1995 NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM
JOHN F. KENNEDY SPACE CENTER
UNIVERSITY OF CENTRAL FLORIDA

510-71
7750
P. 29

CAVITATION EFFECTS IN ULTRASONIC CLEANING BATHS

Dr. Barbara H. Glasscock
Associate Professor
Chemical and Materials Engineering Department
California State Polytechnic University, Pomona
Pomona, California

KSC Colleague - Gale Allen
Material Science

Contract Number NASA-NGT-60002
Supplement 19

August 18, 1995

ACKNOWLEDGMENTS

I would like to express my sincere appreciation to the Office of Educational Affairs, NASA Headquarters, Washington, D.C. and the American Society for Engineering Education (ASEE) for their sponsorship of the 1995 NASA/ASEE Summer Faculty Fellowship Program at Kennedy Space Center. My summer research experience at KSC has been stimulating and rewarding and I know that I will carry with me to my home university many of the new ideas I have gained here.

The program was well organized and ran smoothly, thanks to the efforts of Dr. Ray Hosler and Ms. Kari Stiles, both of the University of Central Florida and to Gregg Buckingham of NASA/KSC.

A very warm thanks to all of the folks in the Materials and Chemistry Branch at KSC for their help enabling me to accomplish all that I did. Gale Allen, my NASA Colleague, was very supportive and often helped remove road blocks that appeared along the way. Bryan Feddersen, a co-op student from Georgia Tech, worked closely with me on this project, and was a valuable colleague. The SEM photos which were so revealing and intriguing were taken by Virginia Cummings and Stan Young. X-ray photoelectron spectroscopy is credited to Orlando Melendez. Laura Bales helped with the coupon acquisition and preparation. And many more thanks to the others at KSC with whom I interacted.

I am indebted to the folks at the Component Refurbishment and Chemical Analysis Laboratory at KSC operated by Wiltech of Florida. Bob LaVoie was the originator of the idea of using aluminum foil in a frame to measure the bath intensity and also conducted much of the coupon testing for us. Also thanks to Ken Fishell and Sam Kershner of Wiltech for their assistance and ideas.

I would also like to express my appreciation to my husband, John, for his support and willingness to relocate to Cocoa Beach for the summer so that I could pursue this work.

ABSTRACT

In this project, the effect of cavitation from aqueous ultrasonic cleaning on the surfaces of metal and non-metal sample coupons was studied. After twenty cleaning cycles, the mass loss from the aluminum coupons averaged 0.22 mg/cm^2 surface area and 0.014 mg/cm^2 for both stainless steel and titanium. The aluminum coupons showed visual evidence of minor cavitation erosion in regions of previously existing surface irregularities. The non-metal samples showed some periods of mass gain. These effects are believed to have minor impact on hardware being cleaned, but should be evaluated in the context of specific hardware requirements. Also the ultrasonic activity in the large cleaning baths was found to be unevenly distributed as measured by damage to sheets of aluminum foil. It is therefore recommended that items being cleaned in an ultrasonic bath be moved or conveyed during the cleaning to more evenly distribute the cavitation action provide more uniform cleaning.

SUMMARY

Aqueous ultrasonic cleaning has been proposed as a replacement at KSC for CFC solvents which deplete stratospheric ozone. In ultrasonics, cavitation bubbles are created by high frequency vibrations which can cause stresses at solid surfaces. The purpose of this study was to see if the effects from ultrasonic cavitation bubbles was severe enough to damage the surfaces of metals and non-metals being cleaned. If damage from ultrasonics was found, it would limit the applicability of this process in the intended use for cleaning of flight and ground support hardware at KSC.

Coupons of stainless steel, aluminum, titanium, Vespel™ and Viton™ were exposed to a full-scale ultrasonic cleaning process consisting of four ultrasonic baths. The first bath operated at 27 kHz and contained the degreaser Brulin™, the remaining baths all operated at 40 kHz. The second and fourth baths contained only deionized water and the third bath contained the surfactant Zonyl™. After twenty cleaning cycles, the mass losses from the metal were very low: 0.22 mg/cm^2 surface area for the aluminum coupons and 0.014 mg/cm^2 for both stainless steel and titanium. The aluminum coupons showed visual evidence of minor cavitation erosion in regions of previously existing surface irregularities. The non-metal samples showed some periods of mass gain.

The distribution of ultrasonic activity throughout the large cleaning baths was evaluated using sheets of aluminum foil spanning the entire widths of the baths. Aluminum foil is easily damaged by ultrasonics and the pattern of damage to the foil was used to map the areas of greater and lesser ultrasonic intensity. The sheets of aluminum foil were placed approximately 10" apart in the baths. The ultrasonic intensity in the baths was found to be far from uniform. The aluminum foil sheets in Bath A, operating at 27 kHz, showed a greater amount of damage than those in the other baths operating at 40 kHz. Within a given bath there was a great amount of variation on a single sheet as well as from adjacent sheets. These findings suggest that parts being cleaning in ultrasonic baths should be moved throughout the field to obtain the best cleaning efficiency. Evidence of the presence of standing wave patterns were observed on some of the aluminum foil sheets from two of the ultrasonic baths, B and CR, and the space between clusters of holes agreed with the theoretical value for the wavelength.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

I. INTRODUCTION

1.1 Background

Researchers at Kennedy Space Center and elsewhere have been working to develop replacement chemicals for chlorofluorocarbons (CFCs) because of the link between CFCs and the depletion of the protective layer of ozone in the stratosphere. For example, 1,1,2 trichloro - 2,2,1 trifluoroethane (CFC-113) was used at the Kennedy Space Center for precision cleaning of aerospace hardware. Many of the replacement cleaning systems that have been developed have focused on aqueous cleaning solutions which would be more environmentally acceptable.

One of the replacement cleaning systems includes the use of ultrasonics with aqueous solutions to enhance the cleaning. In ultrasonics, cavitation bubbles are created at the solid/liquid interface by high frequency vibrations. The stresses caused by the formation and collapse of the bubbles loosens debris from the surface, and the turbulence caused by the bubbles helps to transport the debris into the bulk solution. [Reference 1]

Ultrasonic cavitation can produce high temperatures and pressures that are very localized and are present for short durations of time. The stresses at the solid surface due to the cavitation are severe enough to potentially corrode the solid surface. A previous study [Reference 2] investigated the effect of pure water and ultrasonics on the surfaces of small precision parts for cleaning verification. Using Scanning Electron Microscopy and weight loss measurements, the study concluded that the corrosion effects and mass losses were insignificant for ultrasonic exposure periods of up to 2 hours.

An ultrasonic cleaning system proposed for cleaning of flight hardware at the Component Refurbishment and Chemical Analysis Laboratory (CRCA) at the Kennedy Space Center includes the use of surfactants, Brulin™ or Zonyl™, which reduce the surface tension of the water, enhancing the cleaning of small cavities. This system involving surfactants has not been evaluated as to its possible effects on the surfaces of the parts being cleaned.

1.2 Purpose

The purpose of this study was to evaluate the effects of cavitation from ultrasonic cleaning on the surfaces of metals and non-metal coupons. It will be necessary to assure minimal effects from ultrasonic cleaning before the process can be approved for cleaning of flight hardware. The purpose of the second part of this study was to evaluate the uniformity of the ultrasonics in the baths. The motivation for the uniformity study was to ensure that coupons being studied would be placed in areas of high ultrasonic activity.

II. MATERIALS AND METHODS

2.1 Ultrasonic Baths

The ultrasonic cleaning baths used in this study are used for cleaning of aerospace hardware at the Component Refurbishment and Chemical Analysis Laboratory (CRCA) located at the Kennedy Space Center. The baths are part of a cleaning process that has been developed as a replacement for a vapor degreasing process that used CFC-113.

Four large ultrasonic baths, as described in Table 2-1, are operated in series. The ultrasonic transducers are in the bottoms of the tanks. Parts to be cleaned are placed into baskets which rest on support racks, approximately eight to ten inches from the transducers. The baskets are moved from bath to bath after each ultrasonic cycle. The solutions within each tank are circulated through filtration systems to remove particulates. During actual cleaning operation, the parts may be placed into one of various chemical baths between immersion in Bath B and Bath C.

Table 2-1. Ultrasonic Bath Descriptions

Bath Number	Ultrasonic Power	Work Area (inches)	Volume (gal)	Solution	Temperature
A	9600 watts @ 27 kHz	65 x 36	260	Brulin 815 GD	150°F
B	7200 watts @ 40 kHz	54 x 36	205	D.I. water	150°F
C	4800 watts @ 40 kHz	48 x 28	160	DuPont Zonyl FSN	150°F
CR	3600 watts @ 40 kHz	36 x 28	120	D.I. water	150°F

2.2 Metal and Non-Metal Coupons

Two different shapes of coupons were used in this test: large rectangular coupons to simulate larger parts being cleaning, and small disks to simulate smaller parts. The coupon specifications are summarized in Table 2-2.

2.3 Coupon Preparation

The large coupons were previously engraved with identification numbers. The small disks were labeled with a handheld engraving tool, except the Viton™ disks which were identified with notches in the edges. Because this test was only concerned with cavitation effects and not cleaning efficiency, it was desirable to start with clean coupons in order to use weight changes as a measure of material loss. All of the metal coupons were cleaned in advance of the study by rinsing first with CFC-113 and then acetone.

Table 2-2. Test Coupon Specifications

	Material	Rockwell B Hardness (HRB)		Approx.
Large Coupons (rectangular)	Type	average	range	Dimensions (cm)
Aluminum	3003	20.7	14.6 - 23.8	3.7 x 6.5
Stainless Steel	304	85.1	84.3 - 85.7	4.0 x 6.3
Titanium	-	108.1*	107.2 - 108.6*	3.7 x 6.3
Small Coupons (disks)				Diameter (cm)
Aluminum	T6-6061	55.6	53.0 - 57.1	1.8
Stainless Steel	316L	97.2	95.7 - 99.4	1.8
		Shore durometer "A" scale		
Vespel™	-	97	96 -98	2.2
Viton™	-	79	76 - 82	1.8

* Note: Values of HRB over 100 are usually converted to Rockwell C scale.

2.4 Coupon Arrangement

Most of the large coupons were bolted to the washing baskets so that they would remain stationary, simulating the cleaning of large parts. The large coupons were fabricated with a hole (approximately 1 cm diameter) at one end of the rectangle, used for the bolting. Teflon washers were used to eliminate abrasion. Six large coupons were bolted to each of the baskets, designated α and β. One coupon of each material type (aluminum, stainless steel, and titanium) was positioned vertical and one horizontal in each basket. All of the small coupons plus one large coupon of each material were placed in a mesh basket with a lid that allowed some movement during the ultrasonics. Thus, a total of five large coupons of each material were tested and four small disks of each material. The different placements and orientations of the coupons were chosen to see if any of these factors played a role in the cavitation effects. Figure 2-1 shows the placement of the baskets within the ultrasonic baths. These locations were chosen because they represented areas of relatively high ultrasonic activity as described in Section 3.2.1. Care was taken to always place the coupons in the same locations on the baskets, which was placed in the same locations in the baths. (The designations A6, A5, etc. shown in Figure 2-1 were used for the aluminum foil tests and will be discussed in Section 2.7.)

2.5 Coupon Test Procedure

The coupons were exposed to a number of ultrasonic cleaning cycles with evaluations performed after 1, 10, and 20 complete cycles. Twenty cycles was chosen to be a reasonable upper bound for the number of times that a piece of hardware would be returned during its lifetime to the CRCA for cleaning. A complete cycle refers to 3 minutes of ultrasonification at 150°F (65.5°C) in each of the four cleaning baths (A, B, C, CR). Before evaluation of the coupons, the coupons were further rinsed with deionized water and allowed to dry.

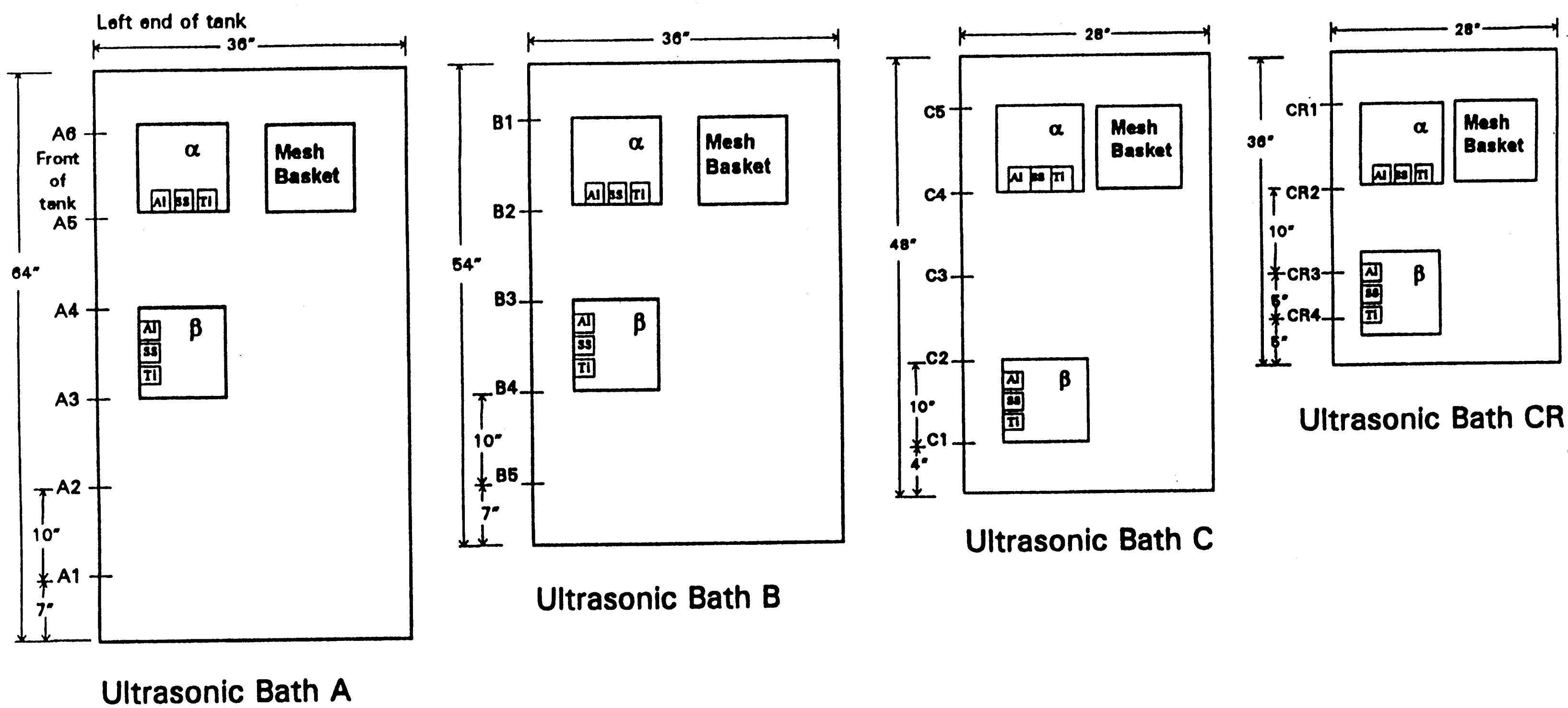

Figure 2-1. Placement of test coupon baskets in the ultrasonic baths (locations A6, A5,... were used for aluminum foil tests, see Section 2.7.)

2.6 Coupon Evaluation

Before the first ultrasonic cleaning cycle and after 1, 10, and 20 complete cycles, the coupons were weighed to evaluate for mass loss. One coupon of each material type was also analyzed using Scanning Electron Microscopy (SEM) and X-ray Photoelectron Spectroscopy (XPS). To facilitate viewing exactly the same microstructure on the metals using SEM, a microhardness mark was placed on the surface as a reference, each mark being 200 to 300 microns in length, depending on the hardness of the metal.

2.7 Aluminum Foil Test Procedure

In ultrasonic applications, strips of aluminum foil are sometimes used to evaluate ultrasonic intensity because of the ease with which the foil erodes. In this study rather than using small strips, large sheets of aluminum foil were used to obtain a full cross-sectional cut of the bath to develop a map of the activity in the bath. The sheets of aluminum foil were held taut in a frame such as those used to hold window screens. The sheets of aluminum foil were placed in the baths, one at a time, approximately 10 inches apart, for a duration of three minutes of ultrasonification. Figure 2-1 shows the placement of the foil sheets by the marks A6, A5, etc. The lower numbers (A1, B1, C1, CR1) indicate the inlet of the recirculated liquid.

2.8 Aluminum Foil Evaluation

After exposure to the ultrasonic baths, the aluminum foil sheets were examined visually for cavitation effects. Each 4" x 4" square of a sheet was given a rating as to the damage to the foil and then a map was compiled from the data. In addition, several localized areas from selected sheets were examined using the SEM.

III. RESULTS AND DISCUSSION

3.1 Coupon Tests

3.1.1 Material Loss

All of the coupons were weighed initially and after 1, 10, and 20 cleaning cycles. This data is presented in Table 3-1, along with the basket identification (α, β, or Mesh) and the orientation of the coupon (Vertical or Horizontal). The aluminum coupons lost between 0.05% and 0.11% mass after 20 cycles, depending on the alloy and coupon dimensions. If the data is expressed as (mass loss/surface area) then the data for both coupon types is very close with an average of 0.22 mg/cm^2. The surface area used is an apparent surface area using the area of just the two faces of the coupon. No difference in mass loss was observed for vertical versus horizontal orientation and only a slight difference between basket location.

The stainless steel and titanium coupons showed very low mass loss rates with average losses after 20 cycles of 0.0014% and 0.0019% respectively. When expressed as (mass loss/surface area) the stainless steel and titanium coupons lost an average of 0.014 mg/cm^2.

The behavior of the non-metal coupons was more complex. The Vespel™ gained mass after 10 cycles, presumably absorbing liquid, and then lost some of its mass between 10 and 20 cycles, but still ended above its original weight. The Viton™ coupons lost weight after 1 cycle but then appear to have absorbed liquid, gaining weight after 10 and 20 cycles. (Note that Table 3-1 reports mass loss and thus negative values are actually mass gain.)

The mass loss data is summarized in Figures 3-1 and 3-2 where the mass loss/surface area is presented graphically as a function of the number of cycles. The data for the two different types of aluminum have been combined and the data for the stainless steels and titanium have been combined.

3.1.2 Scanning Electron Micrographs

Scanning Electron Micrographs (SEMs) were taken of one coupon of each material type before exposure to the ultrasonics (initial) and after 1, 10, and 20 cycles. Very little change was observed in all cases. Figures 3-3 to 3-7 present the SEM's of the coupons in the initial conditions and after exposure to 20 ultrasonic cleaning cycles. In all of these photos, the diamond-shaped mark in the center is the microhardness mark that was used for reference enabling examination of exactly the same microstructure each time.

In Figures 3-3 and 3-4, some of the inclusions of the aluminum alloys appear more evident after exposure to the ultrasonics. The large stainless steel coupon in Figure 3-5 shows no damage from the ultrasonics with the only effect being perhaps that the surface appears cleaner. The small stainless steel coupon, shown in Figure 3-6, appears to have small trails of very fine dents which may have been caused by vibrations in the mesh basket. The titanium coupon showed no damage after 20 cycles as seen in Figure 3-7.

Table 3-1. Material loss due to ultrasonic cleaning.

sample #	Basket	orientation	Surface Area (cm^2)	weights (g)				mass loss (mg)			% mass loss			mass loss/S.A. (mg/cm^2)		
				Initial	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl
Aluminum - large coupons																
Al 5-01*	α	H	46.08	18.7562	18.7554	18.7499	18.7453	0.8	6.3	10.9	0.0043	0.0336	0.0581	0.017	0.137	0.237
Al 5-03	α	V	46.23	18.6673	18.6663	18.6611	18.6563	1.0	6.2	11.0	0.0054	0.0332	0.0589	0.022	0.134	0.238
Al 5-04	β	H	46.48	18.8794	18.8789	18.8742	18.8693	0.5	5.2	10.1	0.0026	0.0275	0.0535	0.011	0.112	0.217
Al 5-05	β	V	46.53	18.8520	18.8516	18.8469	18.8419	0.4	5.1	10.1	0.0021	0.0271	0.0536	0.009	0.110	0.217
Al 5-02	M	H	46.50	18.8749	18.8743	18.8687	18.8635	0.6	6.2	11.4	0.0032	0.0328	0.0604	0.013	0.133	0.245
						Al-large avg ->		0.7	5.8	10.7	0.0035	0.0308	0.0569	0.014	0.125	0.231
Aluminum - small coupons																
Al-A3*	M	H	4.98	1.0173	1.0173	1.0167	1.0163	0.0	0.6	1.0	0.0000	0.0590	0.0983	0.000	0.120	0.201
Al-A6	M	H	4.98	1.0185	1.0187	1.0180	1.0174	-0.2	0.5	1.1	-0.0196	0.0491	0.1080	-0.040	0.100	0.221
Al-12	M	H	4.98	1.0177	1.0176	1.0172	1.0166	0.1	0.5	1.1	0.0098	0.0491	0.1081	0.020	0.100	0.221
Al-17	M	H	4.98	1.0148	1.0148	1.0142	1.0139	0.0	0.6	0.9	0.0000	0.0591	0.0887	0.000	0.120	0.181
						Al-small avg ->		0.0	0.5	1.0	-0.0025	0.0541	0.1008	-0.005	0.110	0.206
Stainless Steel - large coupons																
SS 3-57*	α	H	47.41	60.0458	60.0461	60.0455	60.0453	-0.3	0.3	0.5	-0.0005	0.0005	0.0008	-0.006	0.006	0.011
SS 3-74	α	V	49.14	62.0991	62.0991	62.0991	62.0989	0.0	0.0	0.2	0.0000	0.0000	0.0003	0.000	0.000	0.004
SS 3-99	β	H	49.24	62.3486	62.3483	62.3481	62.3476	0.3	0.5	1.0	0.0005	0.0008	0.0016	0.006	0.010	0.020
SS 3-47	β	V	47.64	60.2772	60.2773	60.2770	60.2767	-0.1	0.2	0.5	-0.0002	0.0003	0.0008	-0.002	0.004	0.010
SS 3-92	M	H	48.89	61.8543	61.8537	61.8530	61.8524	0.6	1.3	1.9	0.0010	0.0021	0.0031	0.012	0.027	0.039
						SS-large avg ->		0.1	0.5	0.8	0.0002	0.0007	0.0013	0.002	0.009	0.017
Stainless Steel - small coupons																
SS-A1*	M	H	4.86	3.3842	3.3842	3.3841	3.3841	0.0	0.1	0.1	0.0000	0.0030	0.0030	0.000	0.021	0.021
SS-A6	M	H	4.98	3.4340	3.4339	3.4339	3.4339	0.1	0.1	0.1	0.0029	0.0029	0.0029	0.020	0.020	0.020
SS-A7	M	H	4.98	3.2874	3.2874	3.2874	3.2874	0.0	0.0	0.0	0.0000	0.0000	0.0000	0.000	0.000	0.000
SS-A9	M	H	4.86	3.2053	3.2053	3.2053	3.2053	0.0	0.0	0.0	0.0000	0.0000	0.0000	0.000	0.000	0.000
						SS-small avg ->		0.0	0.0	0.0	0.0007	0.0015	0.0015	0.005	0.010	0.010

Table 3-1. Material loss due to ultrasonic cleaning (cont).

sample #	Basket	orientation	Surface Area (cm^2)	weights (g)				mass loss (mg)			% mass loss			mass loss/S.A. (mg/cm2)		
				initial	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl	1 cycl	10 cycl	20 cycl
Titanium - large coupons																
Ti-29*	α	H	45.30	34.1122	34.1119	34.1114	34.1114	0.3	0.8	0.8	0.0009	0.0023	0.0023	0.007	0.018	0.018
Ti-20	α	V	45.65	34.2335	34.2335	34.2331	34.2330	0.0	0.4	0.5	0.0000	0.0012	0.0015	0.000	0.009	0.011
Ti-21	β	H	45.21	34.0092	34.0092	34.0089	34.0085	0.0	0.3	0.7	0.0000	0.0009	0.0021	0.000	0.007	0.015
Ti-13	β	V	46.00	34.2849	34.2849	34.2847	34.2846	0.0	0.2	0.3	0.0000	0.0006	0.0009	0.000	0.004	0.007
Ti-15	M	H	46.32	34.5995	34.5992	34.5990	34.5986	0.3	0.5	0.9	0.0009	0.0014	0.0026	0.006	0.011	0.019
						Ti avg ->		0.1	0.4	0.6	0.0003	0.0013	0.0019	0.003	0.010	0.014
Vespel - small coupons																
Vespel A1*	M	H	7.60	0.9109	0.9108	0.9126	0.9120	0.1	-1.7	-1.1	0.0110	-0.1866	-0.1208	0.013	-0.224	-0.145
Vespel A5	M	H	7.60	0.9055	0.9054	0.9071	0.9068	0.1	-1.6	-1.3	0.0110	-0.1767	-0.1436	0.013	-0.211	-0.171
Vespel A6	M	H	7.60	0.9078	0.9077	0.9095	0.9090	0.1	-1.7	-1.2	0.0110	-0.1873	-0.1322	0.013	-0.224	-0.158
Vespel A13	M	H	7.60	0.9116	0.9115	0.9133	0.9129	0.1	-1.7	-1.3	0.0110	-0.1865	-0.1426	0.013	-0.224	-0.171
						Vespel avg ->		0.1	-1.7	-1.2	0.0110	-0.1843	-0.1348	0.013	-0.220	-0.161
Viton - small coupons																
Viton 100*	M	H	4.86	0.7488	0.7481	0.7484	0.7484	0.7	0.4	0.4	0.0935	0.0534	0.0534	0.144	0.082	0.082
Viton 200	M	H	4.86	0.6805	0.6798	0.6800	0.6801	0.7	0.5	0.4	0.1029	0.0735	0.0588	0.144	0.103	0.082
Viton 300	M	H	4.98	0.7533	0.7526	0.7527	0.7528	0.7	0.6	0.5	0.0929	0.0796	0.0664	0.141	0.120	0.100
Viton 500	M	H	4.86	0.7289	0.7282	0.7283	0.7285	0.7	0.6	0.4	0.0960	0.0823	0.0549	0.144	0.123	0.082
						Viton avg ->		0.7	0.5	0.4	0.0963	0.0722	0.0584	0.143	0.107	0.087

* denotes SEM analysis done

Figure 3-1. Mass loss/surface area for metal coupons

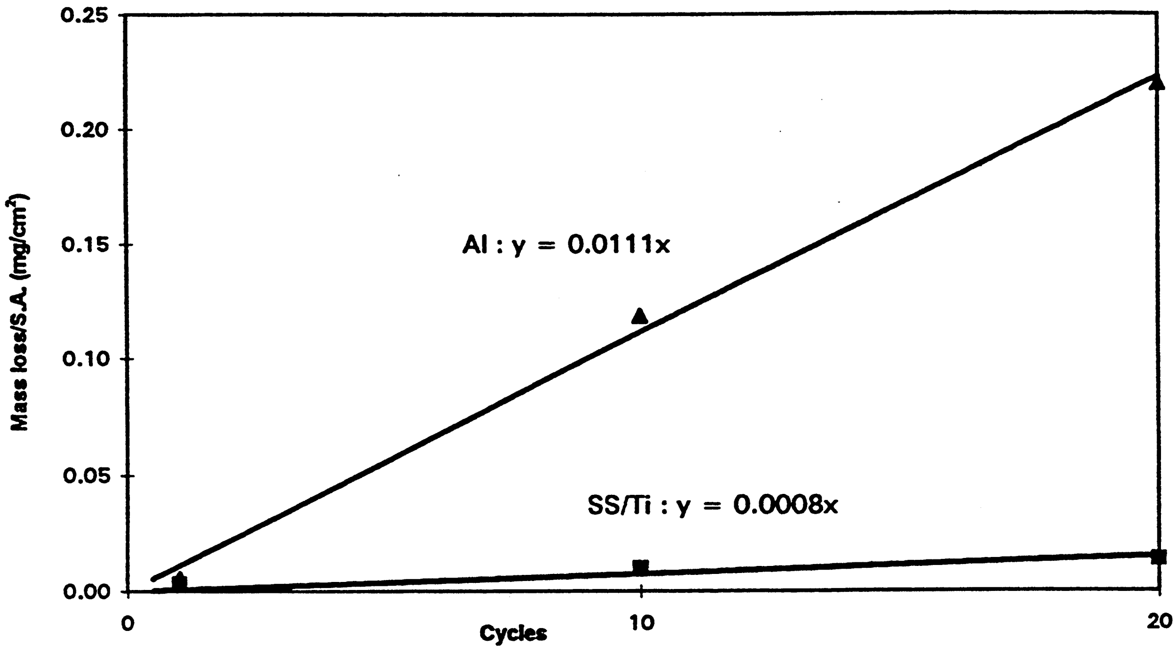

Figure 3-2. Mass loss/S.A. vs. cycles for non-metals

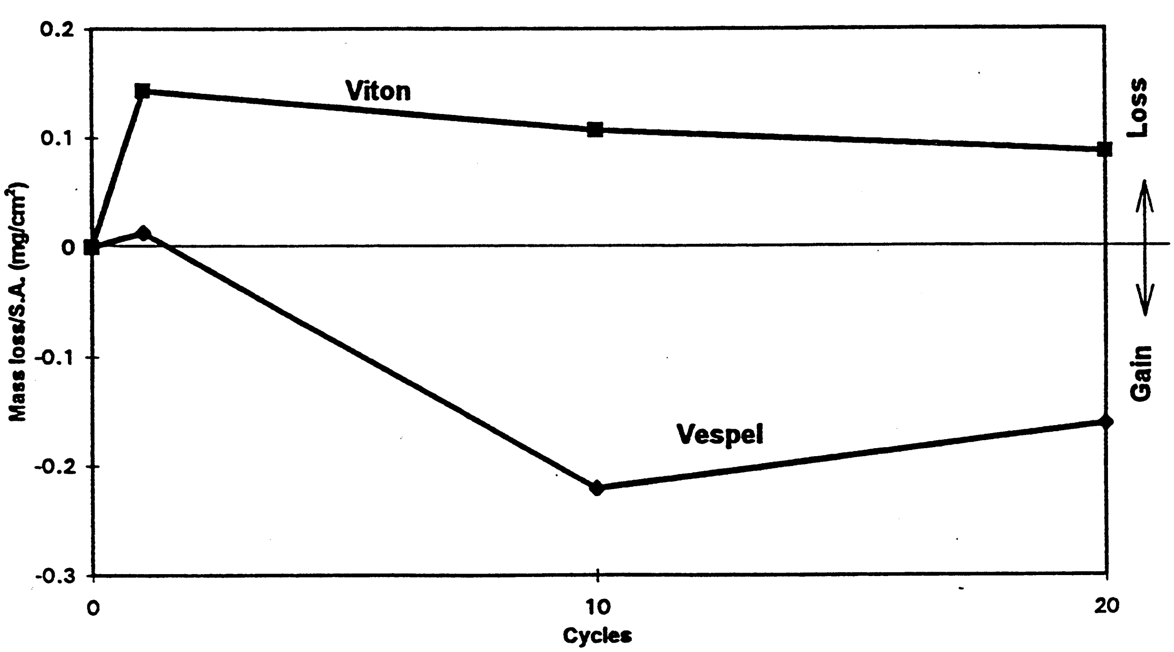

Figure 3-3. SEM of Aluminum (sample AL5-01) in its initial condition (left) and after 20 ultrasonic cleaning cycles (right). Microhardness mark in the center is for reference.

Figure 3-4. SEM of Aluminum (sample AL-A3) in its initial condition (left) and after 20 ultrasonic cleaning cycles (right).

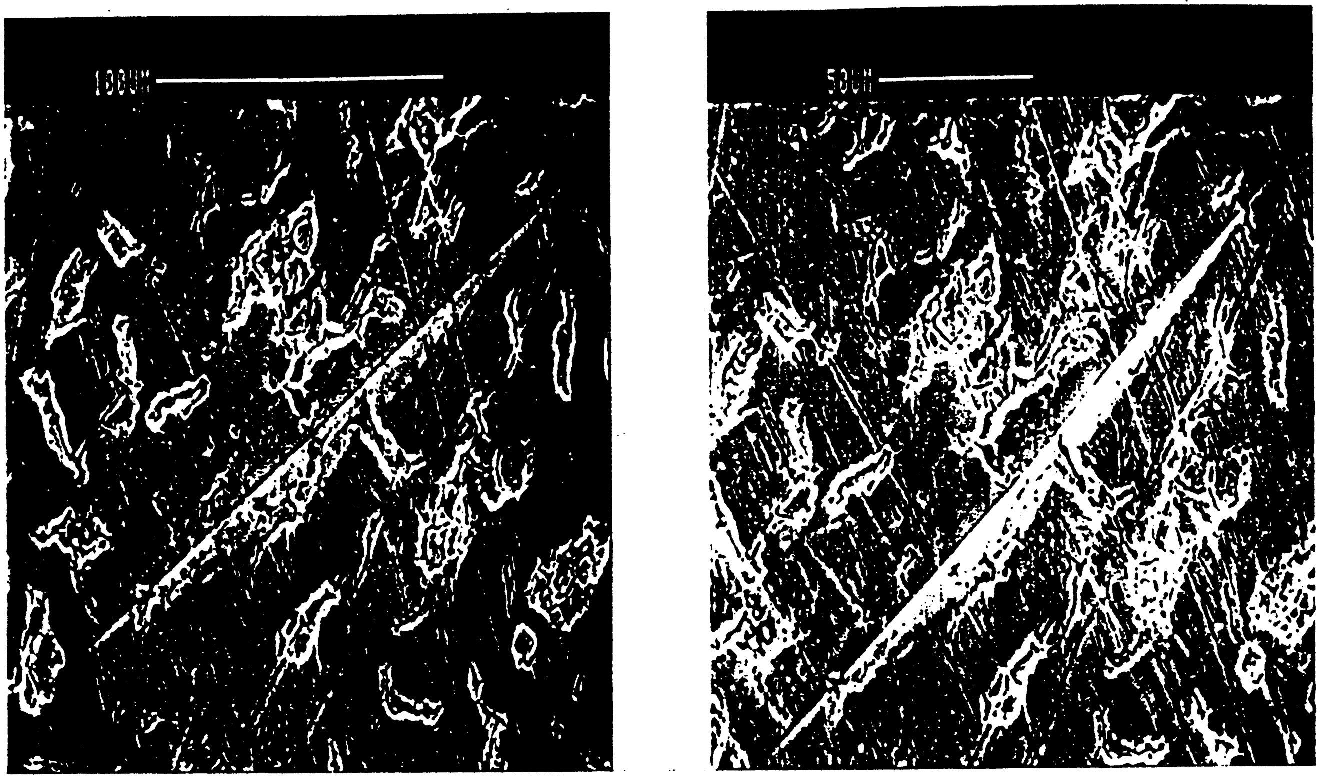

Figure 3-5. SEM of Stainless Steel (sample SS3-57) in its initial condition (left) and after 20 ultrasonic cleaning cycles (right).

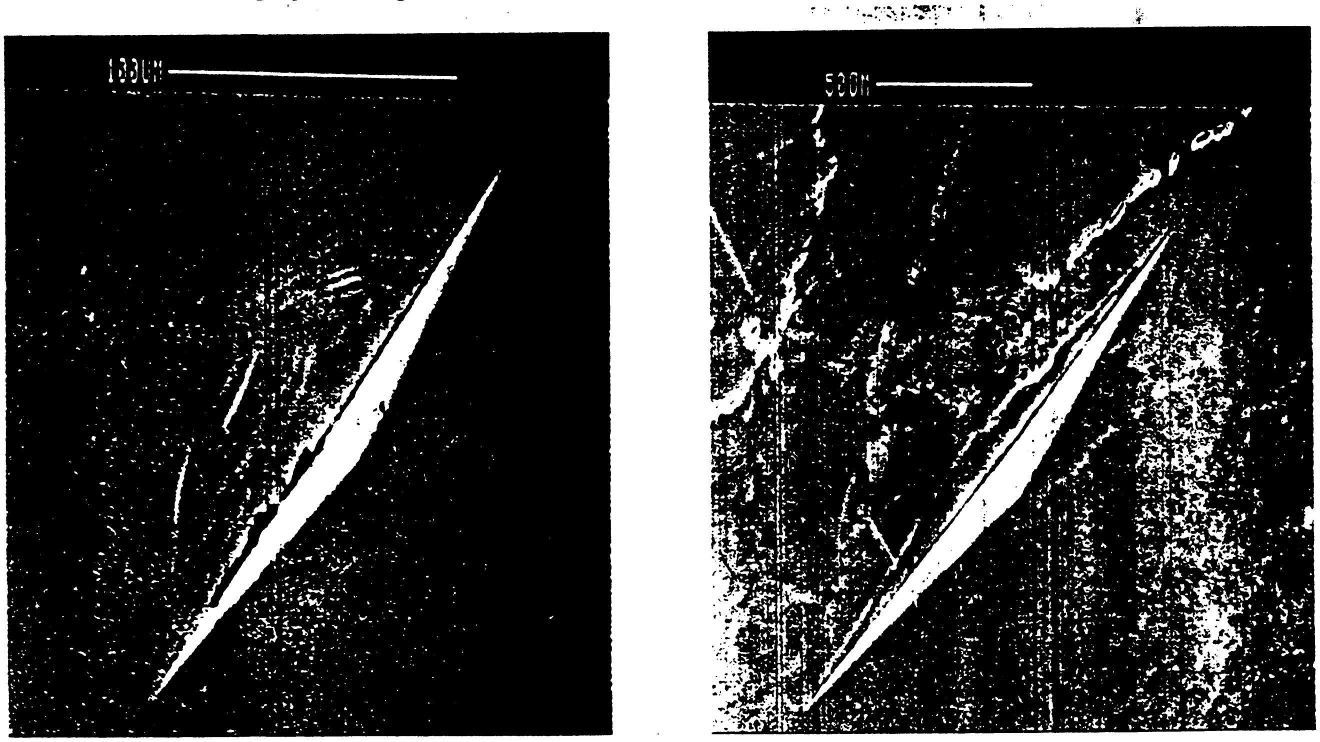

Figure 3-6. SEM of Stainless Steel (sample SS-A1) in its initial condition (left) and after 20 ultrasonic cleaning cycles (right).

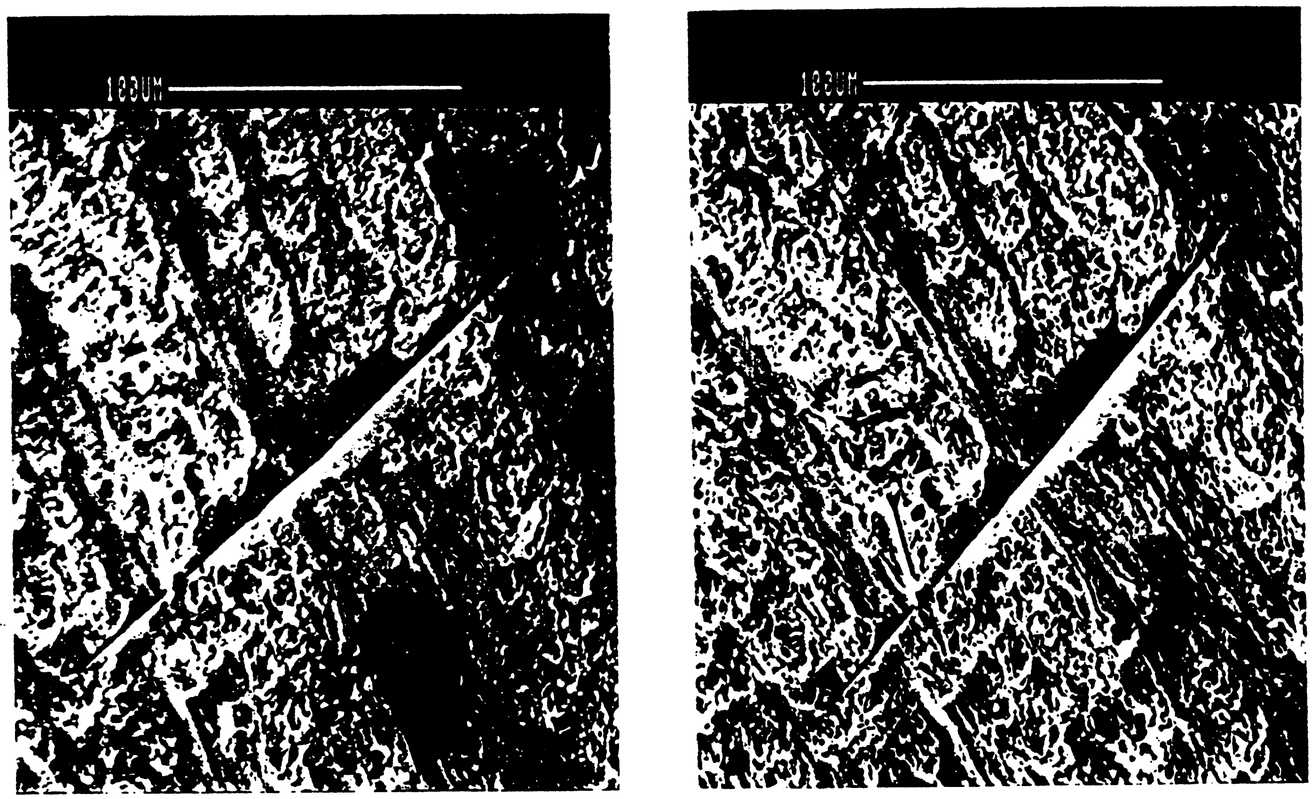

Figure 3-7. SEM of Titanium (sample Ti-29) in its initial condition (left) and after 20 ultrasonic cleaning cycles (right).

The aluminum coupons did exhibit an interesting phenomenon after 20 ultrasonic cycles. As shown in Figure 3-8, in the vicinity of surface irregularities, such as the engraved numbers, there were areas of cavitation damage to the surface, having the appearance of a "shadow". It is possible that the surface irregularities perturbed the local flow conditions and caused very localized cavitation. This phenomenon was seen only on the aluminum coupons.

The non-metal coupons were also viewed with an optical microscope and with the SEM but no surface changes were apparent.

3.1.3 X-Ray Photelectron Spectroscopy

One coupon of each material type was also examined using X-Ray Photoelectron Spectroscopy (XPS) initially and after 1 and 20 cleaning cycles. The XPS data is presented in Table 3-2. XPS examines only the top few atomic layers of the surface which includes a significant amount of ambient carbon. The large aluminum coupon (#AL5-01) and the large stainless steel coupon (#SS3-57) showed a reduction in the carbon levels after ultrasonic cleaning and an increase in the oxygen levels suggesting an increasingly oxidized surface or that the oxidized surface is being revealed by the removal of carbon. The other coupons were not analyzed after the twentieth cycle in a timely manor and may have picked up carbon again from the atmosphere.

3.2 Aluminum Foil Tests

3.2.1 Overall Evaluation

The aluminum foil sheets that were placed in the ultrasonic baths for a duration of three minutes were evaluated visually as to the extent of the damage caused by cavitation. Each sheet spanned the entire width of the bath (36" or 28") and the complete depth above the rack frame (18"). To summarize the damage, each sheet was divided into a grid of 4 x 4 inch squares and a number assigned to each square of 1 to 5 with 5 being the most severe damage. This data was then converted into gray-scale values and is presented in Figure 3-9. Each column of rectangles represents the family of sheets from one bath. Thus, the left-most column shows the six vertical slices from Bath A. The locations (A6, A5, etc.) were previously shown in Figure 2-1 and are approximately 10 inches apart.

As can be seen in Figure 3-9, the ultrasonic intensity of the baths was far from uniform. Bath A showed the highest intensity of the four baths which was not surprising since it was the only bath operating at 27 kHz. What is surprising is the variation in intensity within a given sheet, such as B1, or from sheet to sheet, such as B1 and B2. The foil sheet at B2 showed virtually no cavitation effects, yet it was only 10 inches from sheet B1 which showed the highest intensity in Bath B.

It is interesting to compare the intensity maps of Baths C and CR because both baths are operating at the same frequency (40 kHz) and the main difference between them is that Bath C contains Du Pont Zonyl™ FSN and Bath CR is a rinse bath containing only deionized water. Bath CR showed a much higher intensity than Bath C.

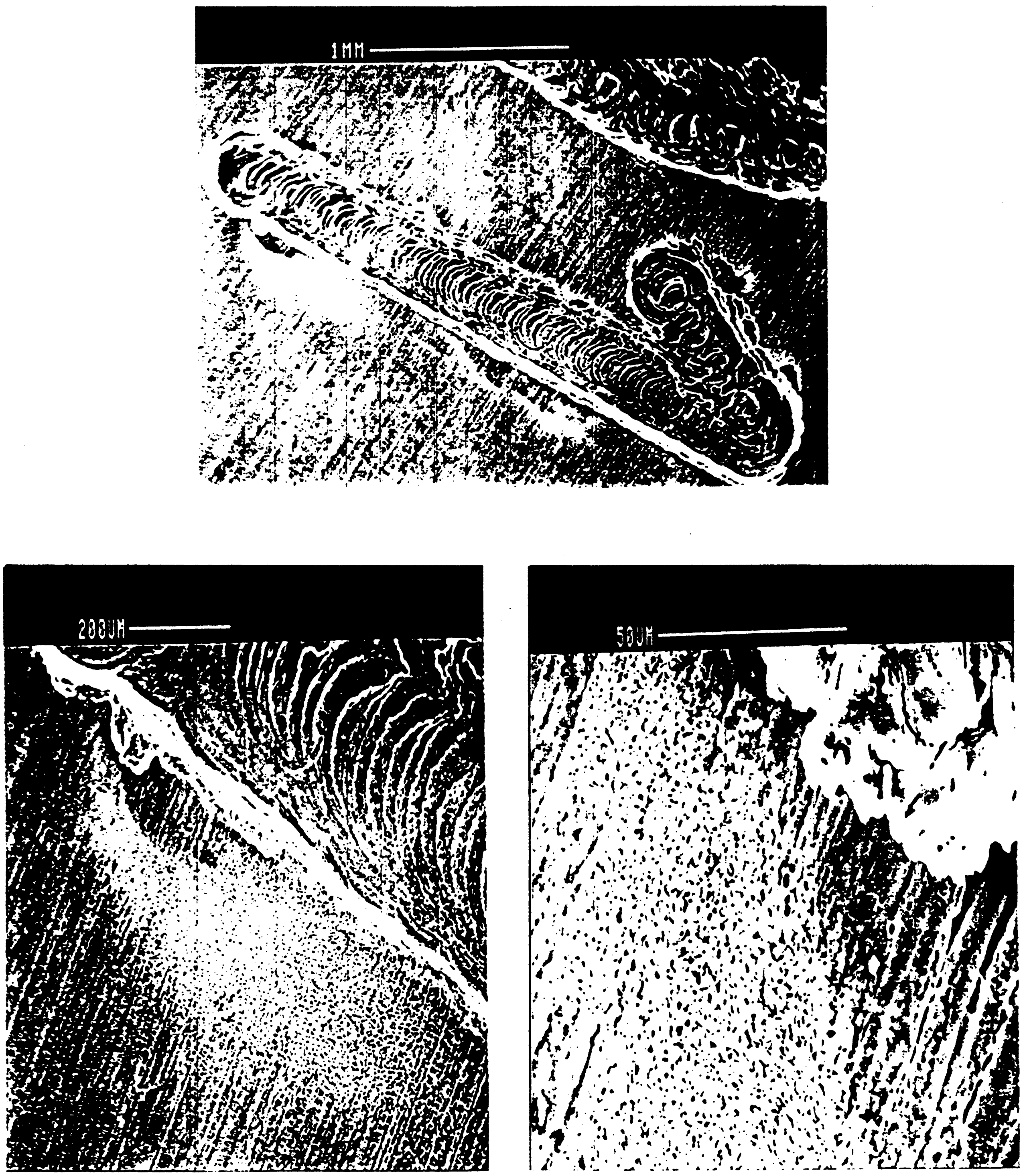

Figure 3-8. SEM of Aluminum (sample Al5-01) after 20 ultrasonic cleaning cycles showing cavitation damage in the vicinity of the engraved number "1".

Table 3-2. XPS Analysis of Coupons.

	Atomic concentration, %		
	Initial	1 cycle	20 cycles
Aluminum: AL5-01			
Carbon	30.75	22.39	21.21
Aluminum	18.97	21.40	20.37
Oxygen	50.28	54.28	58.41
Fluorine	0	1.94	0
Aluminum: A3			
Carbon	61.94	25.30	39.63
Oxygen	28.94	51.71	43.45
Aluminum	9.11	20.52	16.91
Fluorine	0	2.48	0.00
Stainless Steel: SS3-57			
Carbon	51.70	39.51	28.77
Oxygen	43.26	45.49	54.20
Chromium	5.04	7.29	10.00
Fluorine	0	4.34	6.37
Iron	0	3.37	0.66
Stainless Steel: A1			
Carbon	50.75	35.71	51.37
Oxygen	42.83	53.05	44.83
Chromium	3.43	5.70	3.80
Fluorine	0	4.00	0
Iron	2.99	1.54	0
Titanium: Ti-29			
Carbon	34.66	26.96	29.27
Titanium	10.77	15.19	21.15
Oxygen	53.50	57.32	46.11
Sodium	1.07	0	0
Lead	0	0.53	0.89
Fluorine	0	0	2.57
Vespel: A1			
Carbon	76.83	77.13	68.92
Nitrogen	4.69	4.68	4.41
Oxygen	15.09	13.77	19.59
Fluorine	3.40	4.43	7.08
Viton: V100			
Carbon	60.69	53.27	50.84
Oxygen	17.44	13.18	12.89
Fluorine	21.87	33.55	36.27

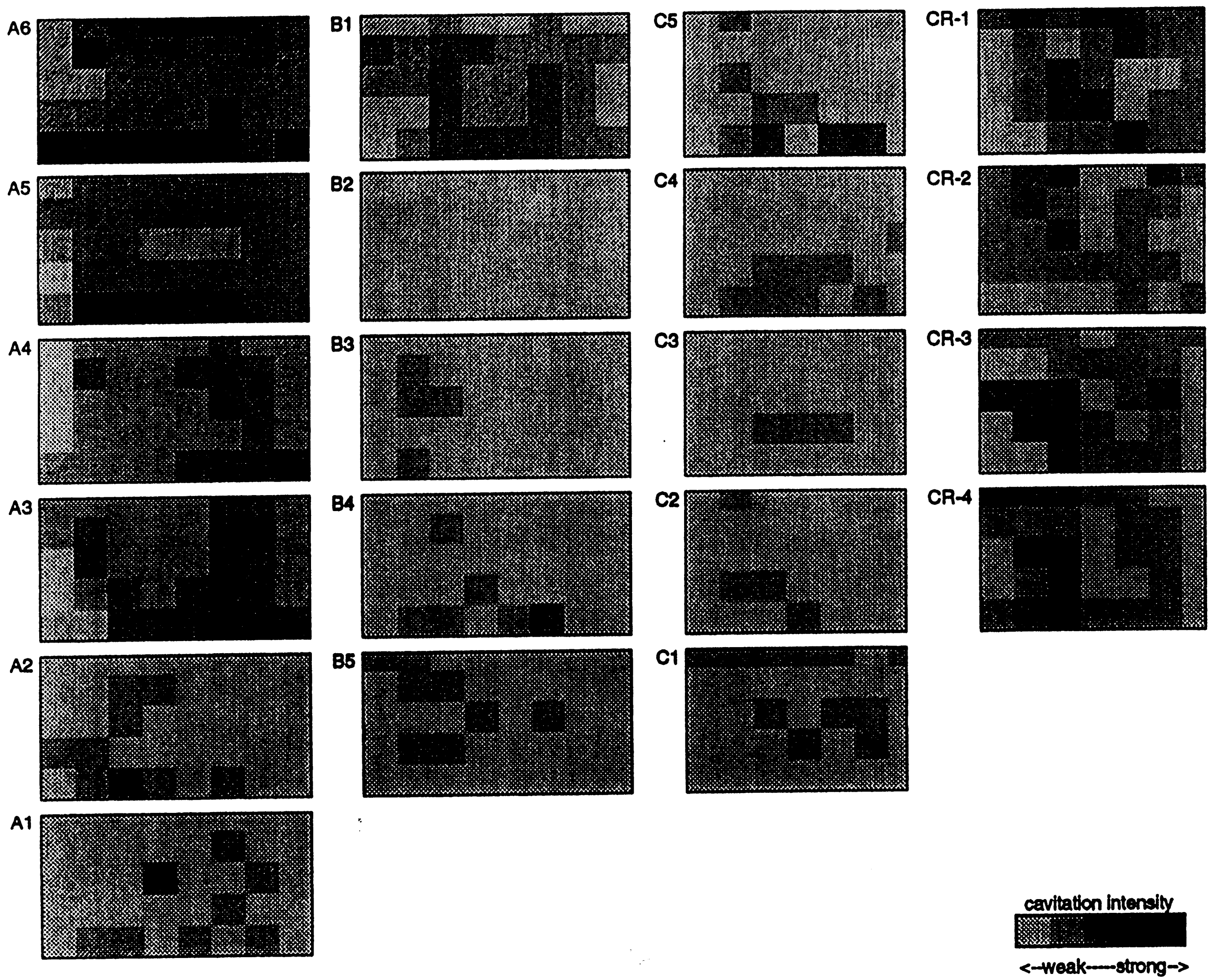

Figure 3-9. Ultrasonic intensity maps of baths as measured by aluminum foil cavitation damage. Each rectangle illustrates the damage to a sheet of aluminum foil placed vertically in the baths. For locations (A6, A5,...) see figure 2-1.

3.2.2 Localized Damage

The aluminum foil sheets, after exposure to the cavitation baths, were marked on the surface by dimples, dents, small and large holes, and crinkling. Figure 3-10 shows examples from sheet A5 of crinkling, which may have been caused by collapsed dents, and dimples which were often arranged in trails. In Figure 3-11 are seen examples of large dents on sheet A1 with small holes in the dents that were formed from both directions. Figure 3-11 also shows a magnified view of a crinkled area on sheet A3. Examples of small and large holes are shown in Figure 3-12. The single hole in the left frame of Figure 3-12 is approximately 320 microns in diameter and was caused by a single event such as a cavitation bubble bursting. The much larger hole in the right frame of Figure 3-12 (3 mm x 5 mm) was formed by the agglomeration of many smaller holes in close proximity with the small holes going in both directions.

Figure 3-13 shows an interesting phenomenon that was observed several times on sheet C5: a circular pattern of dimples, some of which have burst, and all are going in the same direction with the foil in the center of the circle forming a dome in the opposite direction of the dents. The sample was tilted at a 45° angle for the photograph. Additional examples of holes in the aluminum foil from sheet CR2 are seen in Figure 3-14. The small holes were about 100 microns to 200 microns in diameter with a larger hole again formed by the agglomeration of smaller ones.

3.2.3 Standing Wave Patterns

Evidence of the presence of standing wave patterns were observed on some of the aluminum foil sheets from two of the ultrasonic baths: B and CR. The other two baths, A and C, contained surfactants, Brulin™ and Zonyl™ respectively, which may dampen or disturb the wave patterns in some way. Figure 3-15 is a photograph of one entire sheet of aluminum foil (#CR3), measuring 18" x 28", illuminated from behind to show the pattern of holes. As can be seen in the figure, the holes are clustered together and the clusters are spaced in relatively regular intervals. The ultrasonic waves in the water should obey the following equation:

$$C = \lambda \, v$$

where C is the velocity of sound in water, λ is the wavelength, and v is the frequency. The velocity of sound in water is 1594 m/s at 65°C (149°F) [Reference 3] and the frequency in Bath B and CR is 40 kHz. Substituting these values into the above equation, one can calculate a theoretical wavelength of 40 mm. Measurements were taken of the spacing between several of the periodic clusters of holes on sheets from Baths B and CR, and assuming the spacing between two adjacent clusters is one-half a wavelength, experimental values of the wavelengths were obtained and are summarized in Table 3-3. As seen in Table 3-3, the agreement between theory and experiment is excellent.

Table 3-3. Ultrasonic Wavelength Comparisons

	λ = wavelength
Theoretical	40 mm
Experimental - Bath B	38 mm
Experimental - Bath CR	41 mm

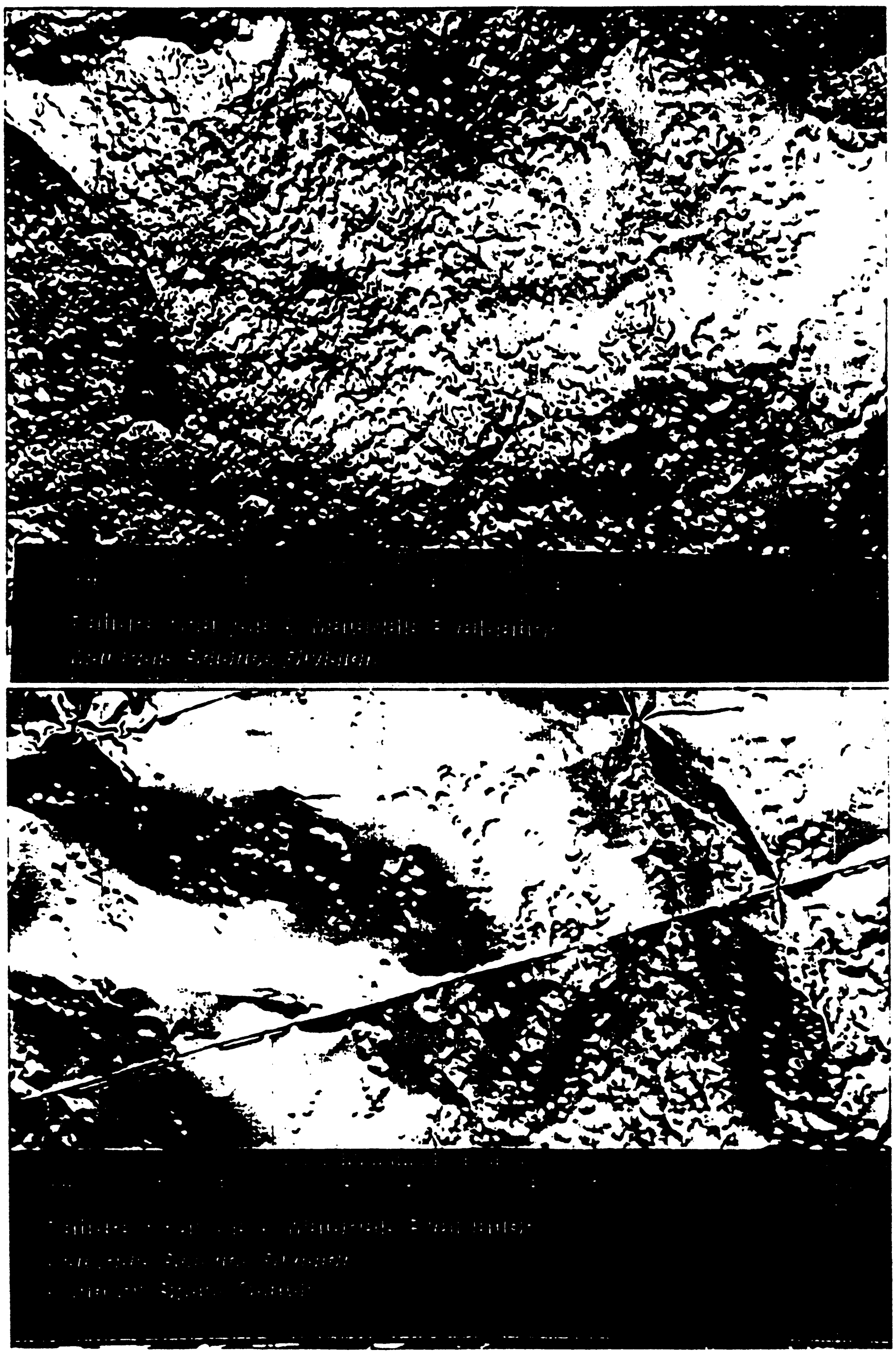

Figure 3-10. Examples of damage to aluminum foil #A5: crinkling (above), and trails of dimples (below).

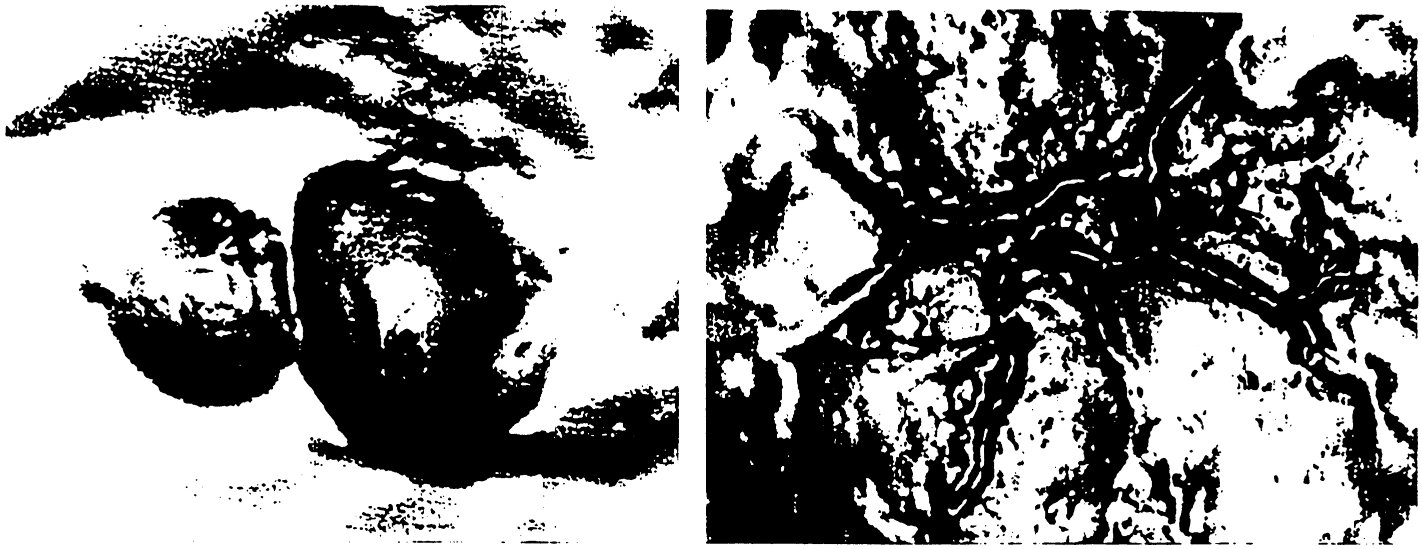

Figure 3-11. Examples of damage to aluminum foil: large dents with small holes in foil #A1 (left); crinkling on foil #A3 (right). Magnification is 6.3x.

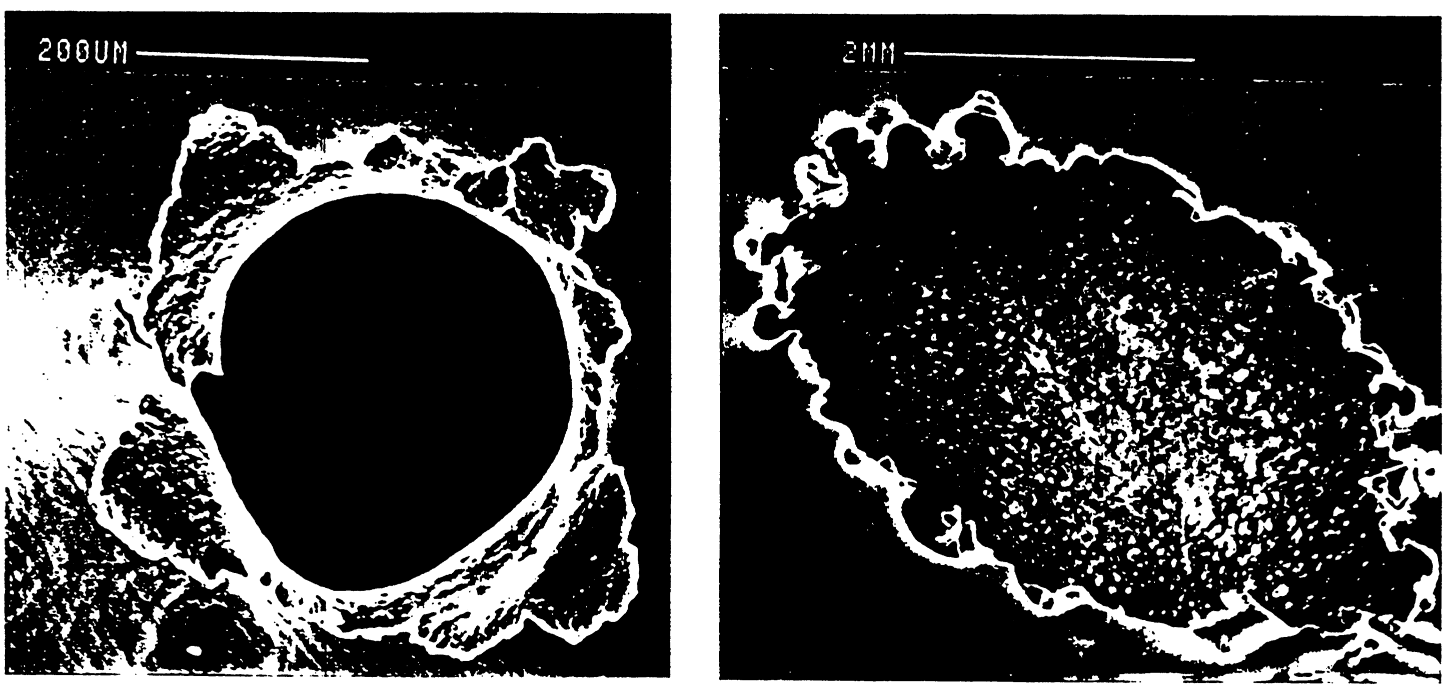

Figure 3-12. Examples of holes in aluminum foil #A3: single hole, diameter 320 microns (left); large hole, diameter 5 mm, resulting from numerous smaller holes (right).

Figure 3-13. Examples of holes in aluminum foil #C5: the holes and dimples are arranged in a circle (left); the holes and dimples are oriented in the same direction (right).

Figure 3-14. Examples of holes in aluminum foil #CR2: holes going in both directions (left); large hole formed by several smaller holes (right).

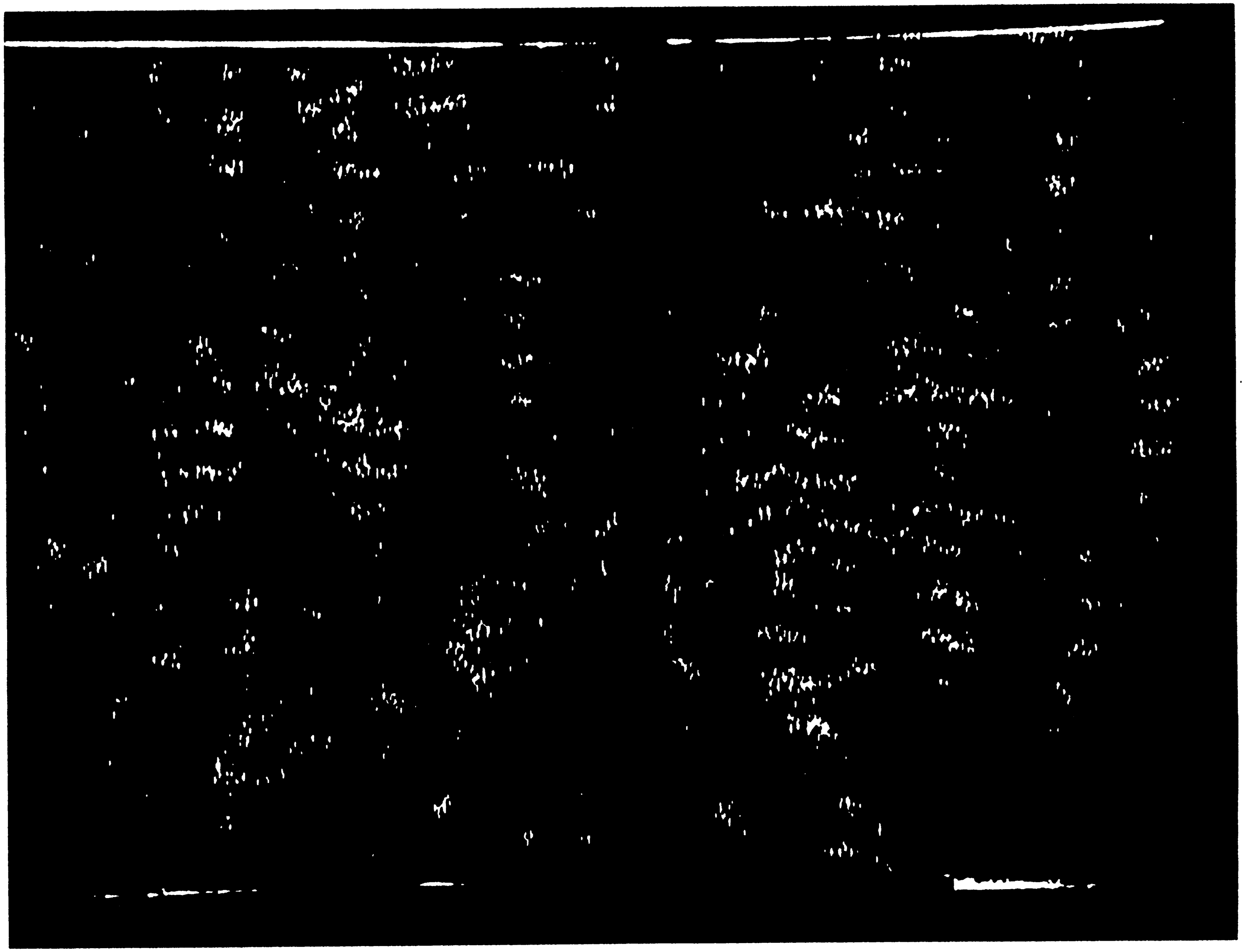

Figure 3-15. Backlight photograph of aluminum foil #CR3 showing hole patterns.

IV. CONCLUSIONS

The primary focus of this work was to evaluate the effect of the ultrasonic cleaning process at CRCA on the surfaces of metal and non-metal coupons. The mass loss rate from the metal coupons after 20 cycles of ultrasonic cleaning was very low: 0.11% or less for aluminum, 0.0019% for titanium, and 0.0014% for stainless steel. The non-metal coupons, Vespel™ and Viton™, showed some periods of weight gain as well as weight loss.

A secondary focus of this work was to characterize the intensity of the ultrasonic baths as a function of location. The primary motivation for this was to identify possible areas of low activity to be avoided in the coupon study. It was found that the ultrasonic baths were not uniform. The two baths that did not contain a surfactant showed evidence of standing waves that agreed with the theoretical value for the wavelength.

V. REFERENCES

1. Ensminger, Dale, Ultrasonics, Fundamentals, Technology, Applications, Marcel Dekker, Inc., New York, 1988.

2. Mehta, N.H., "Evaluation of Ultrasonic Cavitation of Metallic and Nonmetallic Surfaces", NASA/ASEE Summer Faculty Fellowship Program, NASA CR-191004, September, 1992, pp. 258-315.

3. Weast, Robert C., editor, CRC Handbook of Chemistry and Physics, 46th ed., The Chemical Rubber Co., Cleveland, Ohio, 1965, p.E-29.