

DESIGN OF NOVEL INSTRUMENTS TO MAXIMIZE NEWLY DISCOVERED SONOCHEMICAL EFFECTS FOR CERAMIC REACTIONS ACCELERATION

Interim Progress Report #L-4404-1, May 25, 1991

TECHNICAL SECTION

Project Number: 90-009

Project Year: 1991

Project awarded by: Small Business Research "Seed" Grant Program
Pennsylvania Department of Commerce, Office of Technology Development, Room 352 Forum Building
Harrisburg, PA 17120

Firm: Ultram Laboratories, Inc., 139R North Gill Street
State College, PA 16801

Principal Investigators: Mahesh C. Bhardwaj, Director R&D, Ultram Laboratories, Inc., and Dinesh Agrawal, Research Associate, and Rustum Roy, Professor, Materials Research Laboratory, Penn State University, University Park, PA 16802.

INTRODUCTION

In the technical proposal on this topic, we exhibited the evidence of simple chemical reactions in ceramics and cements produced by Very Low Frequency (VLF), high power ultrasound. Reaction mechanism at VLF ultrasound (<100KHz) is believed to be thermo-mechanical in character. "Intense" heat and pressures are produced locally by the formation, and subsequent collapse of bubbles (produced by cavitation in reaction liquids), and by the friction between the grains of reacting materials at VLF. Energy produced by this mechanism is sufficient to induce phase transformation without actually applying the necessary high temperatures and pressures for such reactions to occur.

The main objective of this project is to determine the feasibility of these newly discovered sonochemical reactions through the design and applications of relatively High Frequency (HF) and high power ultrasound. It is believed that HF ultrasound would enhance chemical reactions by transforming molecular bonds due to reduced wavelengths at such frequencies. Here is the summary of our progress on this project.

1 SAMPLE PREPARATION

Stoichiometric amounts of γ -AlOOH (Boehmite) and H_3PO_4 acid were prepared for HF sonochemical reaction experiments. Precursors were mixed at room temperature and some de-ionized water was also added to dilute the solution. These samples were prepared at the Materials Research Laboratory, Penn State University.

2 TRANSDUCERS AND POWER MEASUREMENT

2.1 Transducers: Characteristics of two specially designed and manufactured transducers are given in TABLE I.

TABLE I. Salient characteristics of transducers designed for sonochemical studies.

TRANSDUCER DESIGNATION	FREQUENCY MHz	ACTIVE DIAMETER in (mm)	NUMERICAL APERTURE	FOCAL POINT in (mm)	POWER*
KS150-0.5-P1.5-X	0.5	1.5 (38)	1.0	~0.23 (5.9)	~100mW
KS150-1-P1.5-X	1.0	1.5 (38)	1.0	~0.12 (3)	ND

*At focal point. ND - Not determined, yet.

2.2 Power measurement: Output power of each transducer was determined by measuring the amplitude of resonant frequency spectrum using a broadband hydrophone placed at the transducer focal point in water, Fig. 1. Test transducers were excited by Ultrason Burst Pulser BP 9400A. Its settings for 0.5 and 1.0MHz transducers are shown in TABLE II. It is important to note that at this time no special provisions were made to further increase the power of this system or to develop transducer matching networks.

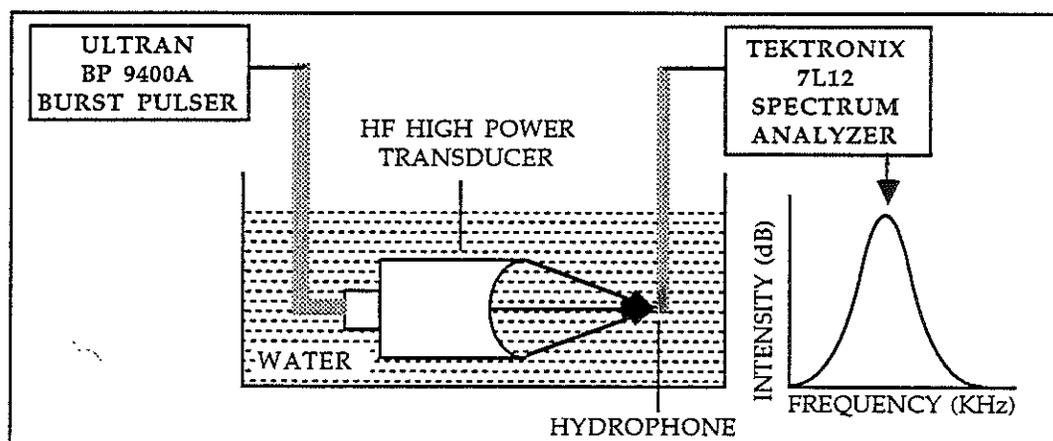


Fig. 1. Schematic layout of the setup used for transducer power measurement.

TABLE II. Characteristics of transducer excitation pulse for power measurement and for sonochemical experiments.

TRANSDUCER	BURST PULSER BP 9400A SETTINGS			
	PRF* (Hz/s)	PULSE WIDTH (μs)	PULSE SEPARATION (μs)	PULSES/BURST
KS150-0.5-P1.5-X	1000	1	2	9
KS150-1-P1.5-X	1000	05	1	9

*Pulse Repetition Frequency.

3. INITIAL EXPERIMENTS

3.1 Experimental Setup: Details of experimental setup are shown in Fig. 2. Initial experiments were performed by using 0.5MHz transducer excited by pulser settings shown in TABLE II.

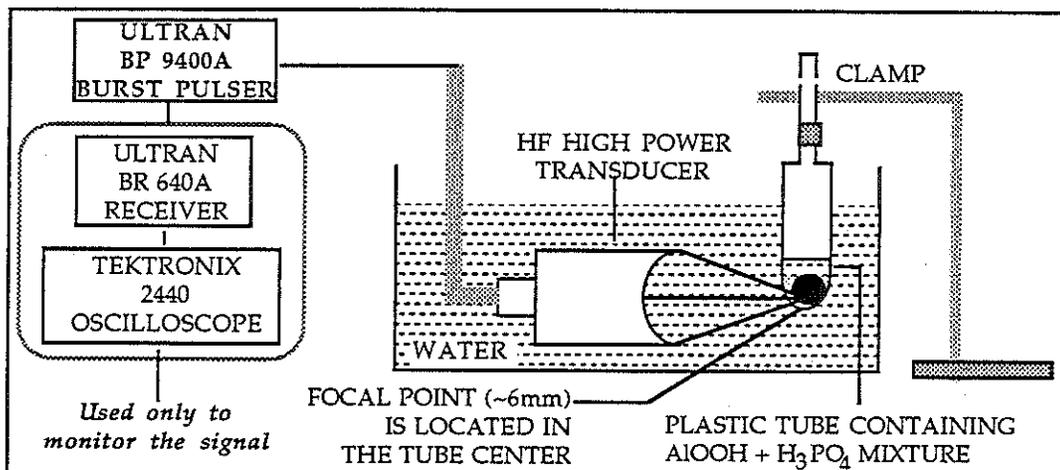


Fig. 2. Experimental setup used for sonochemical studies. Receiver and oscilloscope were used only to monitor the transmitted signal.

3.2 Observations and Results: As soon as the transducers for this work were finished, experiments were performed with an easily available ultrasonic excitation system at Ultrason. Inadequacy of this system for HF high power generation was known before these experiments were conducted. However, our curiosity played a more dominant role! Here are our initial observations.

A number of small batches containing AlOOH + H₃PO₄ were irradiated by 0.5MHz at various intervals of time. All samples were analyzed by x-ray diffraction in order to determine the formation of new compounds.

Although no new compounds were formed during the initial trials, reduced x-ray diffraction intensities and broadening of their peaks as a function of increased irradiation time suggest particle size reduction. X-ray diffraction data is shown in Fig. 3.

TABLE III. Results of initial sonochemical experiments on AlOOH + H₃PO₄ by 0.5MHz.

IRRADIATION TIME (minutes)	X-RAY DIFFRACTION RESULTS	
	PHASE	GRAIN MORPHOLOGY
80	γ-AlOOH	Similar to original grain size
120	γ-AlOOH	Reduction of grain size
900	γ-AlOOH	Further reduction of grain size

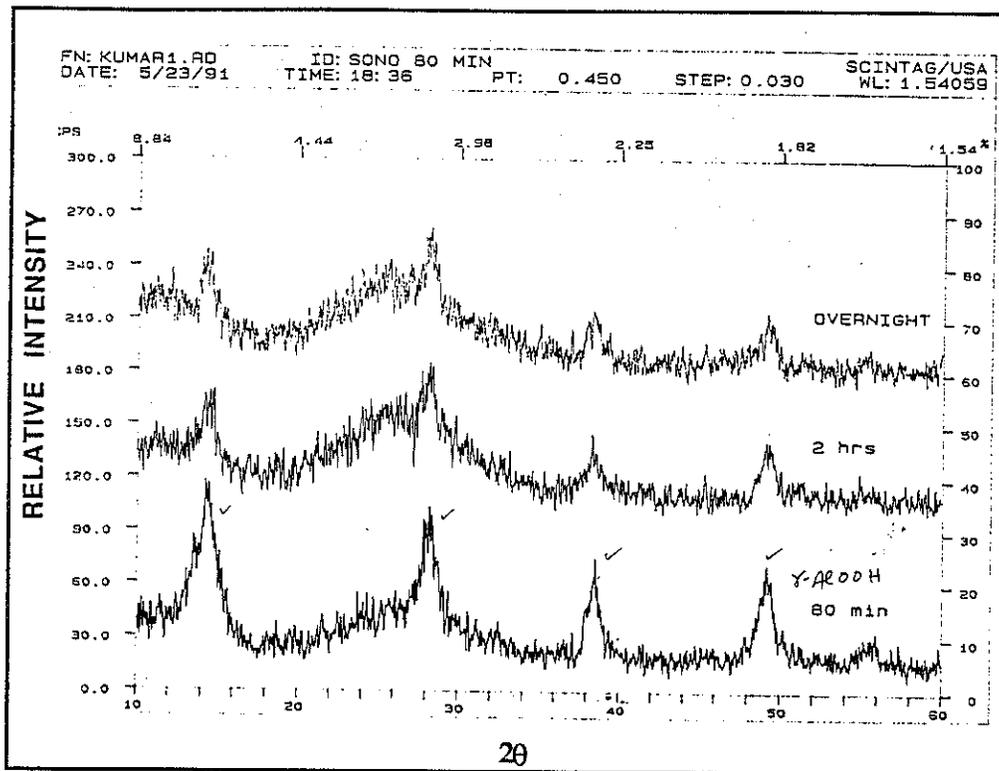


Fig. 3. X-ray diffraction data of $\text{AlOOH} + \text{H}_3\text{PO}_4$ irradiated with 0.5MHz at ~100mW power ultrasound as a function of time. See TABLE III for details.

BOTTOM TRACE: 80min
MIDDLE TRACE: 120min
TOP TRACE: 900min

4. WORK IN PROGRESS

- 4.1 Matching networks for two transducers.
- 4.2 Application of higher energy system/s for the transducer excitation.
- 4.3 Combination of #4.1 and 4.2 will be used to irradiate HF high power ultrasound in $\text{AlOOH} + \text{H}_3\text{PO}_4$ and Rhone-Poulenc $\text{Al}_2\text{O}_3 + \text{H}_3\text{PO}_4$ systems at various time intervals.

End Narrative
MCB: cbm