fiber (2°C/min up to 1240°C, hold for 40 minutes). This heat treatment produced sintered fibers with high density, average grain size of about 2.5 - 4 micron (see Figure 1) and good electromechanical properties (Table 1). Pyrometric cones were used to calibrate the Rutgers furnace to facilitate the transfer of the heat treatment conditions to the newly purchased and installed control-atmosphere furnace at ACI. The crucibles for firing the preforms are produced in-house from high-purity alumina and zirconia. Alumina is used for the crucibles which hold the PbO-rich sintering atmosphere. High-purity yttria-stabilized zirconia is used for the setter plates on which the fiber is fired, as the fiber does not stick to the zirconia as it does to alumina- or silica-containing materials. Source powder, a mechanical mixture of PbO and ZrO_2 powders is used to minimize lead-loss during fiber sintering. Experiments are underway to determine the optimum amount of source for a given crucible and sample size. These experiments are on-going and will likely be completed in the next quarter of the program.

Sintering experiments are currently underway at Rutgers to determine the optimum heat treatment conditions for fiber made from EC76, EC64 and BM740 materials. Sintering experiments for the BM400 and TRS fine-grained 5H powder will occur in the next quarter of the program.

3. Ultrasonic Transducer Prototypes

Krautkramer-Branson Inc. (KB) tested PZT-5H rope preforms at 6 and 14 MHZ and measured an average of -7 dB lower sensitivity than "dice-and-fill" composites. It was hypothesized that the lower sensitivity was a result of both unoptimized sintering conditions for the fiber and a poor impedance match to the driving electronics (too high a capacitance). KB uses the PZT-5H material for the lower frequency (1 - 2.5 MHZ), due to its high dielectric constant. Transducers were made from the same billet of fiber preform and the same tests were done at the lower frequency ranges. A range of sensitivities of 0 to -2 dB were achieved for the fiber transducers compared to the "dice-and-fill". The sensitivity is expected to improve still more with the new sintering schedule. Characterization and certification tests for prototype production of ultrasonic transducer preforms with KB are scheduled for the next quarter of the program. For intermediate (6-10 MHZ) and high (>10 MHZ) frequencies, fiber preforms from lower dielectric constant materials (EC64 and BM740) to provide the proper impedance match to the driving electronics. Both materials spin well and preform construction and sintering experiments are underway. Transducer testing for these materials will occur in the next quarter of the program.

4. Hydrophone Prototypes

Fiber rope preforms have been sintered and potted in epoxy for construction of hydrophone production. A direct replacement of the fiber composite for a sintered ceramic is being done to test the technology in the low frequency ranges of sonar applications. These composites are being machined to size and will be tested in the next quarter of the program. Other sonar devices, such as towed arrays, are being considered for fiber composites. Those application where PVDF film is used may see better output from a PZT fiber composite, yet still have the flexibility associated with the polymer film. These designs and applications will be pursued in the next quarter of the program.

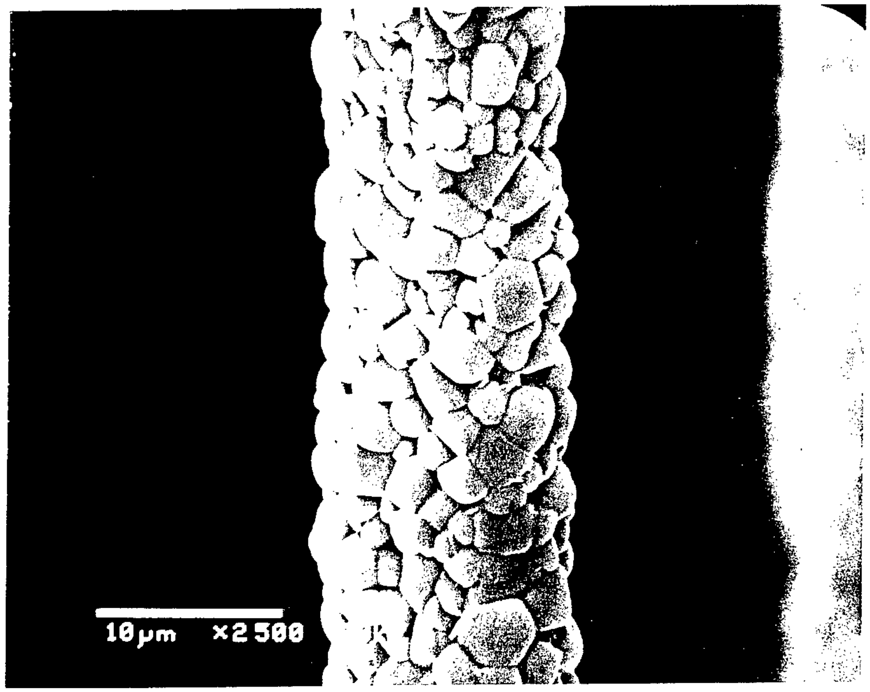

Figure 1. SEM micrograph of a sintered PZT-5H filament (1240°C - 40 minutes). Note the uniform grain gize.

Table I. Electromechanical properties from braided rope preforms of PZT-5H fiber sintered at 1240°C for 40 minutes. Values are the average of four samples.

Property	Composite
Volume % PZT	28
d_{33}, pC/N	331
Dielectric Constant, K	500
tanδ	0.037
k_p	.48
k_t	.55