

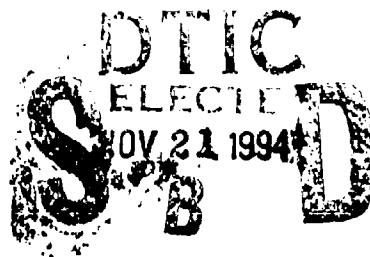
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Applications of Sonochemistry

George J. Kavarnos
Robert S. Janus
Submarine Sonar Department

Harold C. Robinson
Environmental and Tactical Support
Systems Department



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Newport, Rhode Island

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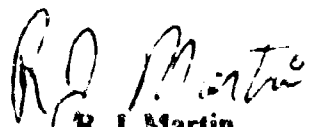
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PREFACE

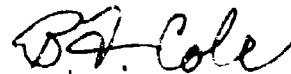
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R. J. Martin
Head, Submarine Sonar
Department (*acting*)



B. F. Cole
Head, Environmental and Tactical
Support Systems Department

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13 ABSTRACT (Maximum 200 words) Application of ultrasound can greatly enhance physical and chemical processes. Through the mechanism of cavitation, tremendous amounts of energy are released which can enhance chemical reaction rates, for example. This report reviews several experimental studies of sonochemistry applications with dual-use technology implications. A discussion of potential dual-technology applications, including production of higher powered ultrasonic processors, is presented.				
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APPLICATIONS OF SONOCHEMISTRY

INTRODUCTION

It has long been established that the application of ultrasound can often greatly enhance physical or chemical processes. In the area known as "ultrasonics" or "ultrasonic engineering," acoustic energy is used to change the physical characteristics of some object in the acoustic field. Some examples of these ultrasonic processes are ultrasonic cleaning, electroplating, and production of foams and aerosols.¹ The area known as "sonochemistry" deals with the effects of acoustic energy on chemical reactions. Insonication can increase the rate of chemical reactions, cause reactions to occur under less restrictive conditions, reduce the number of steps required in a reaction, enhance catalyst efficiency or initiate stubborn reactions.² Examples of sonochemical reactions are the sonolysis of water producing hydrogen peroxide, degassing of liquids and hydrolysis of oils in sodium hydroxide.³ Bridging the gap between these two seemingly divergent areas is the emulsification of immiscible liquids and ultrasonic homogenization.

The primary mechanism for enhancing these processes is cavitation of the fluid, which is produced when an ultrasonic wave of sufficient intensity passes through it. During the cavitation process, bubbles are formed in the fluid around "defects" (microbubbles already present, dust, cracks in the wall of the vessel, etc.) known as nuclei. These bubbles grow and collapse in response to the acoustic wave. A tremendous amount of energy is released during the bubble collapse, energy which is highly localized and, therefore, extremely intense but of very short duration. Since there are potentially millions of bubbles present in the liquid, the cumulative effect of all these bubble collapses can be significant. There are many factors that affect the onset and efficiency of cavitation, and, hence, of the sonochemical reaction, including^{1,3}

- Presence of gas in the liquid--increases number of nuclei,
- External pressure--increased acoustic pressure requires greater acoustic pressure,
- Viscosity and surface tension of the fluid--higher viscosities require higher pressures,
- Temperature--raising temperature lowers cavitation threshold,
- Frequency--increasing frequency shortens time between bubble creation and bubble collapse, decreasing ability to cavitate.

There are two types of bubbles.³ *Transient bubbles* collapse very violently within one or two acoustic cycles. This violent collapse leads to tremendously high localized temperatures and pressures. These high temperatures, for instance, are thought to be the primary cause of radical production to generate hydrogen peroxide. The shock waves due to high pressures enhance chemical reactivity by lowering the vapor pressure. *Stable bubbles* oscillate about some equilibrium size for many acoustic cycles before collapse. They provide lower temperatures and pressures than their transient cousins. They, therefore, have less dramatic but longer term chemical effects and increase reaction rates by increasing diffusion or collision of the reactants.

This study reviewed the application of ultrasound in several areas including

- Preparation of nickel surfaces for electroplating,
- Homogeneous generation of iodine,
- Production of ketals of fructose,
- Removal of paints, and
- Emulsification of oils.

The procedure used in each of these processes shall be presented. The relevant theoretical background will be given, and experimental insights into these processes will be outlined. Finally, an overall view of the future of sonochemistry for dual technology application will be given from the perspective of the transducer technology at the Naval Undersea Warfare Center (NUWC).

DISCUSSION

SONOCHEMICAL TREATMENT OF NICKEL SURFACES FOR ELECTROPLATING

One of the objectives facing the battery research program at NUWC, Detachment New London, is to scale up electrocatalysts used in aluminum hydrogen peroxide electrochemical systems. These electrocatalytic materials consist of a nickel (Ni) substrate coated with an electrocatalyst such as palladium (Pd) or iridium. The key step in this process is the electrodeposition of the metal onto a nickel film. Recent studies at this activity have underscored the need for improvement in the reproducibility and control of the electrodeposition process to achieve a stable and strong bond between the substrate and electrocatalyst. To explore this issue, a Special Initiative was undertaken to investigate the topography of nickel electrode surfaces electrodeposited with Pd using atomic force microscopy (AFM). During the course of this study, the decision was made to explore the effect of insonication on the integrity and structure of the nickel surface prior to electrodeposition. Previous work undertaken by Suslick *et al.*⁴ demonstrated that exposure to ultrasonic energy greatly enhanced the electrocatalytic power of Ni powder as a hydrogenation catalyst. Using Auger electron spectroscopy, Suslick demonstrated compositional changes in Ni samples before and after sonication. This study showed that the Ni surface is coated with a thick oxide layer. The oxide layer then can be removed sonochemically in octane in 1 hour. Removal of oxide probably takes place when solvent molecules bombard the surface to cleave Ni-O bonds. After surface cleaning in this manner, the oxide layer can be restored in air in about 15 minutes.

The experiments so far have focused on changes in the bulk topography of sonicated Ni on a micron scale before, during, and after electrodeposition. In this study, changes in the topography of the Ni surface were examined under various insonication conditions using the AFM, which has the unique ability to provide bulk and atomic-resolved topographic images of surfaces. The nickel foil samples were all prepared using the following nine-step process:

1. Wash with methyl ethyl ketone (MEK),
2. Abrade with 400 grit sandpaper,
3. Wash with distilled, deionized water,
4. Blot dry,
5. Wash with MEK,
6. Rinse with distilled, deionized water,
7. Place in 6.0 M hydrochloric acid (HCl) for 15 minutes,
8. Rinse with distilled, deionized water,
9. Air dry.

Prior to sonication, atomic force images revealed the characteristic morphology of nickel oxide (NiO). The nickel surface appears as an aggregate of 0.1 to 0.3 μ diameter particles. It is likely that these aggregates are nickel oxide. Controlled experiments were performed to determine the effects of various solvents on the nickel surface.

In order to reproduce Suslick's results, first the Ni sample was prepared as above, then the sample was insonified in n-heptane at 5-minute intervals for up to 15 minutes. Sonication took place in a Branson 2000 ultrasonic bath. AFM scans of the surface revealed that there was little or no change in the surface characteristics after sonication in n-heptane. The absence of visible bubbles in the heptane was an indication that the ultrasonic bath was probably not powerful enough to produce cavitation in the n-heptane, and, thus, no sonochemical effects were observed.

A second experiment compared Ni surfaces under different surface preparation conditions. A set of samples was prepared using the standard method outlined above. The second set was prepared in the same way, only the 15-minute HCl bath was sonicated in the Branson ultrasonic bath. The presence of bubbles in the HCl bath indicated that some cavitation was occurring. AFM images showed that the surface was smoothed considerably, and the characteristic NiO particles began to disappear. Therefore, it was concluded that sonication helped to prepare the Ni surface for plating better by removing more of the NiO than the standard method.

Finally, the Ni surfaces were electrodeposited with a Pd plating solution and monitored *in situ* with the AFM over long periods of time. It was determined that the sonicated Ni samples plated more uniformly and faster than the standard samples. This indicates that sonication prior to plating will enhance the plating process. It is still to be determined whether sonication *during* the plating process can be beneficial although intuitively this would seem to be the case. Sonication would effectively keep the plating solution continually stirred, so that "fresh" solution would always be in contact with the Ni surface. That notwithstanding, the potential of ultrasound in enhancing the electroplating of Ni electrodes has been demonstrated.

HOMOGENEOUS GENERATION OF IODINE

When performing sonochemical experiments, it is highly desirable to have a methodology for monitoring the acoustic cavitation, since that is the primary mechanism underlying numerous sonochemical processes. One particularly attractive diagnostic tool for monitoring sonochemistry in water is the starch-iodide indicator. The starch-iodide indicator takes advantage of the fact that during acoustic cavitation of water solutions, numerous reactive radical species are produced. The initial reaction is the dissociation of water to produce hydrogen and hydroxyl radicals, i.e.,



Given their reactive nature, hydrogen and hydroxyl radicals are short-lived species and rapidly undergo transformations leading to stable chemical species, i.e.,





In the presence of an iodide salt, such as NaI or KI, H_2O_2 reacts with I^- to generate iodine vapor, I_2 :



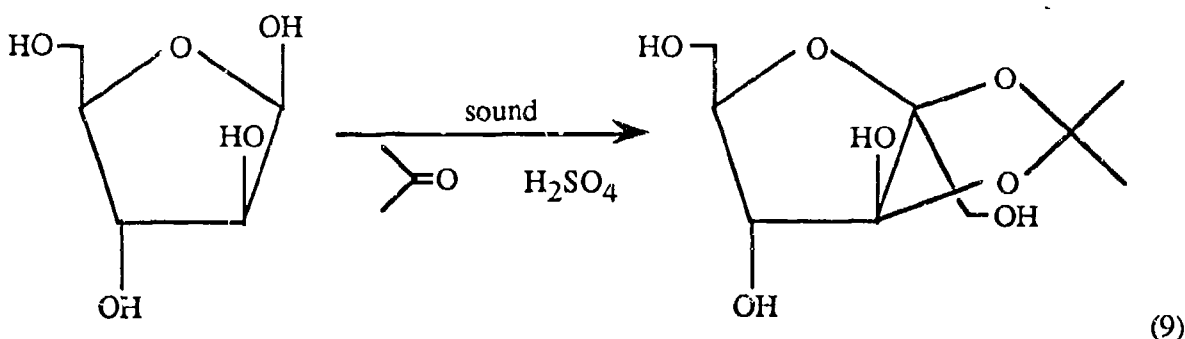
Since I_2 forms a blue color in the presence of starch, a starch-iodide solution can act as a convenient indicator for the cleavage of water to produce reactive radicals.

An experiment was performed to determine the ability of a VibraCell 20 kHz Ultrasonic Processor to cavitate water and, therefore, generate radicals. A 5% NaI solution containing about 0.5 gram powdered starch was prepared in a 250-ml beaker and subsequently sonicated. A few drops of CCl_4 were added to accelerate the cleavage of water. Within minutes, the characteristic blue color was detected, providing evidence of the chemical reaction and, hence, of cavitation. A control experiment, with no sonication, did nothing.

This experiment can be regarded as significant from several perspectives. First, the starch-iodide indicator provides a rapid and reliable approach to measure the ability of an ultrasonic source to produce cavitation. This, of course, is a critical step when designing, testing, and evaluating ultrasonic transducers for acoustic cavitation applications. Second, there are several emerging applications where the ultrasonic production of reactive radicals will play a key role. For example, one potential dual use technology that NUWC Detachment New London may be considering is the *in situ* treatment and remediation of hazardous chemicals in ground water and the environment. Many hazardous chemicals possessing potentially dangerous carcinogenic and mutagenic properties are halogenated hydrocarbons such as PCBs, chloroform, dimethylene chloride, carbon tetrachloride, etc. Recent investigations have demonstrated that ultrasonic cavitation can modify the molecular structures of these substances.⁵ It is likely that the acoustic degradation of halogenated hydrocarbons involves the initial generation of reactive radicals outlined in reactions (equations) (1) to (7) and that the radicals subsequently can react with the halogenated molecule to initiate further chemistry. The final outcome of this chemistry may be the conversion of the hazardous chemical to a less toxic form. Experiments to verify and study these transformations are certainly in order, since much of the chemistry is unknown.

SONOCHEMICAL REACTION OF D-FRUCTOSE AND ACETONE

To demonstrate the concept of sonochemistry in the area of synthetic organic chemistry, D-fructose was sonicated in the presence of acetone.⁶ This reaction converts the sugar into an isopropylidene derivative, which is soluble in organic solvents. By carrying out this transformation, the chemist converts the D-fructose, which like most sugars is quite water soluble, into a ketal derivative that can easily undergo selective chemical reactions that cannot otherwise be carried out in aqueous solutions. The reaction of the furanose form of D-fructose and acetone can be written as



Powdered D-fructose was added to a mixture of acetone and sulfuric acid, and then the mixture was sonicated using the VibraCell 20 kHz Ultrasonic Processor. The processor was set to pulse for 10 seconds, rest for 10 seconds, and then repeat the cycle. This prevented the build-up of heat in the solution. After 1 hour, the reaction mixture was made alkaline with ammonium hydroxide, and the ammonium sulfate was filtered off. The ketal derivative was then extracted with hexane. During solvent evaporation, the ketal derivative crystallized. An infrared study of the crystals is consistent with a ketal structure. Control experiments are now in progress. For example, this reaction will be carried out using an ultrasonic bath for comparison with the ultrasonic probe. It is expected that the ultrasonic bath, due to its lower power levels, would have to be used over much longer time frames than the ultrasonic probe to produce the same amount of ketal. Variations of the sugar-ketone reaction will be attempted using cyclohexanone.

ULTRASONIC REMOVAL OF PAINT

Alkaline materials with a pH range of 13 or higher are known to make ideal detergents to remove paints and primers.⁷ To demonstrate this, a 5% KOH solution was mixed, and various types of paints and primers were immersed in it. First, a metal sample with primer (PRC #420 Marine) on it was placed in the KOH solution and sonicated for 5 minutes using the VibraCell processor. As a control, a similar sample was placed in water and insonified for the same amount of time. The water sample showed some evidence of surface pitting, but this was far from uniform. By contrast, the primer bubbled up in the KOH sample and could easily be scraped off even with a fingernail. This demonstrated that, while some removal was possible simply by the action of the cavitating solution, the combination of cavitation and chemical reaction in the sonicated KOH proved much more effective.

This was born out in the next experiment. A painted gray metal container, possibly without primer, was immersed in water and KOH. The container was first soaked in KOH with no sonication. There was no paint removal of any significance in this passive bath, and the paint could be scraped off only with difficulty. Next, the sample was placed in water and insonicated for 5 minutes. Some scaling and flaking of the paint occurred, but the majority of it was not effected. Finally, the sample was put in the KOH solution and sonicated for 5 minutes. All of the paint was dissolved, and there was even some evidence of pitting of the underlying metal. This result dramatically underlined the need for combining alkaline solutions with acoustic energy to obtain the best paint removal results.

Of course, the success of this method may depend on what type of paint you are trying to remove. A third trial, involving a polyurethane marine paint (PRC PR-475-S), was much less successful. Indeed, this material seemed to be impervious to sonication in the KOH solution. It is possible that the sample has an additional coating on it that prevented removal, or that stronger solutions or longer sonication times are required to make a dent in it. Nevertheless, the overall success of the paint removal experiment indicates that application of this method to commercial and Naval paint removal and surface cleaning applications is worth exploring.

EMULSIFICATION OF OILS

Emulsification is defined as the mixing of two otherwise immiscible liquid phases.³ Two of the more common emulsifications are salad dressings (oil and vinegar) and mayonnaise (oil and eggs). It is well known that when oil and water (or vinegar) are mixed, for instance by shaking, they tend to separate rapidly. Therefore one could say that this emulsion is unstable, i.e. the two components do not stay mixed after the mixing action is removed. Ordinarily, in order to prepare emulsions, a transfer catalyst is added at the beginning of the process, and stabilizers are added to maintain the mix afterwards. The use of power ultrasound can reduce or eliminate the need for both of these additional components.

Acoustic emulsification of an oil-water mixture is really a two-step process. In the first step,⁸ the interface between the two liquid phases is broken up by unstable interfacial waves, which lead to the formation of large oil droplets on the order of 70 μ . This instability will not be reached until the vibration displacement exceeds the critical displacement amplitude ϵ_c , where

$$\epsilon_c = \frac{4k(\eta_w + \eta_o)}{\omega_A(\rho_o - \rho_w)}, \quad (10)$$

where $k=2\pi/\lambda$ is the interface wavenumber, λ being the wavelength of the interface waves, η is the viscosity of the medium, ρ is its density and ω_A is the acoustic angular frequency. The subscripts 'o' and 'w' refer to oil and water, respectively. From equation (10) it is obvious that the critical displacement amplitude is smaller when the viscosity of the oil is low or if it is much denser than water. If this is not the case, only higher acoustic frequencies or higher amplitudes of vibration will cause the mixture to enter this first stage of emulsification. Once ϵ_c is exceeded, the resulting oil droplet size is proportional to the interface wavelength λ .

In the second step or stage,⁹ these relatively large oil drops are broken up further by the cavitation shock waves due to the acoustic field. The droplets are continually bombarded by these shock waves, which effectively excite the surface motion of the droplet beyond a critical velocity. When this velocity is exceeded, the droplet breaks up into smaller droplets. This breakup depends on the dynamic pressure produced by the cavitation shock wave, the initial droplet diameter, and the surface tension. This process continues until the droplets reach a critical droplet diameter. Droplets with diameters smaller than this critical diameter do not undergo further breakup. Longer irradiation times will lead to more droplets at or below the critical diameter. The more droplets with the critical diameter, and the smaller the critical diameter, the more stable the emulsion will be.

Our experimental investigations concentrated on mixtures of transformer oil, castor oil, and hexanes with water. The process for all these materials was the same, i.e.,

1. Place water and water indicator in beaker,
2. Put ultrasonic processor probe into water,
3. Set processor amplitude and running time (typically 30 seconds),
4. Start processor,
5. Pour oil/hexane into water near probe,
6. Transfer emulsion to graduated cylinder to monitor separation.

After processing, the mixture typically was three phased. A thin layer of pure oil sometimes sat on the emulsion, followed by a frothy layer. The majority of the mix was emulsion and exhibited characteristics of neither the pure oil nor the water. Finally, a layer of pure water sometimes sat at the bottom of the cylinder. There was no evidence of a frothy transition layer between the emulsion and the water.

Transformer oil is not very viscous and has a density comparable to that of water. One trial which was performed was varying the amplitude generated by the VibraCell processor. It was found that, even at 20% of the maximum amplitude, a significant degree of emulsification occurred. These emulsions were very stable, and the degree of stability increased with increased power. The 20% trial required nearly 5 hours to separate, whereas the 40% trial had not separated even after 3 days. However, evidence of "creaming,"⁹ or frothy layering on top of the emulsion, was present in all the samples. It was found in an earlier trial that if the oil was simply sitting on top of the water before sonication, emulsification did not occur until at least 50% power was achieved. This is consistent with equation (10); the viscosity and density characteristics of the transformer oil dictated that a certain amplitude was required to destabilize the interface. Pouring the oil into the water destabilized the surface from the beginning, leading to emulsification at lower powers.

Castor oil has much higher viscosity than transformer oil. Therefore, one would expect, from equation (10), that higher power would be required to begin the emulsification process. This was born out experimentally. No emulsification occurred until a 40% drive amplitude was obtained. This transition (from no emulsification to emulsification) seemed to be quite sharp. The castor oil emulsifications were more stable than the transformer oil trials, possibly due to the greater viscosity.

Hexanes were used as an intermediate case which would separate more rapidly than the two oils. The emulsification threshold was lower (30% power) than for castor oil, but this emulsion was not very stable and took about half an hour to separate completely. At higher power (40%), a greater degree of emulsification occurred, but the mixture had nonetheless separated completely within 2.5 hours. In both trials, a frothy transition layer gave evidence of creaming. The dependence of emulsification on insonication time was not explored adequately to draw any conclusions.

CONCLUSIONS AND RECOMMENDATIONS

The sonochemical experiments described in this report represented an exploratory effort funded by a Bid and Proposal Special Initiative Request (Job Order 710B55) to investigate sonochemical processes. The use of acoustic energy to produce chemical changes is an emerging technology. The possible application of this technology to environmental cleanup strategies, oil-spill cleanup, surface modification of electrodes in battery research, and organic chemical and pharmaceutical synthesis was explored in this study. In the spirit of the exploratory nature of this endeavor, our efforts were directed towards several selected areas with potential dual-technology applications. The removal of an oxide layer from a nickel surface for improved electrodeposited electrocatalyst surfaces represents an application of critical importance for battery research. The fact that this technology can be exploited to improve material properties is significant. In addition, it was demonstrated that acoustic cavitation can be useful for *in situ* environmental cleanup of toxic chemicals and oil spills. Finally, acoustic energy, as shown in our laboratory, can be channeled to accelerate simple chemical transformations that otherwise are sluggish or require drastic or possibly dangerous conditions.

Sonochemistry has dual-technology ramifications. Some of these applications fit in nicely with the scientific and engineering expertise in transduction technology at NUWC, Detachment New London. It is recommended that this knowledge be directed to dual-technology efforts involved *in situ* environmental remediation and that funding be assigned to resolve several key issues that have been identified by our exploratory investigation. These issues are

1. To study the effects of frequency, bandwidth, and power on the efficiency and rate of the sonochemical application.
2. To study and delineate the details of the chemistry of the sonochemical application, i.e., degradation of halogenated hydrocarbons.
3. To investigate the feasibility of using towed arrays as sources for large-scale sonochemical applications, such as in oil-spill cleanup or ship/submarine scale paint removal.
4. To apply already existing transduction expertise to producing higher powered ultrasonic processors.

The first step in addressing issue 4 has already been taken. A medium powered ultrasonic processor was designed by Robert Janus of NUWC Detachment New London (Code 2131) using a small transducer, resonant at 20 kHz. A stepped aluminum horn was attached to the head mass of the transducer, and power was supplied to it using off-the-shelf amplifiers and power supplies (see figures 1 and 2). This processor was driven to 127 VRMS, producing electrical power of 32 watts before the amplifier overloaded. When placed in water, visual evidence of cavitation bubbles was present, indicating that even at this relatively low power, sonochemistry is possible. Further evidence of cavitation and the cleavage of water was provided by reproducing the results of the sodium iodide experiment detailed in the section page 3 (*Homogeneous Generation of Iodine*). The transducer itself is nowhere near its voltage limit, so larger powers, and, hence, greater processing capabilities, are achievable with the appropriate electrical equipment.

With this broad range of possible dual-technology applications and this activity's already existing transduction expertise, it would appear that the further investigation of sonochemistry would be both a fruitful and profitable enterprise and would be an ideal area to branch out into non-Defense related technology.

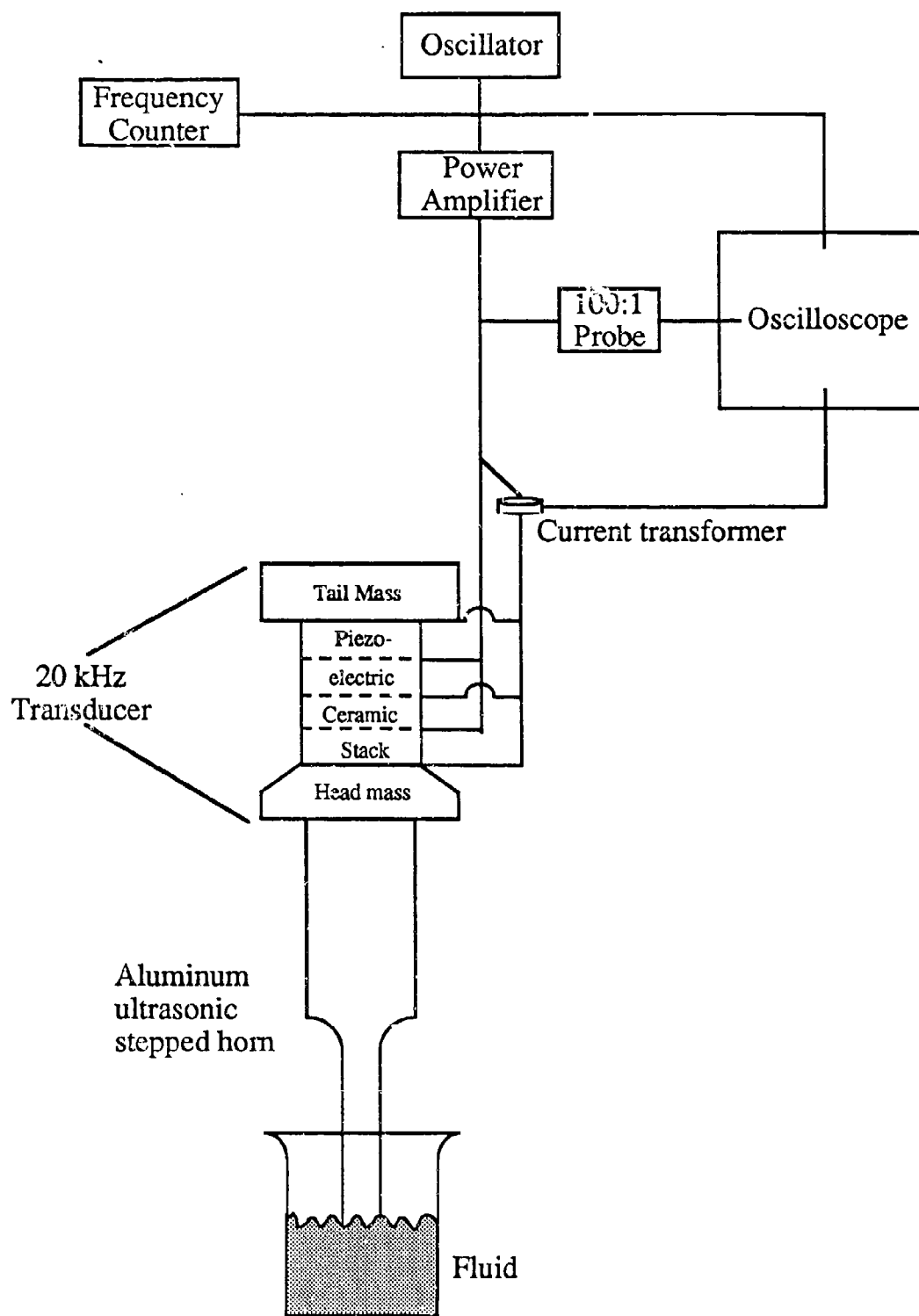


Figure 1. NUWC Ultrasonic Processor Schematic

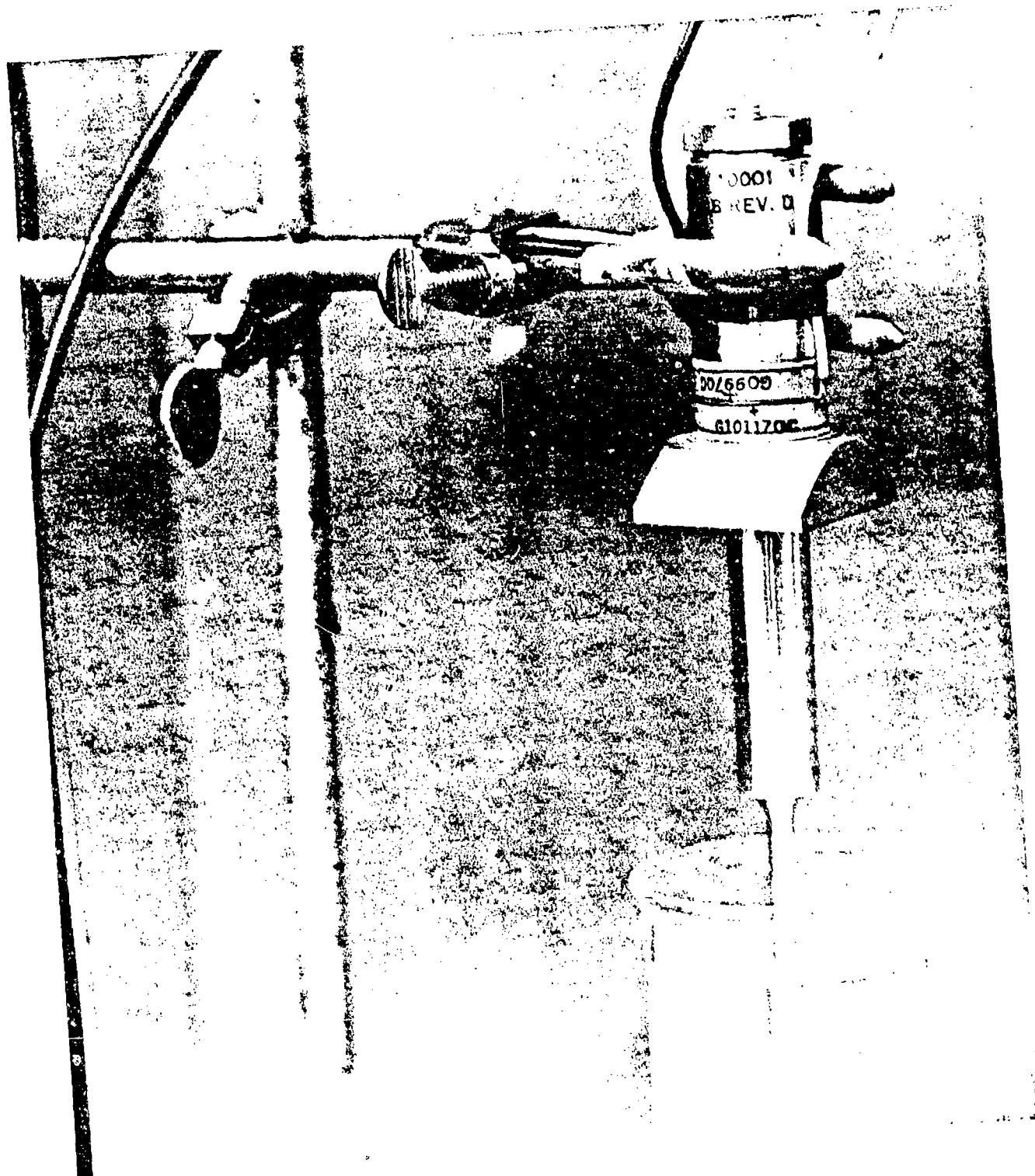


Figure 2. Photograph of NUWC Ultrasonic Processor

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