N93-19400

1992 NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM

JOHN F. KENNEDY SPACE CENTER
UNIVERSITY OF CENTRAL FLORIDA

EVALUATION OF ULTRASONIC CAVITATION OF METALLIC AND NON-METALLIC SURFACES

PREPARED BY:	Dr. Narinder K. Mehta
ACADEMIC RANK:	Investigator
UNIVERSITY AND DEPARTMENT:	University of Puerto Rico - Mayaguez Department of Chemical Engineering College of Engineering
NASA/KSC	
DIVISION:	Material Science
BRANCH:	Microchemical Analysis
NASA COLLEAGUE:	Gale Allen
DATE:	August 2, 1992
CONTRACT NUMBER:	University of Central Florida NASA-NGT-60002 Supplement: 8

DEDICATION

This project is dedicated to my wife, Luz Maria, and to my precious flowers, Jyoti Marie, Tahnee Yngrid and Narinder Jr., who are always there when I need them.

ACKNOWLEDGMENT

I would like to thank NASA/ASEE Summer Faculty Fellowship Program, to Dr. Loren Anderson of the University of Central Florida, to Mr. Charles (Charlie) Hoppesch and Mr. William (Irby) Moore, Mr. Dennis Armstrong and Mrs. Carol Valdes of the Kennedy Space Center (KSC) for providing me with the opportunity to work at KSC for another summer. Thanks to Mrs. Kari Stiles for her willingness to help in all the administrative matters.

Special thanks are extended to Ms. Gale Allen, KSC project coordinator and Mr. Charlie Hoppesch for suggesting the project, and for their guidance and encouragement in all phases of the project.

There are many other professionals who assisted me during the course of this project. My sincere appreciation goes to Mr. Stan Young, Mr. Lee Underhill, Dr. Orlando Melendez, Mr. Mitch Buckley, Mr. Scott Johnson and Ms. Dionne Broxton for their assistance and advice throughout the project.

Special friendly thanks are extended to other professionals and summer participants working in the Microchemical analysis Laboratories who made me feel welcome, and for the pleasant interactions we all had while working in the laboratories.

The support of Dr. Felix Santiago, Dr. A. Rodriguez and Dr. Rafael Munoz of my home institute, the University of Puerto Rico is gratefully acknowledged.

ABSTRACT

The purpose of this summer project was to evaluate the ultrasonic cavitational effect on metallic and non-metallic surfaces, using commercially available ultrasonic baths operating at low frequencies and higher generator power output. Experimental data on the material loss, microphotographic and optical microscopic evaluation of the nozzle area for the three metal alloy specimens, and the hardness data for the non-metallic polymer disks were obtained to assess the applicability of the proposed replacement method of ultrasonic cavitation of the parts with water for the validation process. The results indicate that the proposed method can be utilized for validating cleaned small parts made of stainless steel, brass and non-metal based polymer disks. The method is equally applicable to anodized aluminum parts using moderate piezoelectric ultrasonic baths.

SUMMARY

1,1,2 trichloro-1,2,2 trifluoro ethane (CFC-113) commercially known as Freon-113 is the primary test solvent used for validating the cleaned hardware at the Kennedy Space Center (KSC). Due to the ozone depletion problem, the current United States policy calls for the phase out of Freons by 1995. NASAs chlorofluorocarbon (CFC) replacement group at KSC has opted to use water as a replacement fluid for the validation process since water is non-toxic, inexpensive, and is environmentally friendly. The replacement validation method calls for the ultrasonification of the small parts with water at 52 degrees Celcius for a cycle or two of 10-minute duration each using commercial ultrasonic baths. In this project, experimental data was obtained to assess the applicability of the proposed validation method for any damage of the metallic and non-metallic surfaces resulting from ultrasonic cavitation.

Commercially available ultrasonic baths operating at low frequencies and higher output power do not cause any surface cavitation of the stainless steel and the brass metal parts, and also to the non-metallic polymer disks commercially known as Nylon 6/6 and Vespel-21. Surface area of the anodized aluminum parts is found to be greatly affected by the ultrasonic cavitational effect of the powerful magnetostrictive type Branson and Blue Wave ultrasonic baths.

The experimental data obtained demonstrates that the CFCs proposed method can be utilized for validating cleaned small parts made of stainless steel, brass and the non-metallic polymer disks. The method is equally applicable to small parts made of anodized aluminum using moderate piezoelctric ultrasonic water baths.

TABLE OF CONTENTS

LIST OF ILLUSTRATION

EVALUATION OF ULTRASONIC CAVITATION OF METALLIC AND NON-METALLIC SURFACES

I-INTRODUCTION

1.1 OBJECTIVE:

To evaluate that ultrasonic validation of the cleaned metallic and non-metallic specimens with water as a replacement fluid for Freon-113 does not cause surface erosion of the cleaned surfaces.

1.2 BACKGROUND:

Small precision parts made of metallic and non-metallic materials in quantities are routinely used at the Kennedy Space Center (KSC). For a safe space shuttle launch, it is of utmost importance that they be verified clean of organic materials before use in an oxygen rich environment as that of the Orbiter.

Presently, 1,1,2 trichloro-1,2,2 trifluoro ethane (CFC-113) known as Freon-113 is the primary test solvent used for validating the cleaned hardware at KSC.

Freons including CFC-113 are known to remain in the atmosphere long enough to migrate to altitudes due to their high volatility, and are known to be a threat to the earth's protective ozone layer. The current United States Government policy calls for ending the production of these compounds, and phase out by 1995.

Due to the ozone depletion problem, KSCs chlorofluorocarbon (CFC) replacement group at the Material Science Laboratory is working to find a replacement for CFC-113 for the validation process of the cleaned parts.

Among the options, the CFC group decided to use water as the replacement fluid since water is non-toxic, non-flammable, environmentally friendly, inexpensive, and is compatible with other materials.

CFCs proposed replacement method for the validation calls for the ultrasonification of the small parts with water at 52 degrees Celcius for a cycle or two of a 10-minute duration each, and analyze the resulting water for its Total Organic Carbon Content (TOC). This analytical procedure has demonstrated encouraging results for the removal of the non-volatile residue (NVR) material from small metal parts for the validation process.

Switching to a new methodology for the validation process requires that not only the proposed approach should meet KSCs strict NVR requirement of less than 5 mg/ft^2 but also should not affect the hardware adversely under any circumstances. Ultrasonification may take out the NVR material from the cleaned parts for the validation process but may cause surface etching of the parts. This research project is focussed basically to address the following:

1. Will the proposed ultrasonic validation test method utilizing water and commercial ultrasonic baths operating at low frequencies and higher generator output power may result in the surface cavitation of the nozzle area of the commonly used fittings made of stainless steel, brass and anodized aluminum?

2. How the polymer-based Vespel SP-21 and Nylon 6/6 disks will perform to the proposed ultrasonic validation method i.e., will there be a variation in their performance specifications?

II-MATERIALS AND METHODS

2.1 Metal and Non-metal Specimens:

Small standard commercial metal fittings (Swagelok type), abundantly used at KSC and made of stainless steel (SS-400-6-4AN), brass (B-400-6-AN) and anodized aluminum (A-400-6-AN) were used to monitor the effect of the proposed ultrasonic validation method for surface erosion or activity of the parts. Especially, the precision nozzle area of these metal fittings were monitored for any ultrasonic cavitational effect during the course of this investigation since any resulting surface activity to this area will render them useless for non-compliance with the strict KSC specifications.

Most of the metal fittings were newly purchased for this study except for some previously used specimens made of stainless steel and brass. These used samples were supplied by the CFC group and were tested only in the ultrasonic water bath (blackstone) operating at 24-26 KHz frequency for this study.

The non-metallic polymer disc components commercially known as Nylon 6/6 and graphite reinforced Vespel SP-21 were studied for hardness resulting from the ultrasonic test runs, and were supplied by the failure analysis group of the Material Science Laboratory.

2.2 Ultrasonic Baths:

Two types of technologies are currently used to stimulate cavitation in an aqueous medium. They are Piezoelectric and Magnetostrictive.

Piezoelectric type ultrasonic baths operating at 24-26 KHz frequency having 600 watts of generator output power (Blackstone), and 27 KHz frequency with 1000 watts of output power (Sonic Systems) were used for the experimental studies. The basic difference in their performance is the output power since the higher generator output power normally increases the number of bubbles in the ultrasonic water tank for a higher cleaning efficiency.

Magnetostrictive type sonic water baths (MagnaPak by Branson) and (Blue Wave by Swen) operating on 20 KHz and 30 KHz respectively, and both having 1000 watts of generator output power were also used for this study. These baths operating at low frequencies and generating a larger size of the vacuum bubbles, result in a higher ultrasonic effect of cleaning and erosion.

2.3 Ultrasonic Bath Water Quality:

Water quality is an important factor for the commercially available ultrasonic baths. Pure water is difficult to cavitate while tap water cavitates easily. Tap water having some detergent to improve mixing was used in the ultrasonic baths. Before the ultrasonic runs, the water was degassed to increase the formation of cavitation bubbles.

2.4 Specimen Holder Trays:

Stainless trays having approximately 2 liters of pure heated deionized water maintained at 52 degrees Celcius were used for the ultrasonic test runs. These trays were placed into the ultrasonic bath insuring that the water level in the ultrasonic bath and the sample tray was nearly at the same level.

2.5 Sample Handling:

2.5.1 Rotary System for Metal Specimens:

Single frequency generators normally used in commercial ultrasonic baths, may result in an intense cleaning in some areas and not enough in the other areas of the tank due to the formation of the hot spots. To avoid this localized effect during an experimental run, a slow moving rotary device (carousel type moving system) using a laboratory stirrer and a disc of plexiglass having twelve holes was fabricated in the laboratory. The metal specimens, tied with nylon cords were suspended into the test stainless steel tray having deionized water, and were kept in a constant slow motion all the time to avoid the localized effects of hot spots.

2.5.2 Polymer Discs Specimens:

Polymer discs specimens were placed in separate compartments of a perforated plastic tray. The tray was suspended into the test stainless steel tray having deionized water for providing a uniform action of the ultrasonic waves during a test run.

2.6 Scanning Electron Microscope:

The instrument used for recording the microphotographs was a Cambridge S 200 Scanning Electron Microscope. The methodology used for recording the microphotographs is the standard procedure used in the SEM laboratory for doing this type of work.

2.7 Test Procedure:

2.7.1 Metal Specimens:

Small standard metal fittings of random sizes were cleaned with Freon-113, dried in an oven, air cooled and weighed. Scanning Electron Microphotographs (SEMs) of a pre-identified nozzle area of the selected specimens for a particular test run, were taken before subjecting all the specimens to ultrasonic cavitation for varying intervals. All the ultrasonic test runs were preceded by an initial 10-minute cycle for obtaining uniform water test bath conditions. For certain test runs, a specimen (blank) was taken out of the stainless tray at the beginning of the experiment after the initial 10-minute period. After the required ultrasonic test cycle(s), a part was taken out, dried in an oven, air cooled, weighed, and saved for the SEM photograph.

SEM of the nozzle area of a particular specimen photographed initially was also taken after the ultrasonic test run. In some test runs, water of a complete test cycle of 120 minutes was filtered through a 0.45 micron filter paper for the microscopic evaluation of the residue for the presence of micron-sized eroded metal pieces. A total of twelve 10-minute cycle test runs were made during a complete test of the standard fittings. CFCs proposed validation method recommends a maximum of two 10-minute ultrasonic cycles for validation purposes. Since the parts are reusable, it was decided to test run them for a maximum of twelve 10-

minute cycles. The percent material loss for each specimen was calculated after each cycle.

2.7.2 Polymer Specimens:

Polymer specimens were cleaned with Freon-113, air dried and weighed in batches of four disks each. The specimens were tested in ultrasonic water baths for 4-, 8- and 12 cycles. After the test runs, the samples were placed on a paper towel to remove the excess water, air dried (overnight), weighed and saved for the hardness test. Water absorption for each batch, and for the individual disk was calculated after the ultrasonic test runs. Hardness of the polymer specimens was measured with a Durometer Type D (ASTM D2240) Shore Hardness Tester provided by the Failure Analysis Group of the Material Science Laboratory. Microscopic evaluation was done using a common laboratory optical microscope.

III-RESULTS AND DISCUSSIONS

3.1 Metal Specimens:

The material loss in mass units (mg) due to ultrasonic cavitation for all the tested specimens was found to be on a microscopic level, and is expressed in terms of the percent mass loss. The percent mass loss plotted as a function of the total ultrasonification time in minutes was found to be non-linear, and is presented in Figures 3-1 to 3-3 for the ultrasonic water baths used in this project. In the case of the anodized aluminum and the brass specimens, the small material loss in the early cycles reflects the probable occurrence of an incubation period followed by a rapid surface erosion which levels out to a final steady-state erosion of the material. This trend is highly noticeable with more efficient magnetostrictive ultrasonic water baths.

The material loss in the initial cycles may also be attributed to the presence of statistically weak spots in the solid surface due to the grain size and inhomogeneities of the structure of the material. The stainless steel specimens did not demonstrate this trend up to a maximum of two hours of ultrasonic test run. The data obtained for the specimens utilizing different ultrasonic water baths are described below.

3.1.1 Blackstone Ultrasonic Bath (24-26 KHz, 600 watts):

3.1.1.1 Material Loss:

An anodized aluminum specimen (Figure 3-1) demonstrated the maximum percent mass loss of 11×10^{-3} for a 20-minute ultrasonic test cycle compared to a maximum of 5.4×10^{-3} for a 60-minute test run for a brass specimen (Figure 3-2), and 4.2×10^{-3} for a 20-minute test run for a stainless steel specimen (Figure 3-3). The data obtained on the anodized aluminum specimens reflect a random distribution of the percent mass loss ranging from 10.5×10^{-3} for a 10-minute cycle specimen to a 5.9×10^{-3} for a specimen tested for two hours. The brass specimens demonstrated a similar pattern of random fluctuation in the percent mass loss ranging from 1.1×10^{-3} for a 20-minute test run to 2.7×10^{-3} for a specimen tested for two hours. The mass loss pattern observed for the stainless steel specimens was similar to the brass and the aluminum specimens with the percent mass loss ranging from 1.6×10^{-3} for a 10-minute cycle to 2.5×10^{-3} for two hours of ultrasonic test run. The data obtained for brass and stainless steel are of the used specimens supplied by the CFC laboratory. Some of the material loss may be attributed to the dirty and the greasy material adhered to the specimens, and which were cleaned up during the test runs.

3.1.1.2 Microscopic Evaluation:

SEMs of the nozzle area of the anodized aluminum specimens subjected to ultrasonic cavitation from 40- to 120 minutes revealed surface activity resulting from ultrasonic cavitation on a microscopic level to all the specimens. Figures 3-4 and 3-5 are the microphotographs of four and twelve cycles test run specimens. A slight preferential erosion of the grain boundaries was observed in the 101x magnified microphotograph (Figure 3-5) obtained for a 12-cycle test specimen. An optical microscopic evaluation of the same specimen also revealed an area under the threads where the base aluminum metal was slightly exposed. This may be due to a weaker anodized coating near the thread area of the specimen. No surface activity of the nozzle area was observed under an optical microscope demonstrating that the surface integrity of the specimen was maintained even after 120 minutes of the test run. A specimen tested for a 20-minute cycle did not reveal any surface activity arising from the ultrasonic cavitation of the specimen.

SEMs of the used brass specimens tested for 4- and 8 cycles are presented in Figures 3-6 and 3-7 respectively. An unusual pattern on the nozzle area of the specimens prompted a concern that there may be a surface erosion activity resulting from the ultrasonic cavitational effect. In order to verify this concern, newly purchased specimens were cleaned with a moderate 10% Ferric Chloride acidic solution before subjecting them to ultrasonic cavitation for 4-, 8- and 12 cycles. SEMs (Figures 3-8a to 3-9b) of the cleaned and the raw specimens were obtained before and after the ultrasonic cavitation test runs. A careful evaluation of these SEMs did not reveal the same pattern as was observed earlier with the used parts supplied by the CFC group. The pattern observed previously on the nozzle area of the used samples must be the result of a poor machine finish or heavily used parts which resulted in a pronounced effect (pattern) on the SEM photographs. The microphotograph (Figure 3-10) of a test run brass specimen (new) tested for 12 cycles did not reveal any surface activity or erosion due to ultrasonic cavitation.

Figures 3-11a and 3-11b are the SEMs of a used stainless steel specimen subjected to ultrasonic cavitation for 120 minutes. These microphotographs demonstrate a highly effective cleaning action of the ultrasonic water bath without any cavitational damage to the specimen; Figure 3-11b, a magnified microphotograph especially reflect this effectiveness.

3.1.2 SONIC Systems Ultrasonic Bath (27 KHz, 1000 watts):

3.1.2.1 Material Loss:

The anodized aluminum specimens revealed a higher ultrasonic cavitational effect of the Sonic Systems ultrasonic water bath as demonstrated by the data obtained on the material loss due to the ultrasonic test runs. The maximum percent mass loss for an anodized aluminum specimen (Figure 3-1) was 50×10^{-3} for a 120-minute test run compared to 8.3×10^{-3} for a 80-minute test run for a brass specimen (Figure 3-2), and 4.3×10^{-3} for a 40-minute test run for a stainless steel (Figure 3-3) specimen. The material loss from the anodized aluminum specimens seems to stabilize after a 40-minute test run with the percent mass loss of 43×10^{-3} for a specimen reaching to a maximum of 50×10^{-3} for 120 minutes of test run for another specimen.

The brass specimens demonstrated a random fluctuation in their percent mass loss ranging from 3.8×10^{-3} for a 20-minute test run to 4.6×10^{-3} for two hours of test run. The stainless steel specimens also demonstrated a similar pattern of random loss with the percent mass loss of 1.1×10^{-3} for a 20-minute test run specimen compared to 2.3×10^{-3} for a 120-minute of test run for another specimen.

3.1.2.2 Microscopic Evaluation:

SEMs of the nozzle area of the anodized aluminum specimens tested for 2-, 4-, 8- and 12 cycles in the Sonic Systems ultrasonic water bath demonstrated the impact of ultrasonic cavitation on a microscopic level to all the specimens. Figures 3-12 and 3-13 of a 20- and 120-minute test run specimens revealed a slight surface activity (erosion) of the grain boundaries. The residue obtained after filtering the water sample of the complete test run was subjected to an optical microscopic evaluation. The identification of the metal fragments in the residue (Figure 3-14) prompted to reveal the presence of weak spots or boundaries in the solid surface of the anodized aluminum specimens. The metal fragments from these weak spots on the solid metal surface loosened during the incubation period, and this surface activity of the specimens was reflected in the magnified SEMs of the specimens described before.

The evaluation of the SEMs of the nozzle area of the brass and the stainless steel specimens tested for 4-, 8- and 12 cycles did not reveal any unusual surface activity due to ultrasonic cavitation. However, eroded metal fragments were also identified in the water residue of a complete test cycle for brass specimens. Figures 3-15 and 3-16 are the microphotographs of a complete 120-minute test run for a brass and a stainless steel specimen respectively. The microphotographs clearly demonstrate the ultrasonic cleaning action without any observable cavitational effect to the nozzle area of the specimens.

3.1.3 Branson Ultrasonic Bath (20 KHz, 1000 watts):

3.1.3.1 Material Loss:

The ultrasonic cavitational test runs in this magnetostrictive ultrasonic water bath resulted in a unique trend of material loss compared to the Sonic Systems Peizoelectric ultrasonic water bath of the same generator output power. The material loss in terms of the percent mass loss reached a maxima during the 120 minutes of the test run for all the three metal alloy specimens tested in this project. It reached a maximum value of $61x10^{-3}$ for the anodized aluminum (Figure 3-1) specimen compared to $10x10^{-3}$ and $1.9x10^{-3}$ for the brass (Figure 3-2) and the stainless steel (Figure 3-3) specimen respectively. Ultrasonic cavitational effect to the anodized aluminum specimen was found to be higher with the percent mass loss rising from $12x10^{-3}$ for a 10-minute test run to $61x10^{-3}$ for a 120-minute test run for another anodized aluminum specimen; a five fold increase. A brass specimen demonstrated the percent mass loss from $3.3x10^{-3}$ for a 10-minute test run to $10x10^{-3}$ for another specimen for a two hour test run; a three fold increase. Figures 3-1 and 3-2 reflect a definite steady erosion rate for the anodized aluminum and brass specimens during the ultrasonic test runs using Branson ultrasonic water bath. Again, the stainless steel specimens (Figure 3-3) resulted in a minimum ultrasonic cavitational effect with the percent mass loss distributed randomly from 0.9x10^{-3} for a 20-minute test run rising to 1.9x10^{-3} for two hours of the test run.

3.1.3.2 Microscopic Evaluation:

The evaluation of the SEMs of the nozzle area of the anodized aluminum specimens tested from four to twelve cycles reflected a pronounced effect of surface erosion using the Branson ultrasonic water bath. The microphotographs (Figures 3-17 to 3-19a) clearly demonstrate the presence of random pits and damage to the nozzle area of the specimens subjected to ultrasonic cavitation from four to twelve cycles. Figure 3-19b is a 919x magnified microphotograph of the nozzle area of a 12-cycle anodized aluminum specimen. It revealed an extensive worked surface with widened pits due to ultrasonic cavitation. The damage was not found to be so profound for a 2-cycle specimen (Figure 3-20). A careful optical microscopic evaluation of all the tested anodized aluminum specimens, revealed the absence of the anodized coating from the nozzle as well as from the under-thread areas of the specimens; the erosion of the coating was very much pronounced for a 12-cycle specimen while the 2-cycle specimen revealed the absence of the metal coating to the under-thread area of the specimen (Figure 3-21).

The evaluation of the SEMs obtained for the brass and stainless steel specimens tested from two to twelve cycles did not reveal any unusual surface activity as a result of ultrasonic cavitation. The SEMs of the nozzle area of a 12-cycle test run specimens of brass (Figures 3-22a and 3-22b) and stainless steel (Figures 3-23a and 3-23b) clearly demonstrate the effectiveness of ultrasonic cleaning without causing any surface erosion or damage to the specimens.

3.1.4 Blue Wave (Sven) Ultrasonic Bath (30 KHz, 1000 watts):

3.1.4.1 Material Loss for Anodized Aluminum:

The anodized aluminum specimens tested in this magnetostrictive ultrasonic bath demonstrated a similar trend of material loss as compared to Sonic Systems and Branson ultrasonic Baths. The percent material loss was found to be 14.5x10^{-3} for a specimen tested for 20 minutes as compared to 63.7x10^{-3} for another specimen tested for 120 minutes.

3.1.4.2 Microscopic Evaluation:

The evaluation of the SEMs of the nozzle area of the anodized aluminum specimens (Figures 3-24 and 3-25) tested from two to twelve cycles revealed an extensive etching of the grain boundaries of the specimens; the etching is highly pronounced for 8- and 12 cycles specimens as compared to 2- and 4 cycles specimens. Figure 3-25 demonstrate a highly worked surface area of these specimens due to ultrasonic cavitation. The surface etching to the anodized aluminum surface by this magnetostrictive ultrasonic bath followed a similar pattern of cavitational damage as was observed previously in the case of the more powerful magnetostrictive Bronson ultrasonic bath i.e., surface damage has a linear relationship with the ultrasonification time.

3.2 Polymer Specimens:

3.2.1 Water Absorption:

Nylon 6/6 and graphite reinforced Vespel-21 polymer disks tested in the Sonic Systems and Branson ultrasonic water baths resulted in water absorption by the specimens. The amount of water absorbed by the Nylon 6/6 disks in the Sonic Systems ultrasonic water bath was slightly lower than in the Branson ultrasonic water bath (Figure 3-26). It ranged from 0.027 percent for a 4-cycle test run to 0.112 percent for a 12-cycle run for the Sonic Systems water bath compared to 0.077 percent for a 4-cycle run to 0.27 percent for a 12-cycle run for the Branson ultrasonic bath. For the graphite reinforced Vespel-21 disks, the percent water absorption was on a microscopic level; it varied from 0.002 for a 4-cycle test run to 0.012 for a 12-cycle run using Sonic Systems ultrasonic water bath compared to 0.005 for a 4-cycle test run to 0.032 for a 12-cycle run for the Branson ultrasonic water bath.

3.2.2 Hardness Test:

Nylon 6/6 and Vespel-21 specimens before and after subjecting to ultrasonic cavitation in the Sonic Systems and Branson ultrasonic water baths for 4-, 8- and 12 cycles were tested for hardness. The purpose of this test was to evaluate any variation in their hardness performance specification resulting from the ultrasonic cavitation. The data obtained on Durometer Type D hardness scale varied from 81 to 83 for all the Nylon 6/6 specimens (4 disks/cycle) as compared to 85 to 86 for all the Vespel-21 specimens before and after the test run. For the comparison purposes, a typical laboratory polyethylene bottle cap gave a value of 70 on the Durometer scale. The variation in the hardness data before and after the ultrasonic cavitation of the specimens demonstrate that the polymer disks are very resistant to ultrasonic cavitation.

3.2.3 Microscopic Study:

The specimens (batches of Nylon 6/6 and Vespel-21) before and after the ultrasonic cavitation test of 4-, 8- and 12 cycles for the two ultrasonic baths (Sonic Systems and Branson) were subjected to optical microscopic evaluation. The evaluation did not reveal any unusual surface activity due to ultrasonic cavitation of the specimens.

IV-CONCLUSIONS

1. Ultrasonic cavitation of the stainless steel and brass specimens with water in the three commercially available ultrasonic baths (Blackstone, Sonic Systems and Branson) operating at low frequencies (20-27 KHz) and higher power output (600-1000 watts) did not result in surface cavitation of the nozzle area of the tested parts.

2. The anodized aluminum fittings are found to be slightly affected (microscopic level) in the Blackstone (24-26 KHz, 600 watts) and the Sonic Systems (27 KHz, 1000 watts) ultrasonic baths. The surface integrity of the nozzle area of the specimens are maintained even after two hours of ultrasonic cavitation in these baths.

3. The nozzle surface of the anodized aluminum fittings is found to be greatly affected by the ultrasonic cavitational action of the more powerful magnetostrictive commercial Branson (20 KHz, 1000 watts) and Blue Wave (30 KHz, 1000 watts) ultrasonic baths.

4. On the basis of the material loss, it can be concluded that the anodized aluminum specimens pass through an incubation period of approximately 20 minutes followed by a rapid material loss which levels off to a final steady-state erosion of the material in the ultrasonic baths operating with output power of 1000 watts.

5. The material loss of the specimens subjected to ultrasonic cavitation in different ultrasonic water baths for two hours is found to be on a microscopic level of percent mass loss, and is as follows:

   - About 32 times higher for an anodized aluminum specimen and about 5 times higher for a brass specimen as compared to a stainless steel fitting using Branson ultrasonic bath.

   - About 22 times higher for an anodized aluminum specimen and 2 times higher for a brass specimen as compared to a stainless steel specimen using Sonic Systems ultrasonic bath.

   - About 2 times higher for anodized aluminum specimen as compared to a stainless steel and a brass specimen using Blackstone ultrasonic bath.

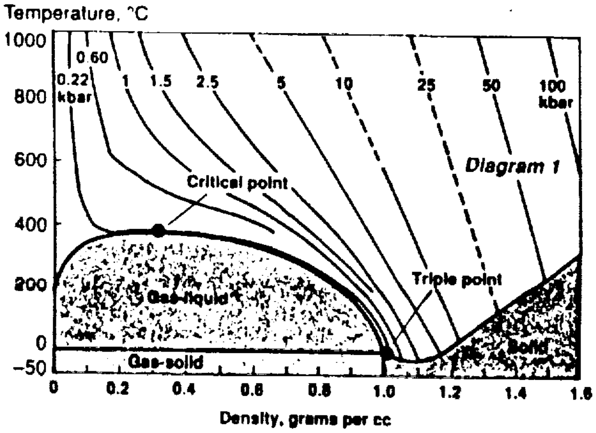
6. On the basis of the hardness test, Nylon 6/6 and graphite reinforced Vespel-21 polymer disks are not found to be affected by the ultrasonic cavitational action of up to two hours using Branson and Sonic Systems ultrasonic water baths.

7. The results have demonstrated that the proposed replacement ultrasonic test method of the CFC group can be used for validating cleaned small parts made of stainless steel and brass using commercial ultrasonic baths operating at low frequencies and higher power output. For the validation of the anodized aluminum parts, more moderate piezoelectric ultrasonic baths are found to be effective without causing adverse surface damage to the specimens.

V-RECOMMENDATIONS

Even if ultrasonic cavitation does affect the nozzle surface area of the anodized aluminum specimens adversely, it is suggested that physical testing of the cavitated parts should be carried out to evaluate the variation in their performance specification due to ultrasonic cavitation.

For the removal of the non-volatile residue (NVR) material for the clean validation process, it is recommended to explore the possibility of using water at or near the supercritical water (SCW) conditions (high temperature and pressure), since at SCW conditions, water has properties as of a fluid and a gas thus increasing its solvation power*. Experimental runs should be carried out to optimize the temperature and pressure conditions for the maximum removal of the NVR materials.



* Shaw, R. W. et al., "Supercritical Water, a Medium for Chemistry", Chem. & Engg. News 1991, 69(51), 26.

APPENDIX

WHAT IS ULTRASONIC CAVITATION ?

Ultrasonic cleaners use transducers which change electric energy into mechanical vibrations. These vibrations produce pressure waves travelling through water at the speed of sound (1450 m/s). High pressure side of the wave causes water to expand to form vapors and during compression, the vapor condenses into unstable and short-lived micron-sized cavitation bubbles (areas of vacuum). These bubbles are alternately expanded and compressed by the applied pressure waves passing through water. The bubbles continue to grow until they collapse when the pressure around them becomes positive. This is like THUNDERCLAPS on a microscopic scale. Minute areas of high pressure are created by these thunderclaps.

Before the bubble implosion, the bubble size is affected by the ultrasonic frequency applied by the transducer. The size of a bubble is inversely proportional to the frequency of the ultrasonic bath. So it is important to increase input power for maintaining a higher number of bubbles per unit volume with higher ultrasonic frequencies. Also, the number of bubbles present in the tank water is increased by increasing electrical input power.

During bubble implosion, the temperature and pressure of the gas within the bubble can reach 5500 degrees Celcius and 70,000 Lb/sq. inch (500 atmospheres) respectively. The liquid surrounding the bubble can reach 2,100 degrees Celcius. When the bubble is next to a surface to be cleaned, the implosion pressure may propel a jet of water towards that surface at about 250 MPH. The formation of millions and millions of cavitation bubbles and their collapse clean the objects literally inside and out but may also cause pitting and erosion of the solid surface.

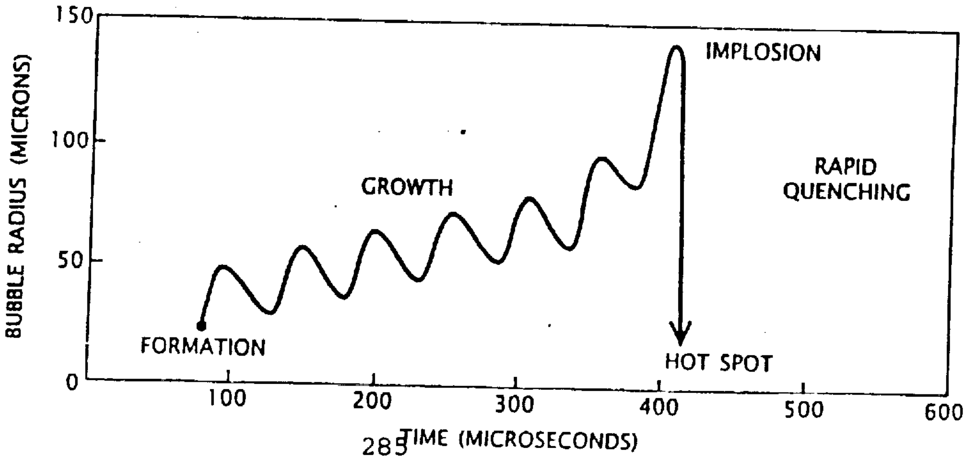


Figure 3-1

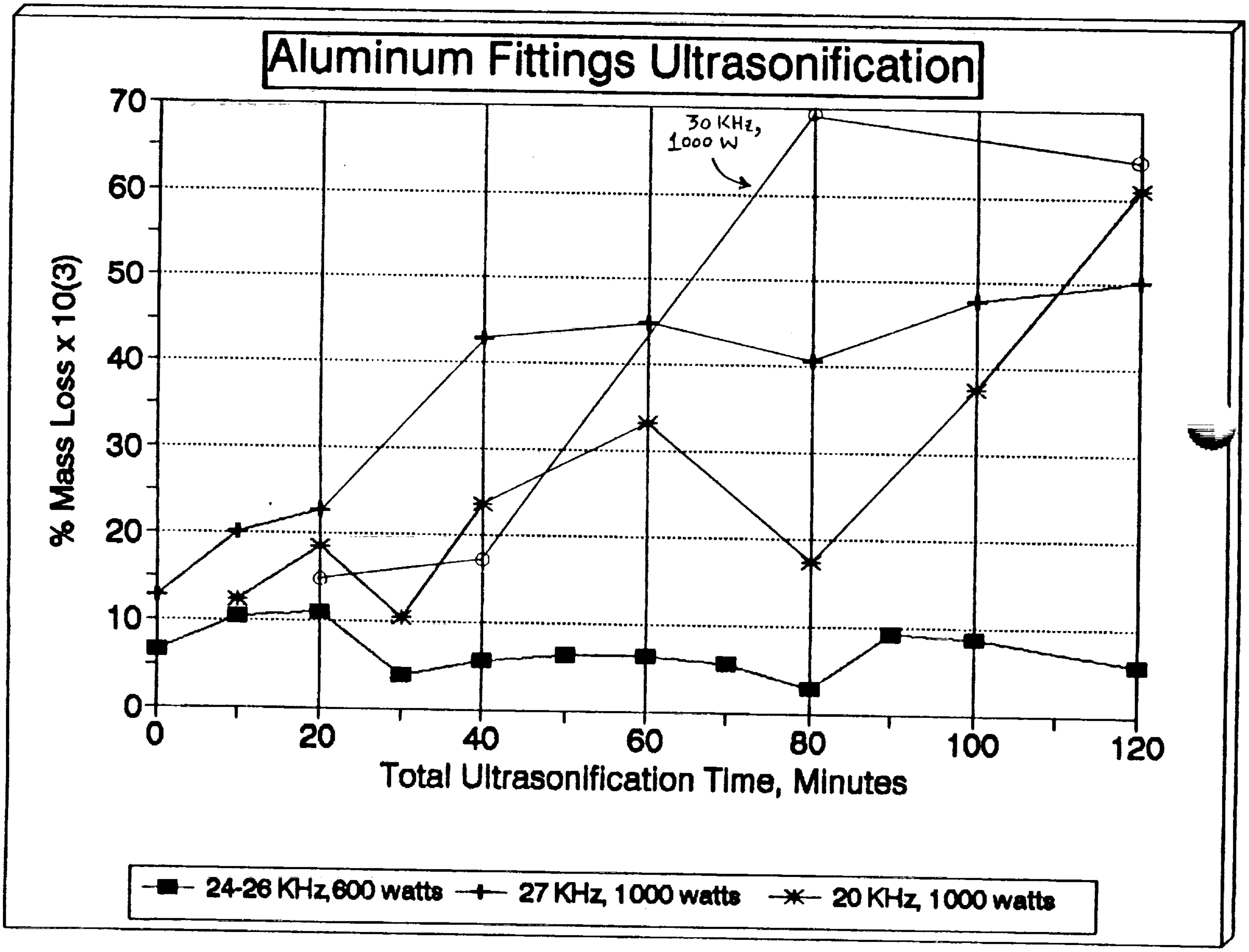
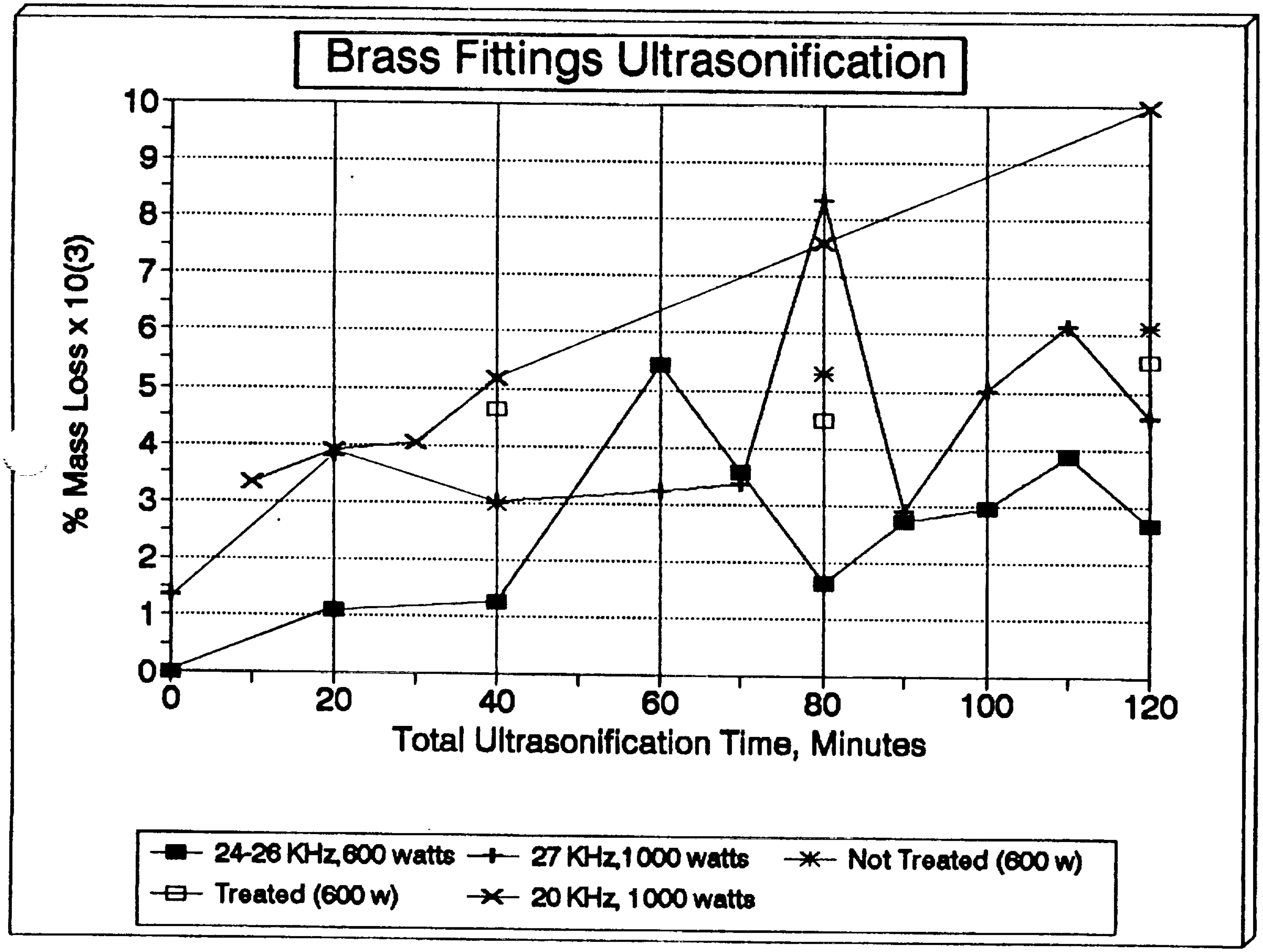

Aluminum Fittings Ultrasonification
% Mass Loss x 10(3)
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Total Ultrasonification Time, Minutes
30 KHz, 1000 W
24-26 KHz, 600 watts
27 KHz, 1000 watts
20 KHz, 1000 watts

Figure 3-2



Brass Fittings Ultrasonification
% Mass Loss x 10(3)
0
1
2
3
4
5
6
7
8
9
10
0
20
40
60
80
100
120
Total Ultrasonification Time, Minutes
24-26 KHz,600 watts
27 KHz,1000 watts
Not Treated (600 w)
Treated (600 w)
20 KHz, 1000 watts

Figure 3-3

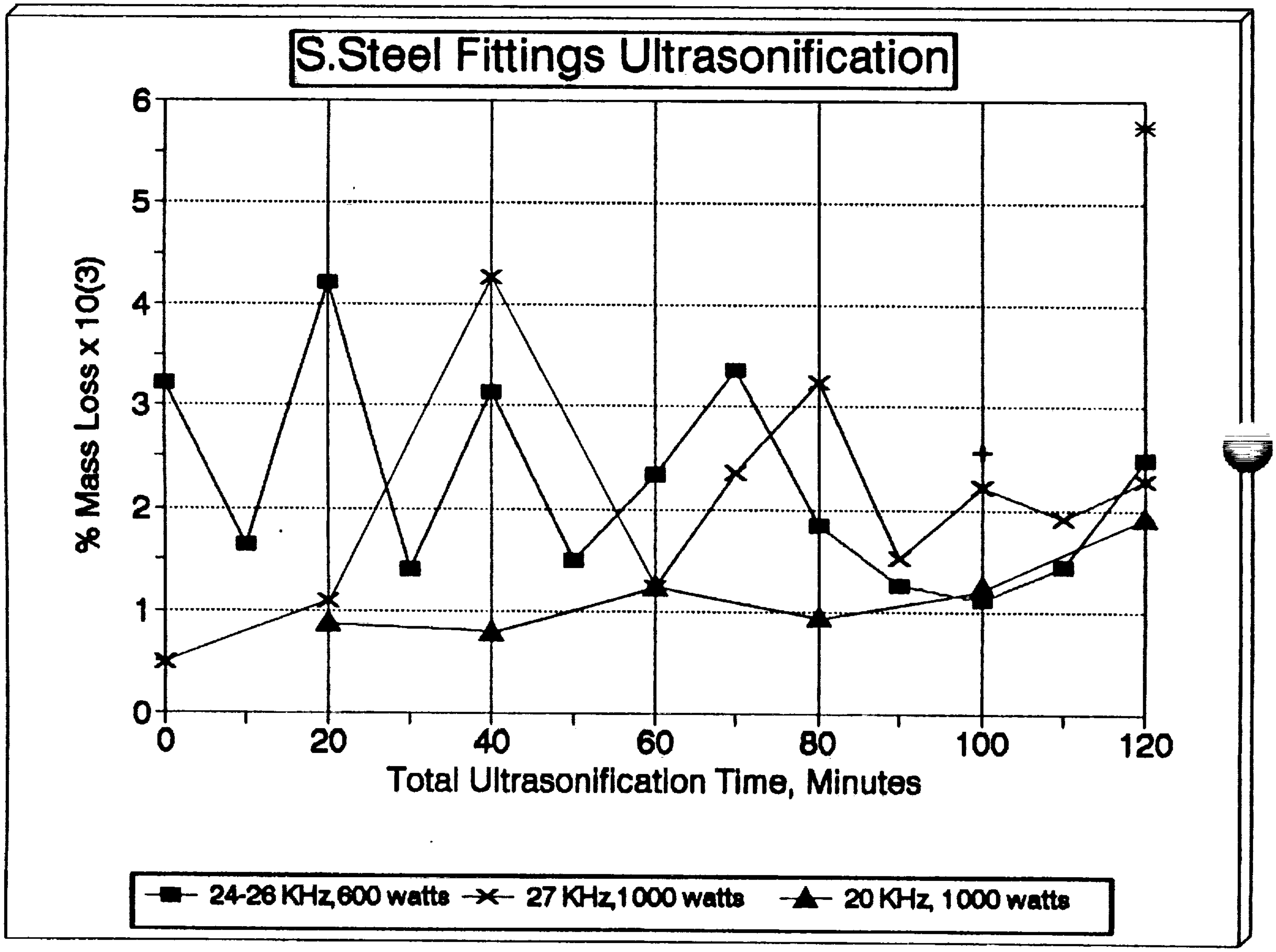
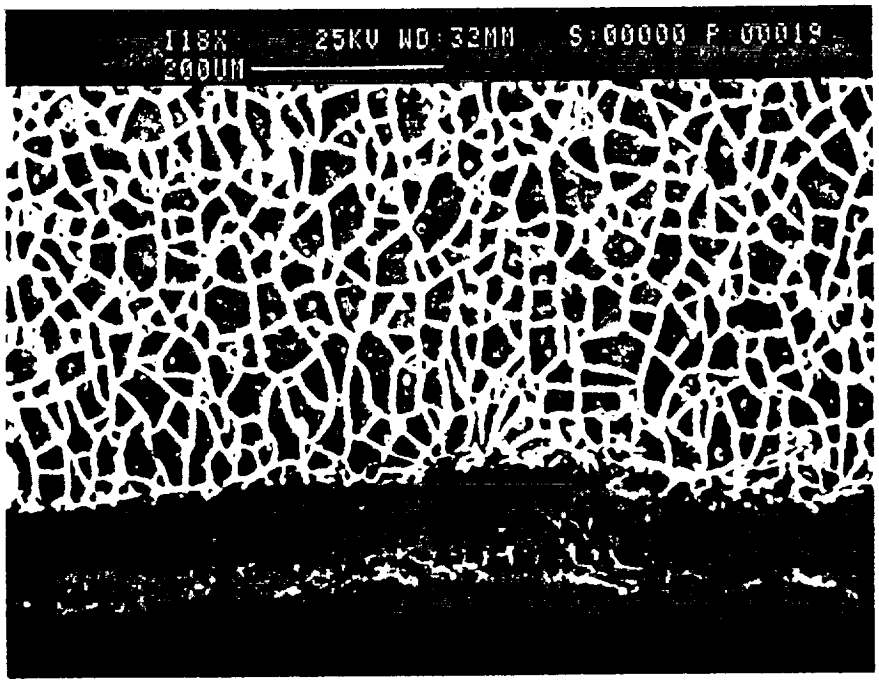
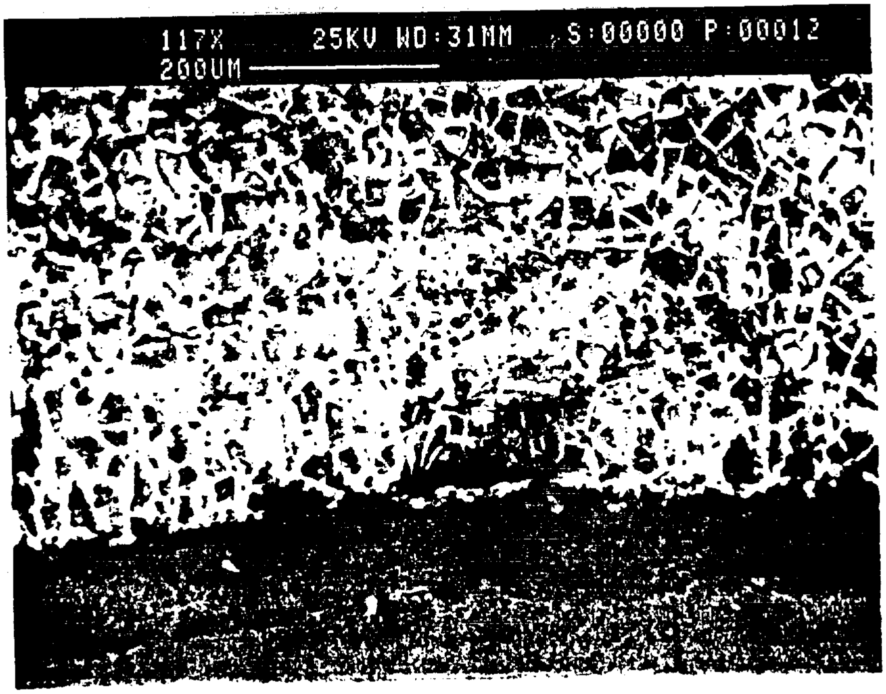

S.Steel Fittings Ultrasonification
% Mass Loss x 10(3)
0
1
2
3
4
5
6
0
20
40
60
80
100
120
Total Ultrasonification Time, Minutes
24-26 KHz,600 watts
27 KHz,1000 watts
20 KHz, 1000 watts

Figure 3-4

SEM/Anodized Aluminum/4 cycles/Blackstone



Anodized
Aluminum
T = 0

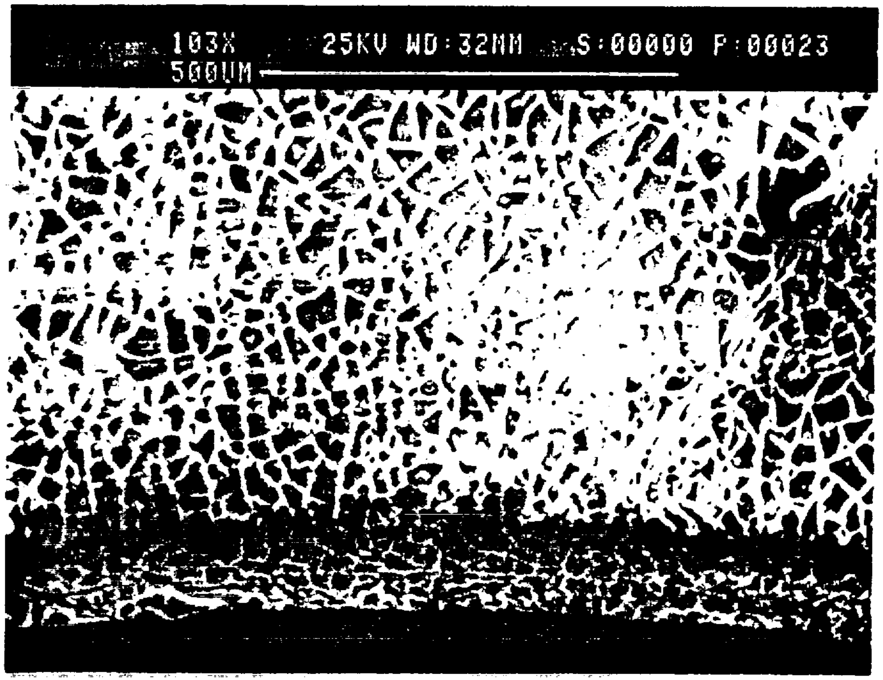


Anodized
Aluminum
T = 40 minutes

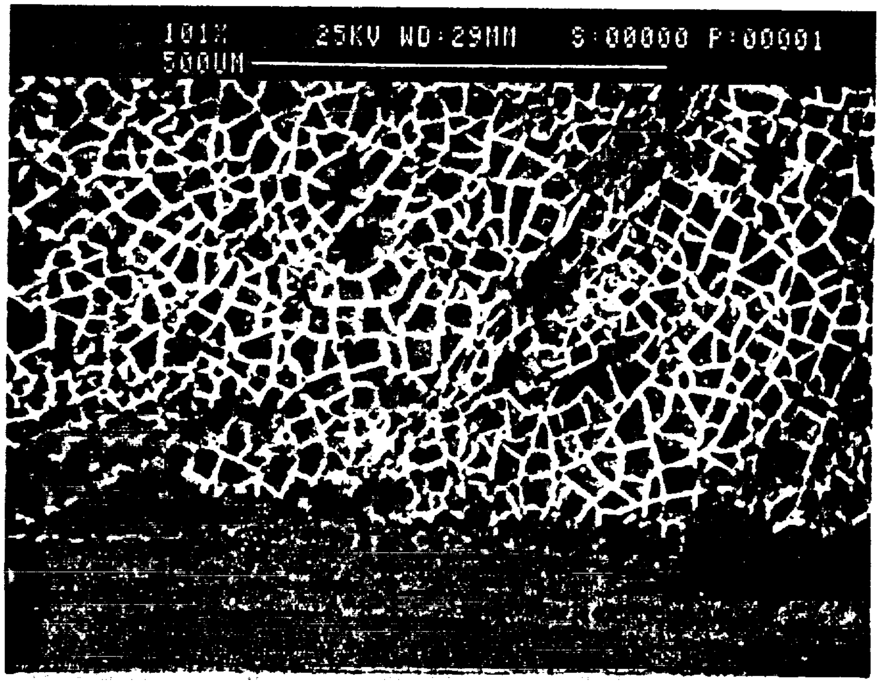
Figure 3-5

SEM/Anodized Aluminum/12 cycles/Blackstone

Anodized
Aluminum
T = 0 minutes



Anodized
Aluminum
T = 120 minutes

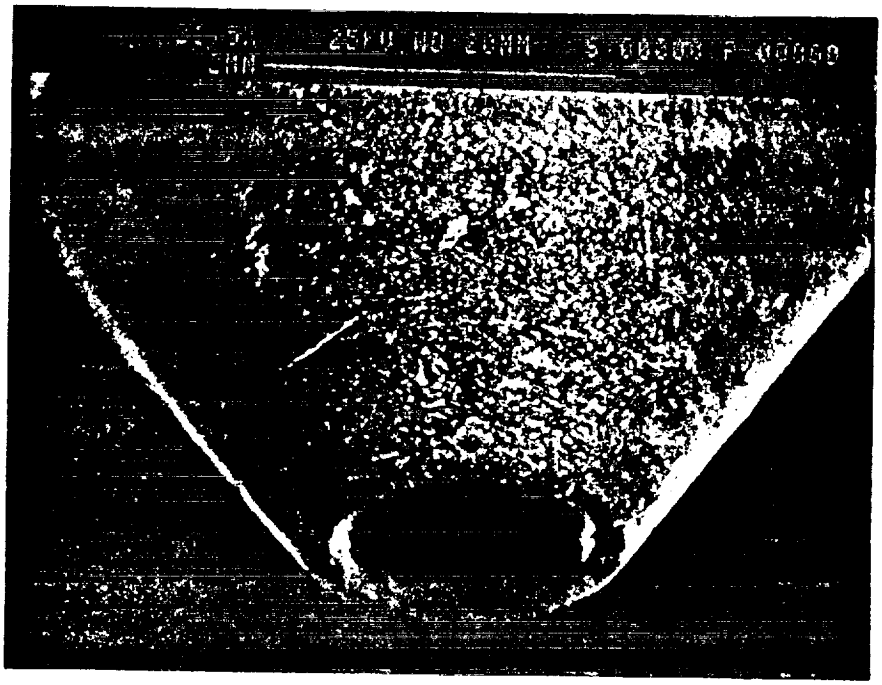


ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-6

SEM/Brass/4 cycles/Blackstone

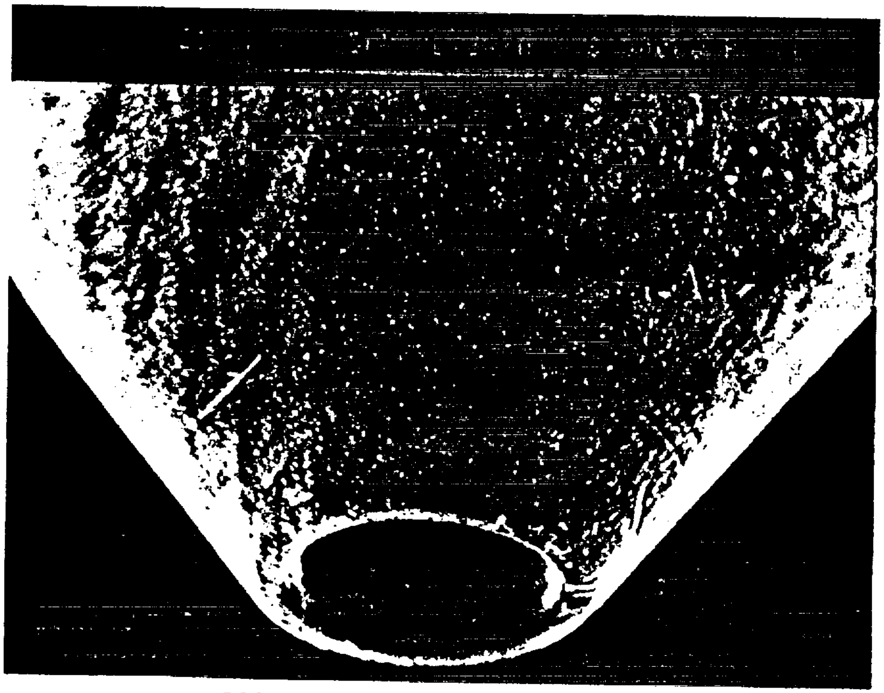
Brass
T = 0



JUNE
0 cycl

#3 BRASS

Brass
T = 40 minutes



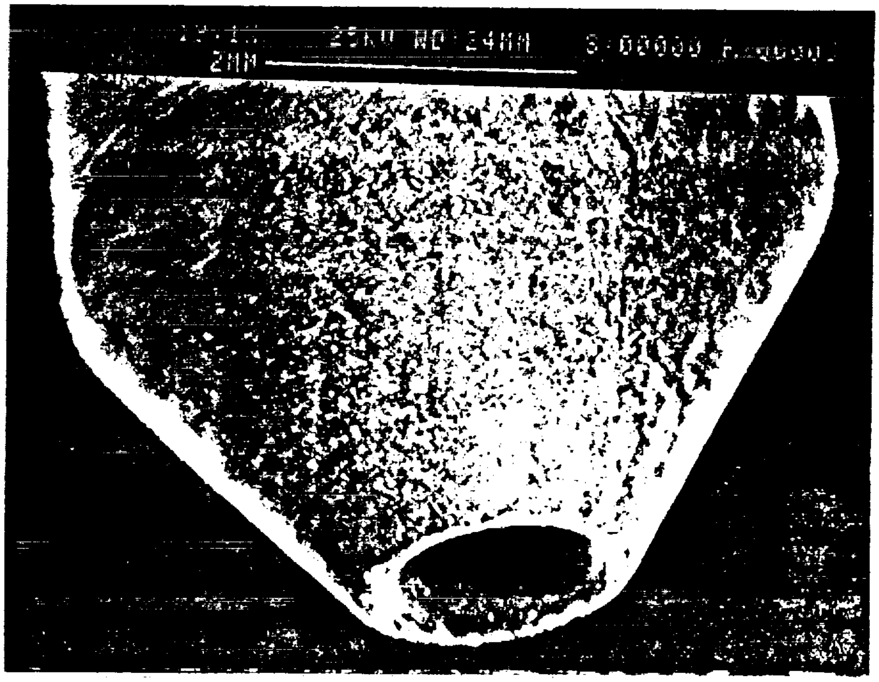
BRASS
4 cycles

#3 June 11

Figure 3-7

SEM/Brass/8 cycles/Blackstone

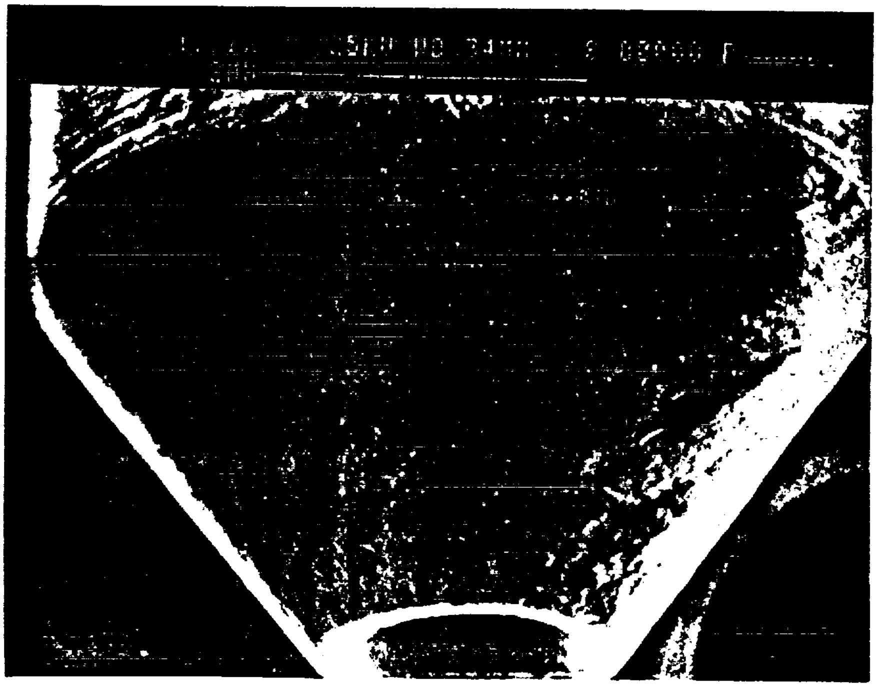
Brass
T = 0



June 5
0 cyd

6 BRASS

Brass
T = 80 minutes

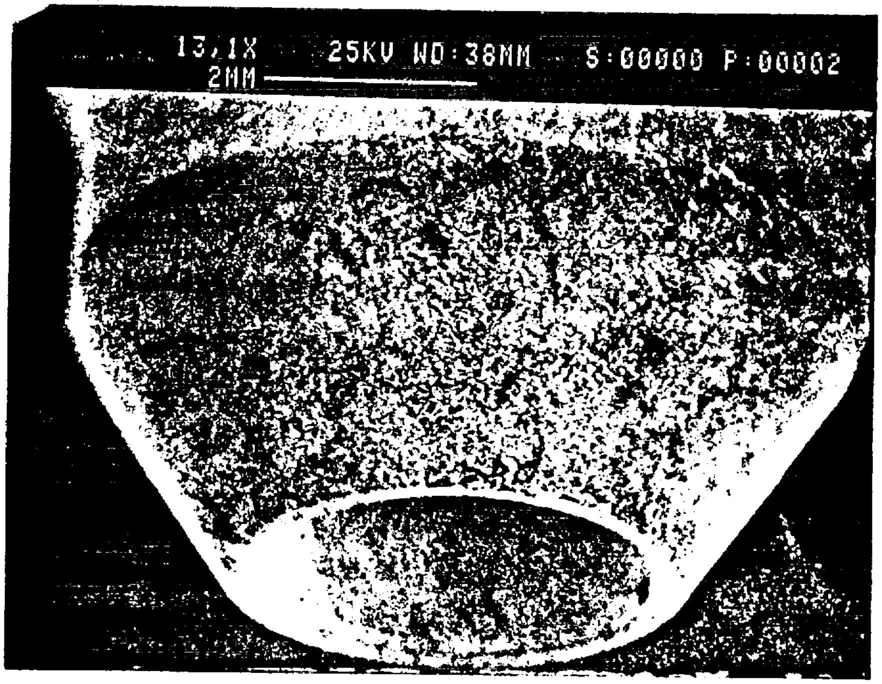


6 June 11 8 BRASS

Figure 3-8a

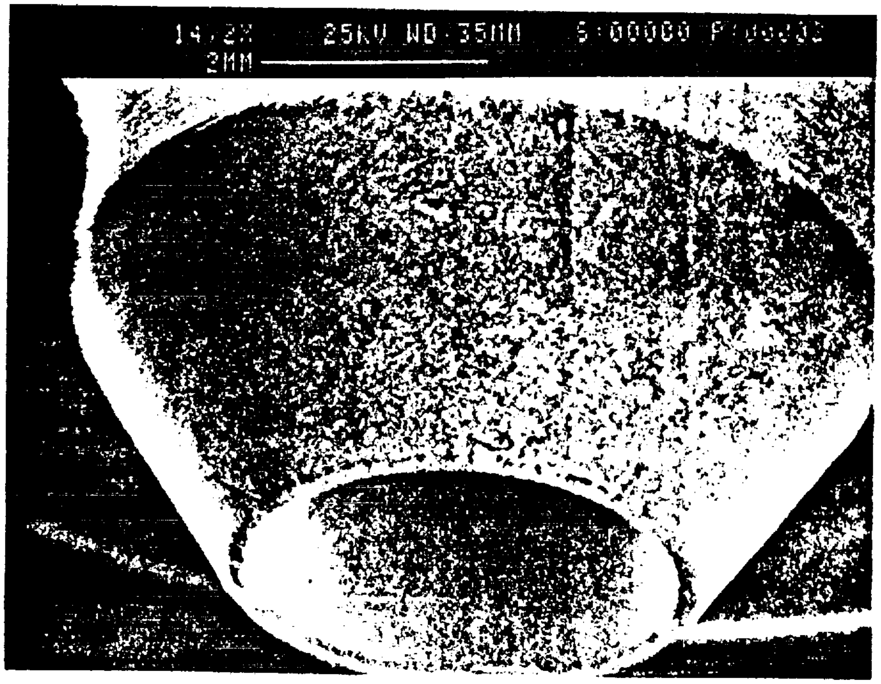
SEM/Brass Treated with ferric chloride/4 cycles/Blackstone

Brass
T = 0



TREATED [illegible]

Brass
T = 40 minutes



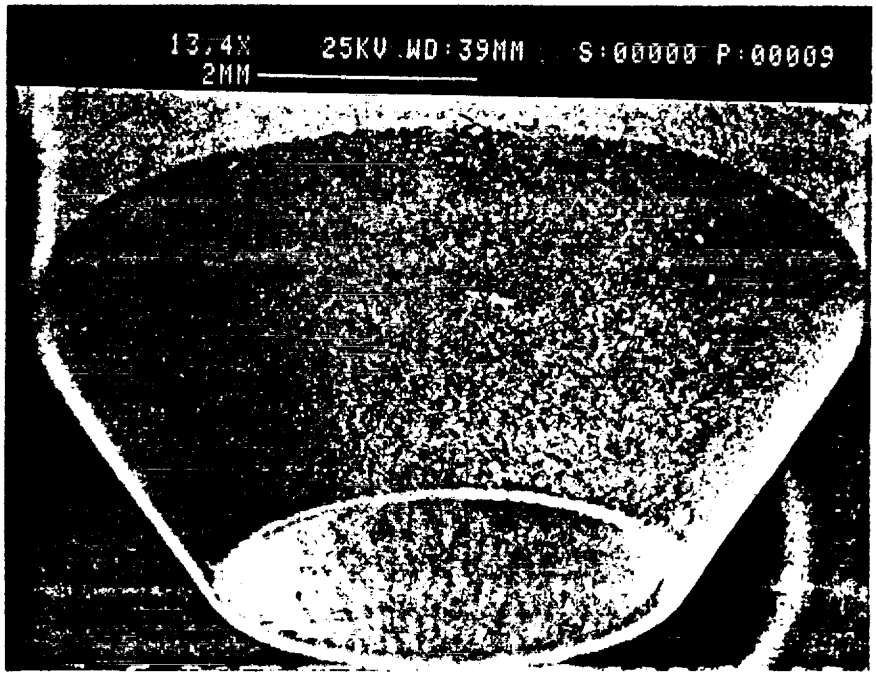
TREATED [illegible]

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-8b

SEM/Brass Not Treated/4 cycles/Blackstone

Brass
T = 0



Brass
T = 40 minutes

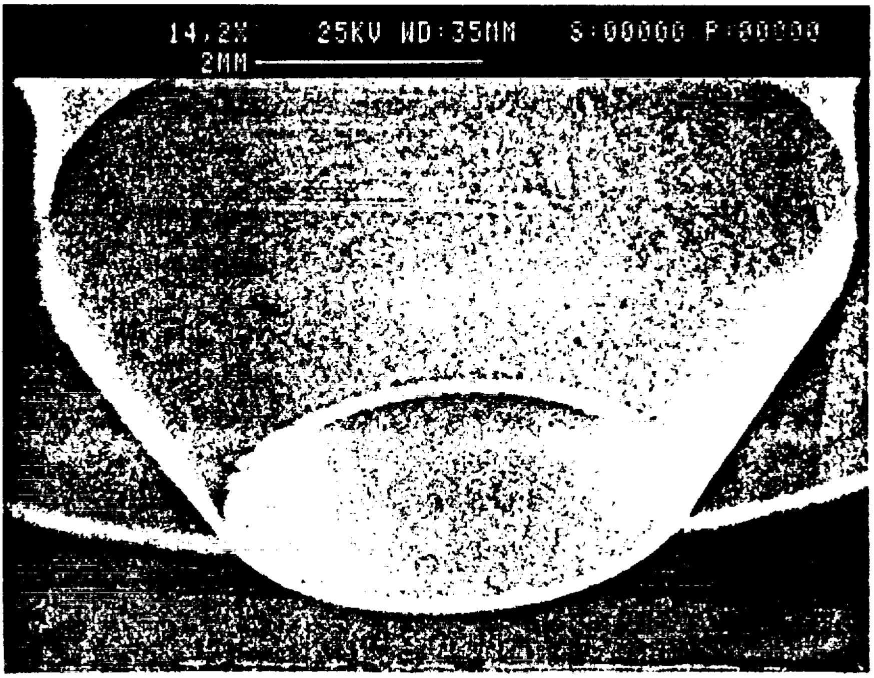
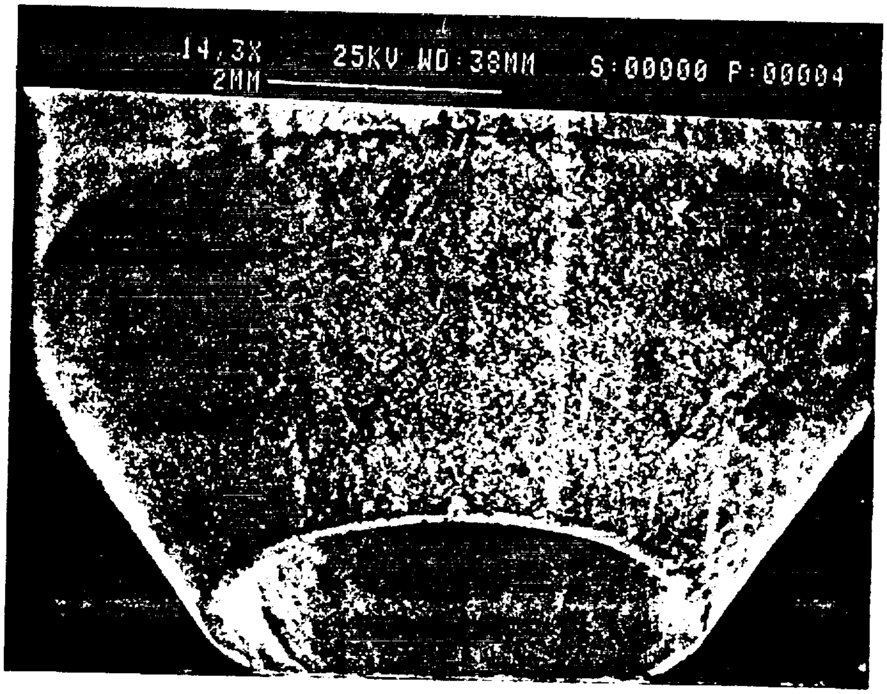


Figure 3-9a

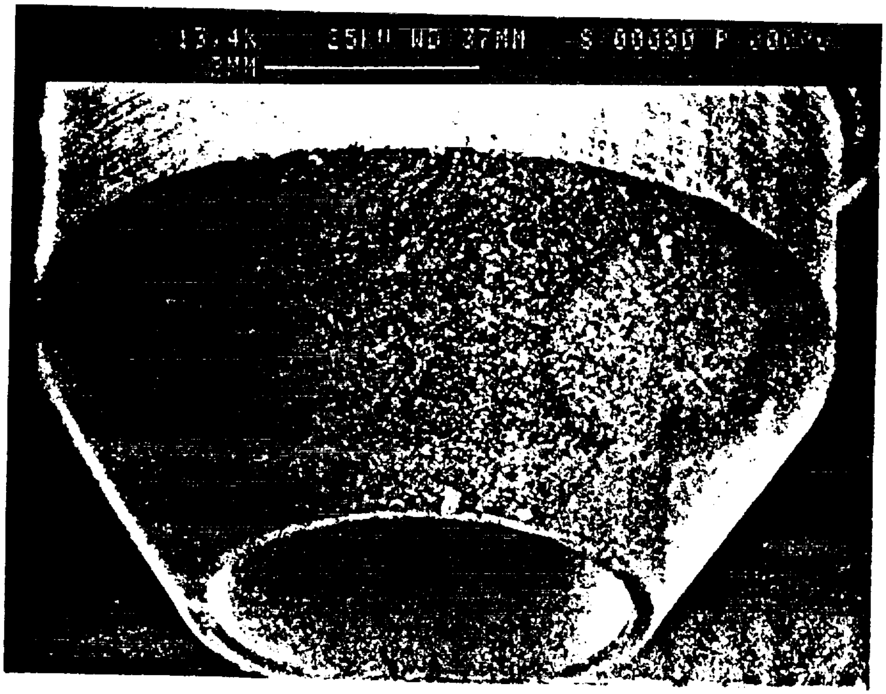
SEM/Brass Treated with Ferric Chloride/8 cycles/Blackstone

Brass
T = 0



BRASS #33 TREATED [illegible]
T = [illegible]

Brass
T = 80 minutes

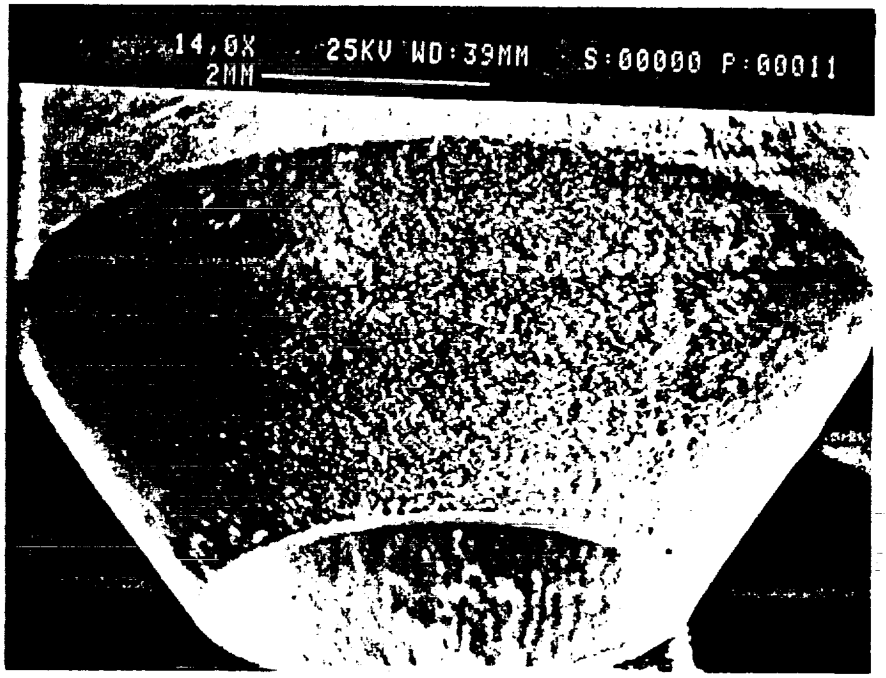


BRASS #33 TREATED WITH [illegible]

Figure 3-9b

SEM/Brass Not Treated/8 cycles/Blackstone

Brass
T = 0



Brass
T = 80 minutes

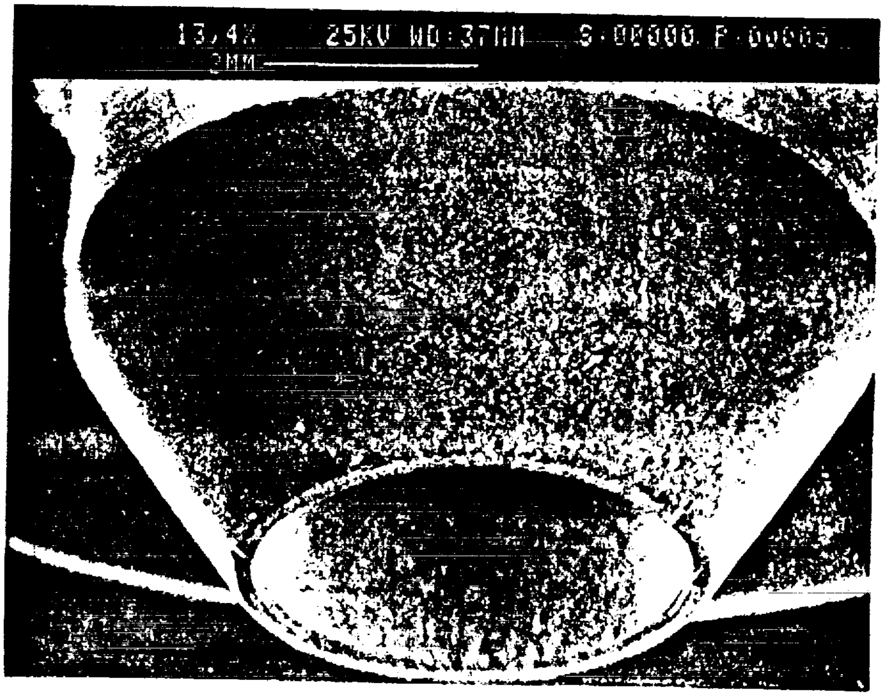
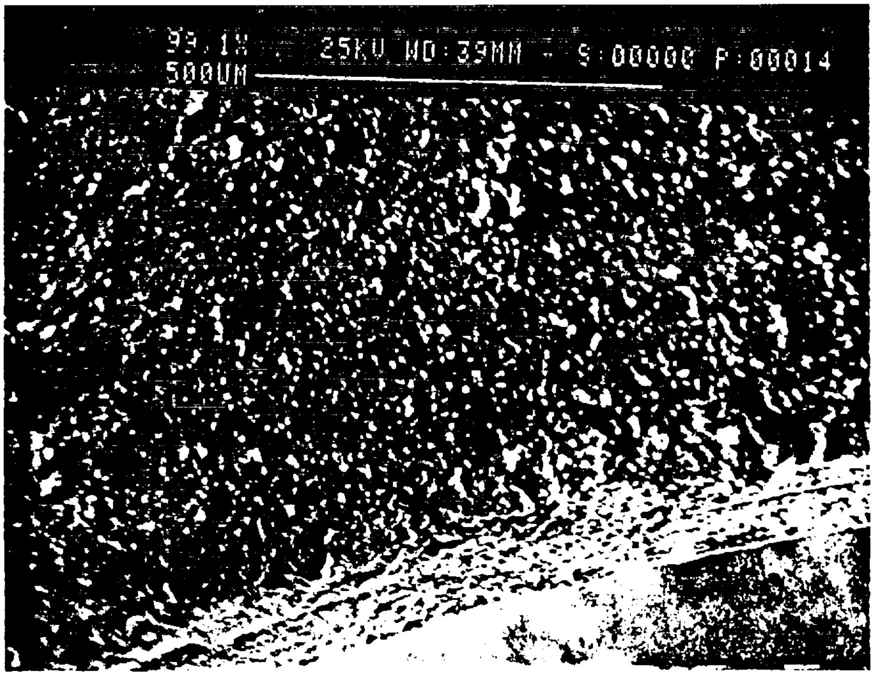


Figure 3-10

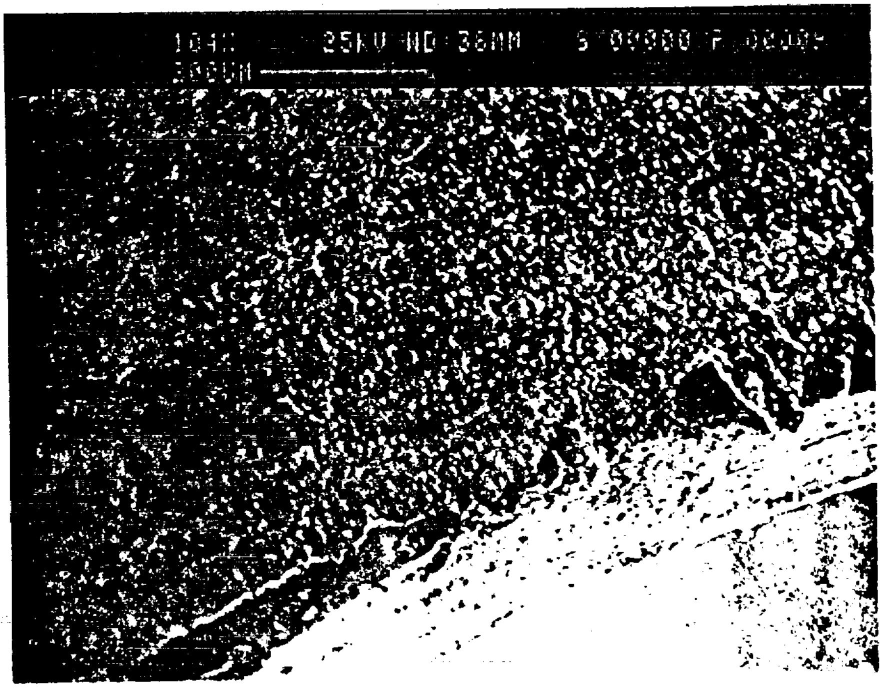
SEM/Brass/12 cycles/Blackstone

Brass
T = 0



BRASS #34 T=0

Brass
T = 120 minutes



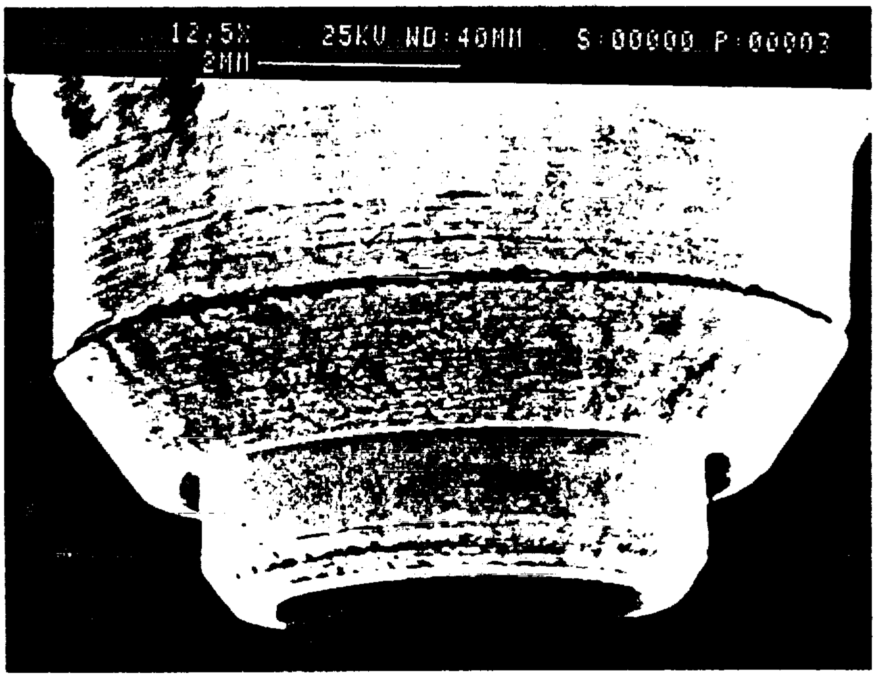
Brass #31 [illegible]

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-11a

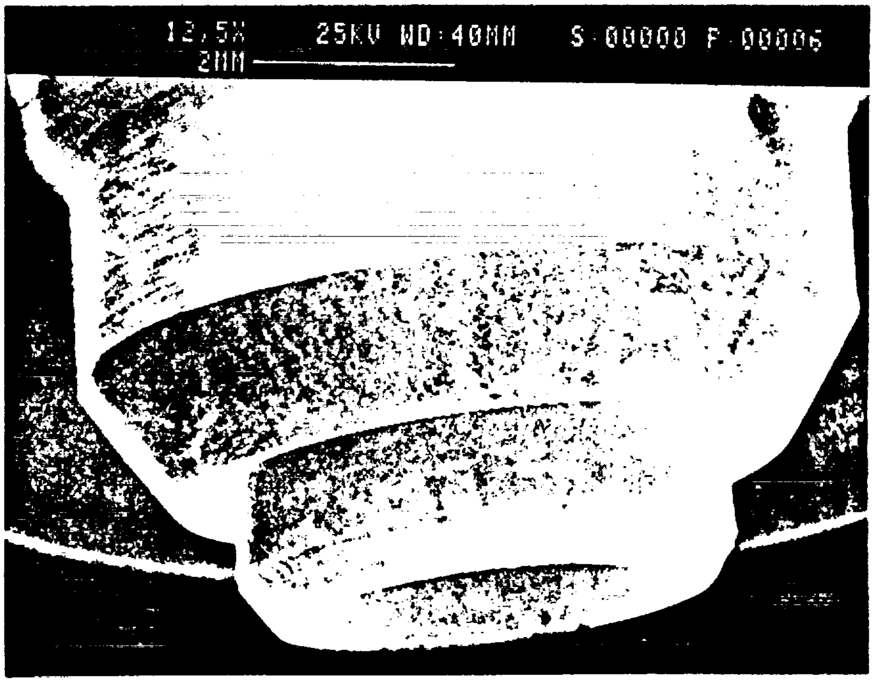
SEM/Stainless Steel/12 cycles/Blackstone

Stainless Steel
T = 0



S.S.
Sampl
#1
June.
orgi
Samp

Stainless Steel
T = 120 minutes



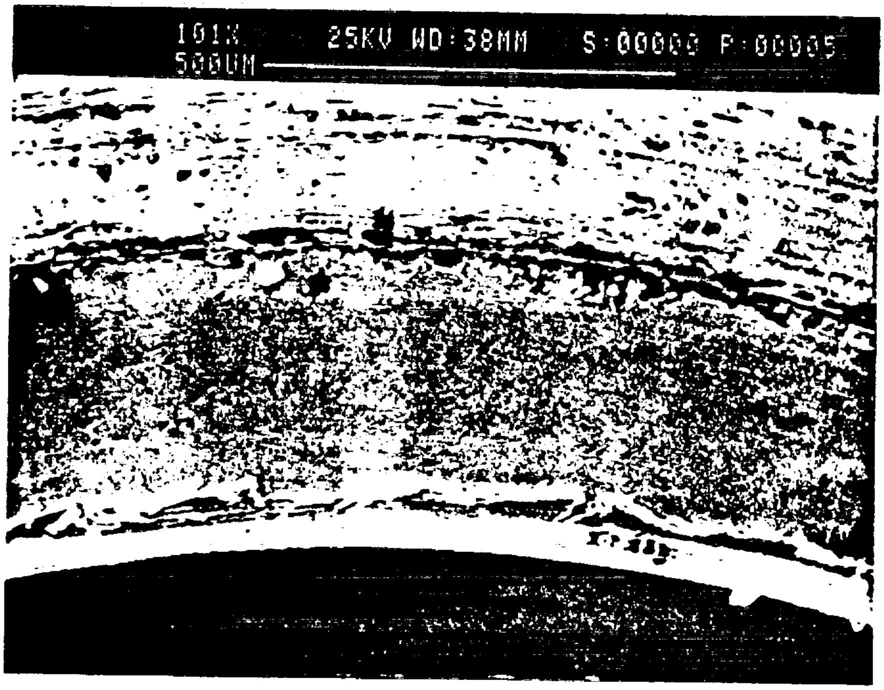
S.S.
Sample
#1
June
8
after
12
cycle

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-11b

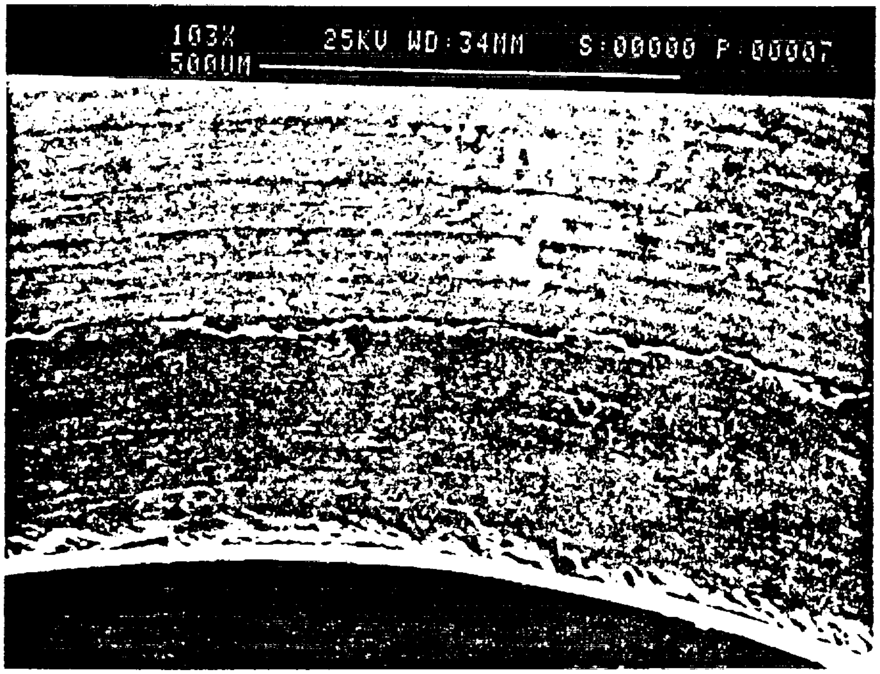
SEM/Stainless Steel/12 cycles/Blackstone

Stainless Steel
T = 0



S.S.
Sampl
#1
Jun
4
orig
Sam

Stainless Steel
T = 120 minutes

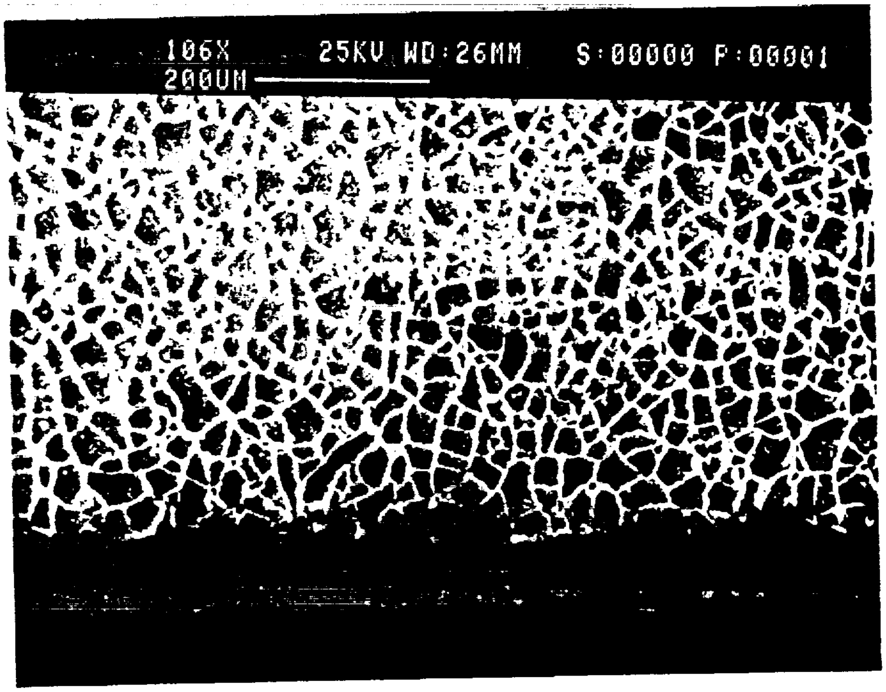


SS.
Sample
#1
Jun 8
after
12
cycle

Figure 3-12

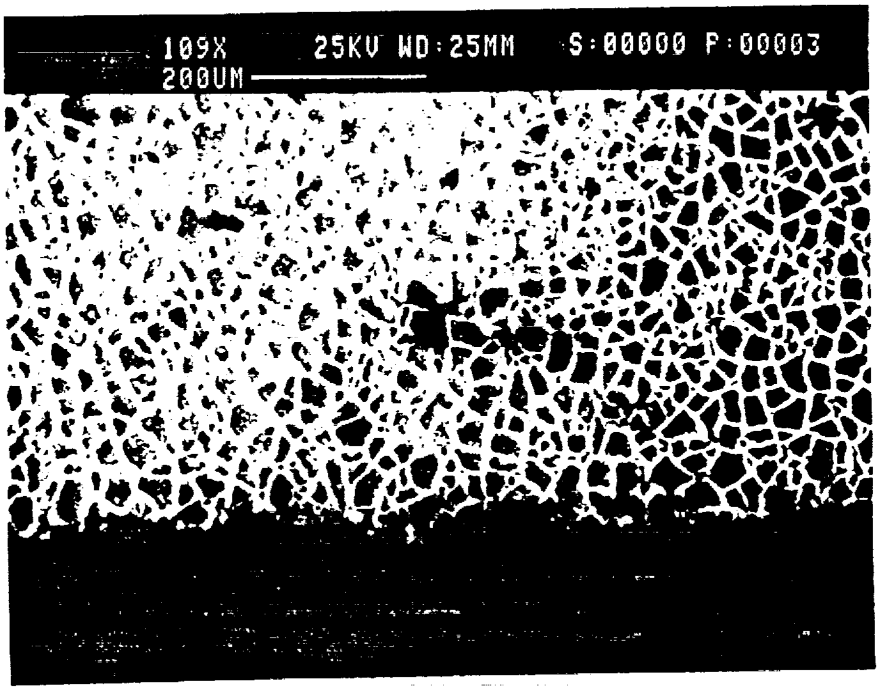
SEM/Anodized Aluminum/2 cycles/Sonic Systems

**Anodized
Aluminum
T = 0**



Al #152, T=0

**Anodized
Aluminum
T = 20 minutes**

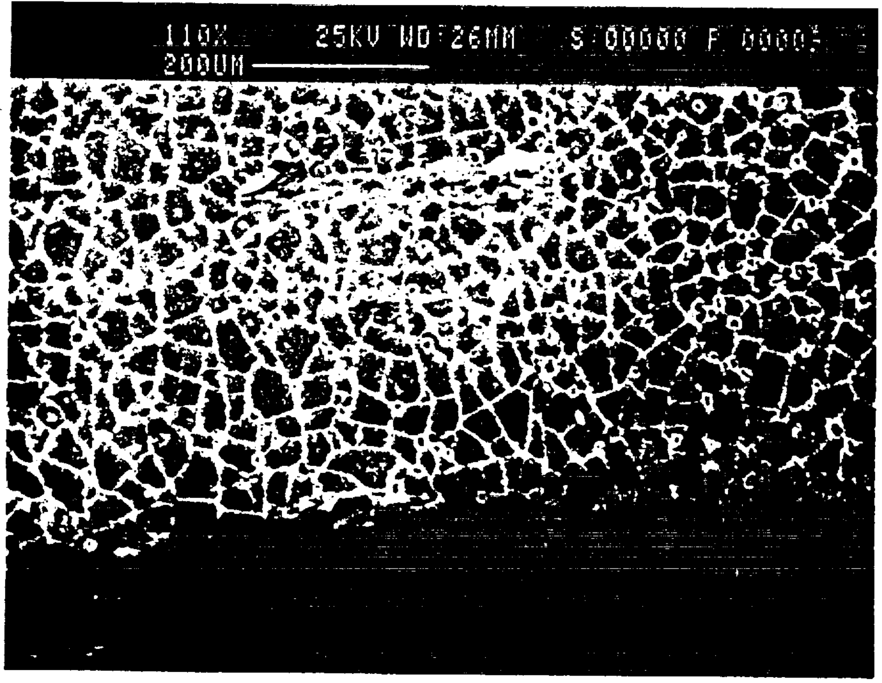


ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-13

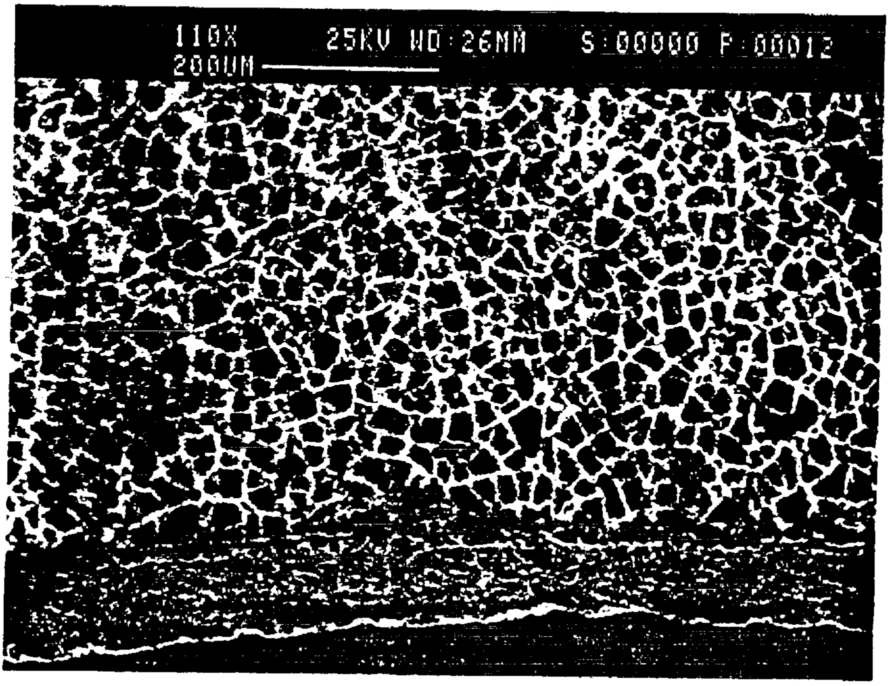
SEM/Anodized Aluminum/12 cycles/Sonic Systems

Anodized
Aluminum
T = 0



Al #152 T-0

Anodized
Aluminum
T =120 minutes



Al #157 12 cycles

ORIGINAL PAGE IS
OF POOR QUALITY

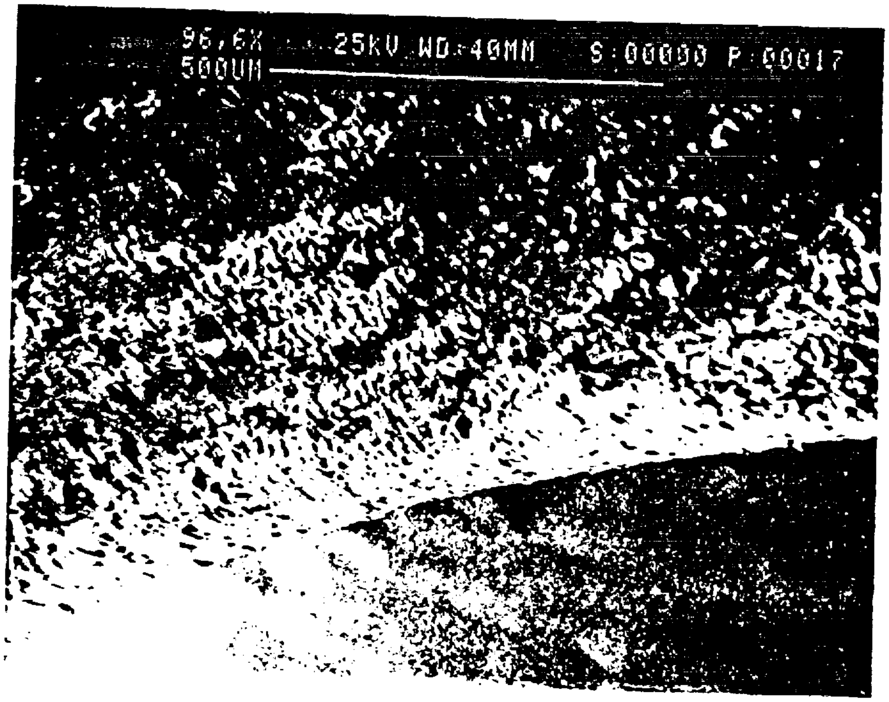
Figure 3-14

Optical Photograph of Water Residue (Anodized Aluminum Run)

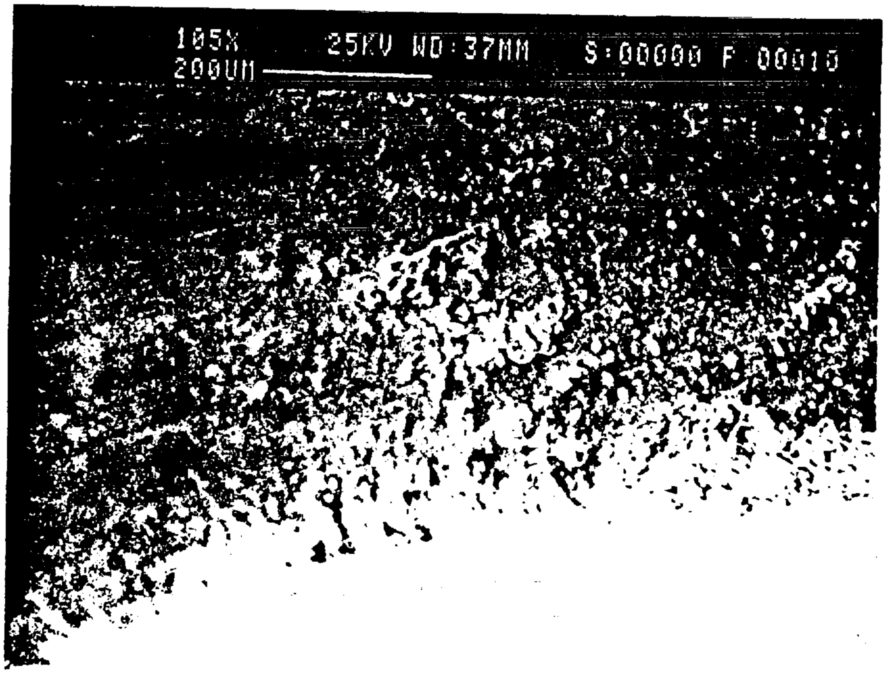
Figure 3-15

SEM/Brass/12 cycles/Sonic Systems

Brass
T = 0



Brass
T = 120 minutes

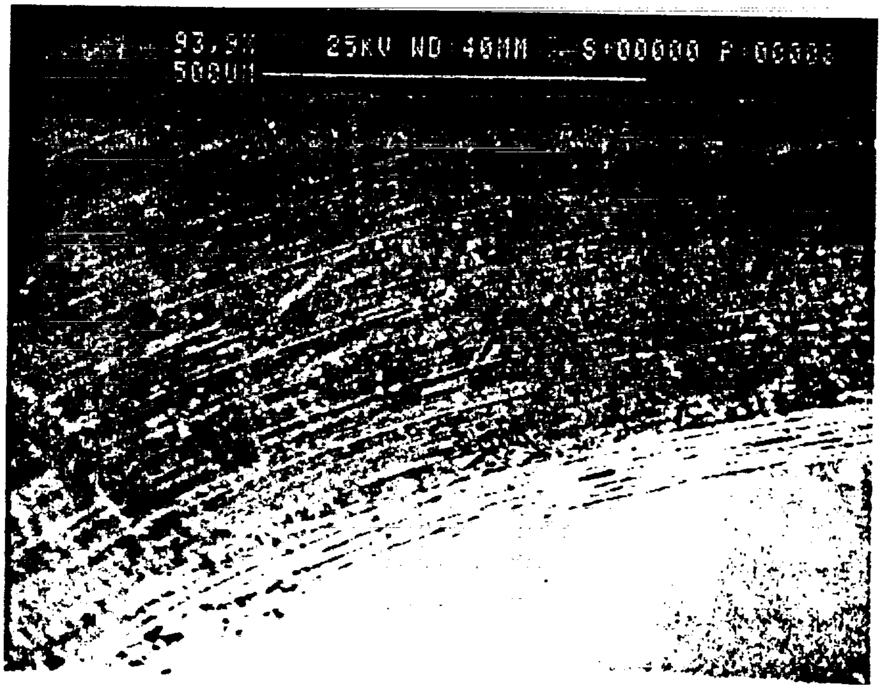


ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-16

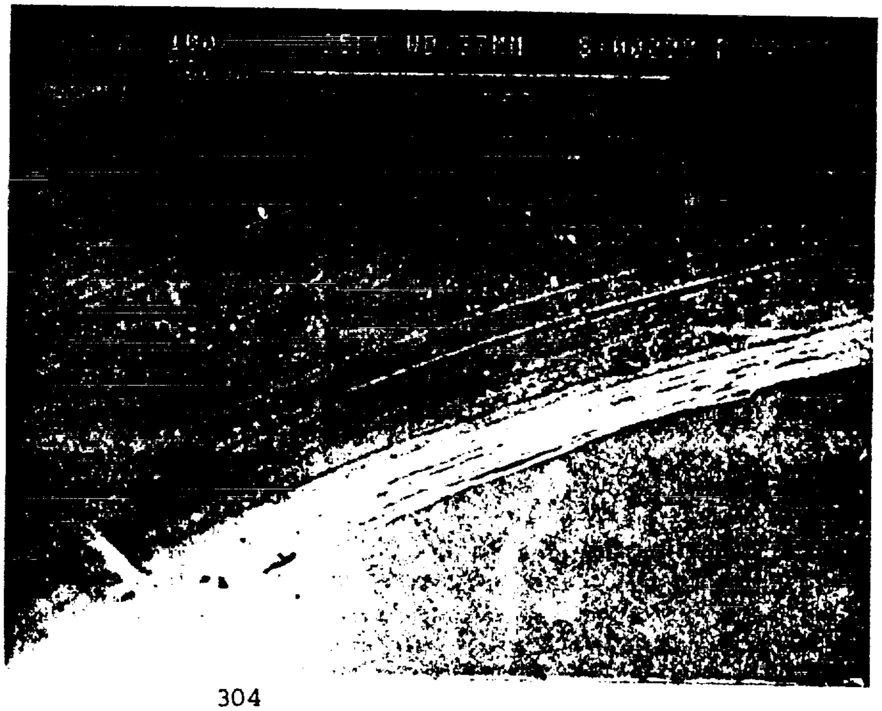
SEM/Stainless Steel/12 cycles/Sonic Systems

Stainless Steel
T = 0



SS #23

Stainless Steel
T = 120 minutes

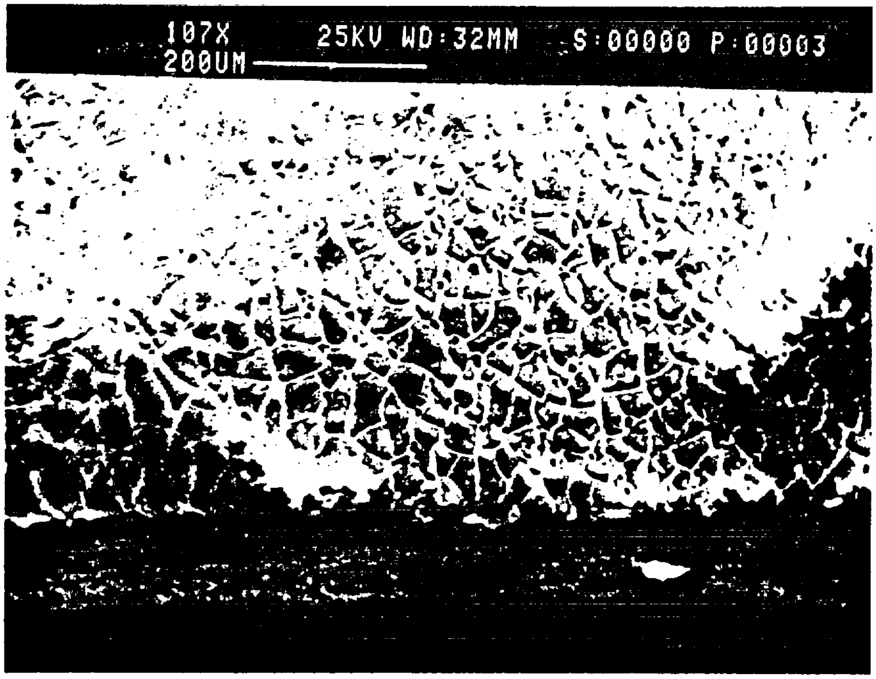


12 cycles

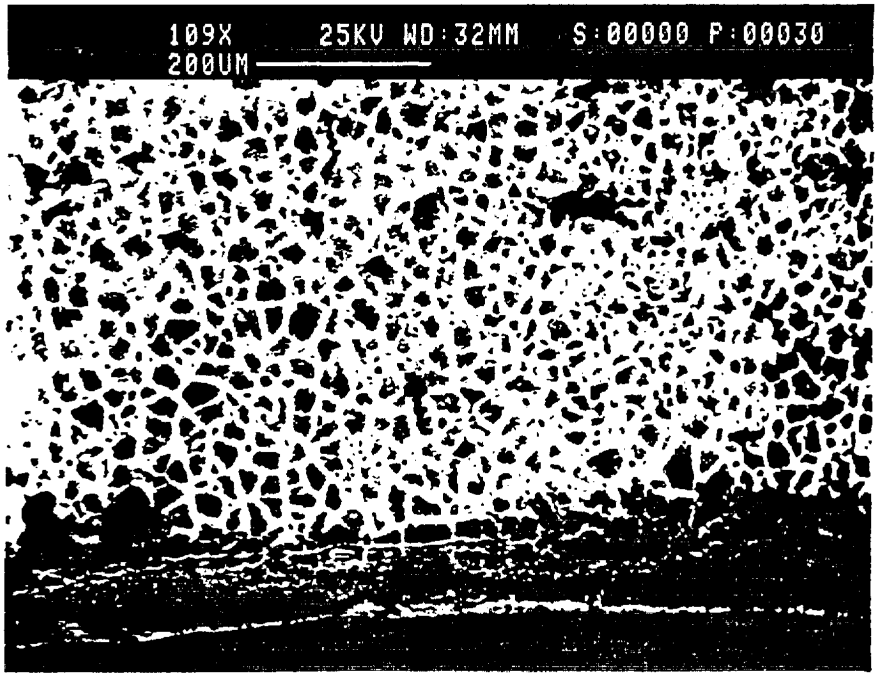
SS #29

Figure 3-17

SEM/Anodized Aluminum/4 cycles/Branson



Anodized
Aluminum
T = 0

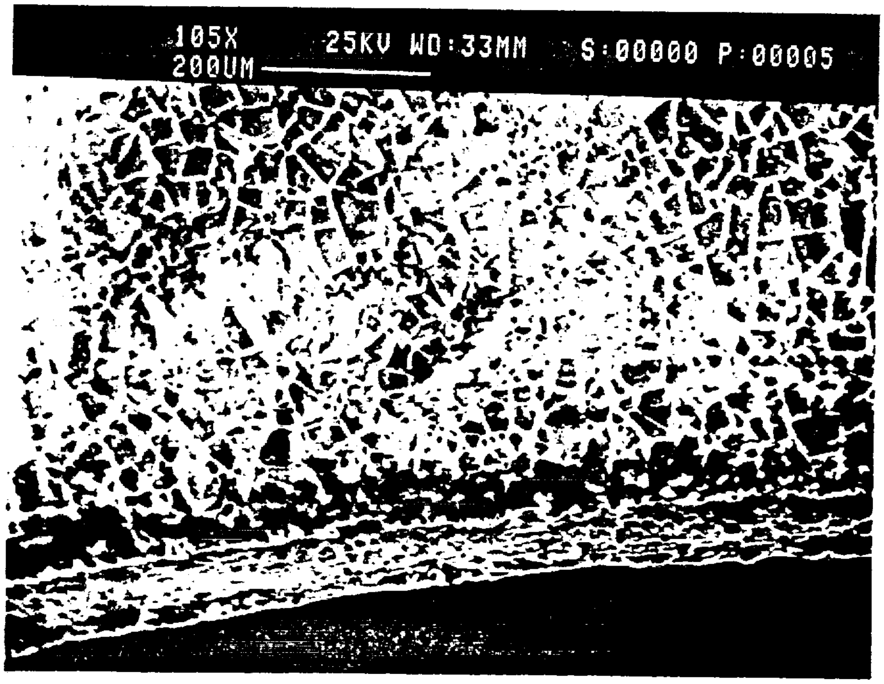


Anodized
Aluminum
T = 40 minutes

Figure 3-18

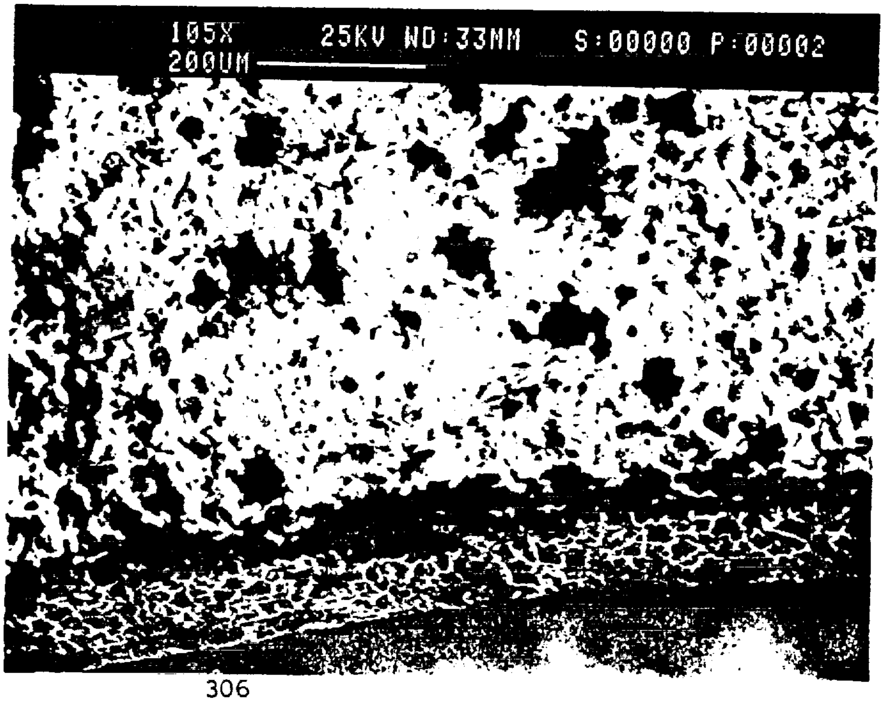
SEM/Anodized Aluminum/8 cycles/Branson

Anodized
Aluminum
T = 0



#307 T=0

Anodized
Aluminum
T = 80 minutes

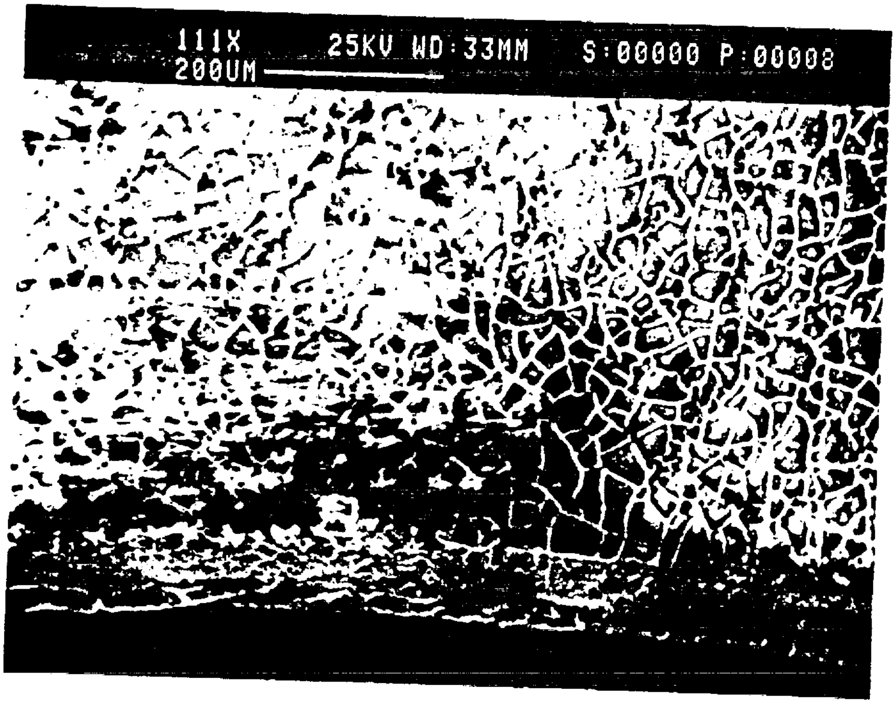


Al #307 8 cycles

Figure 3-19a

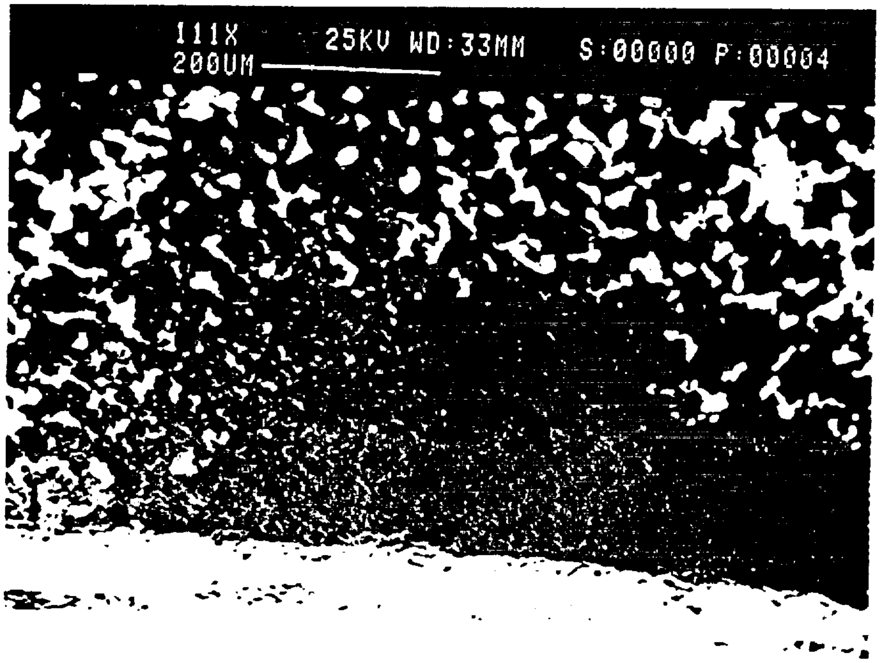
SEM/Anodized Aluminum/12 cycles/Branson

Anodized
Aluminum
T = 0



Al #308 T=0

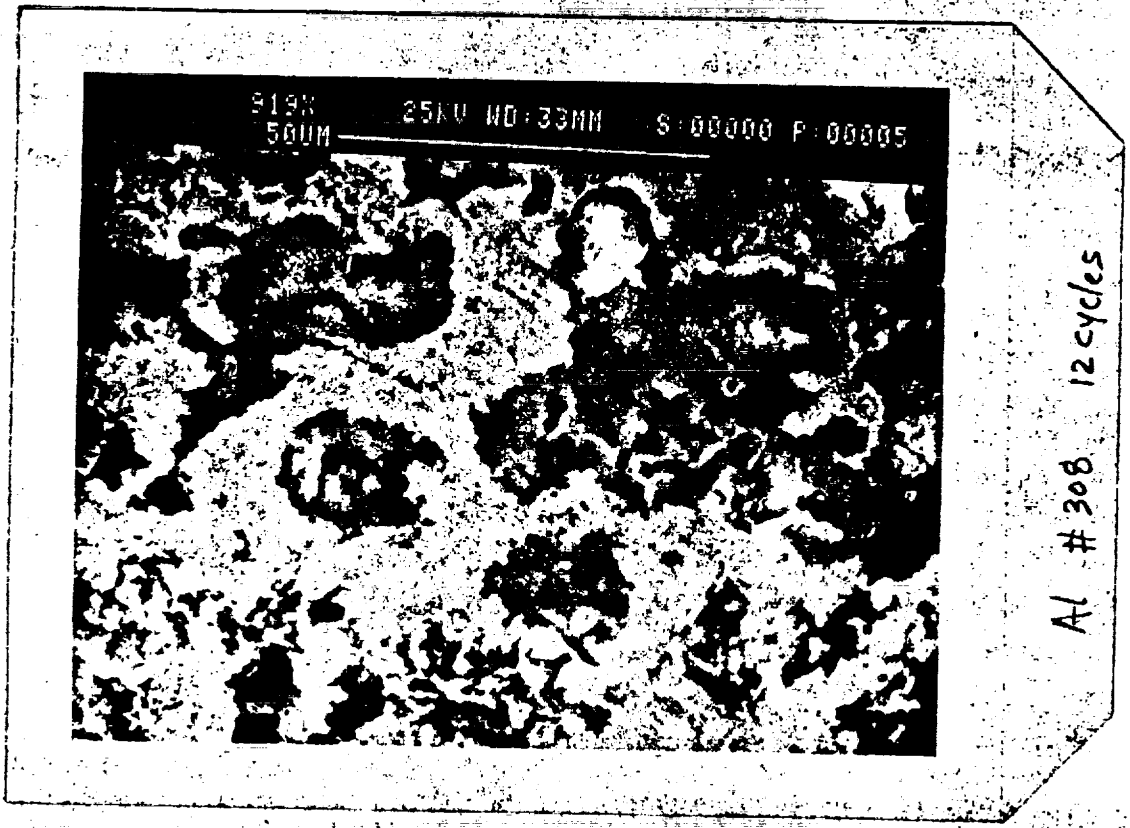
Anodized
Aluminum
T = 120 minutes



Al #305 12cycles

Figure 3-19b

SEM/Anodized Aluminum/12 cycles/Branson

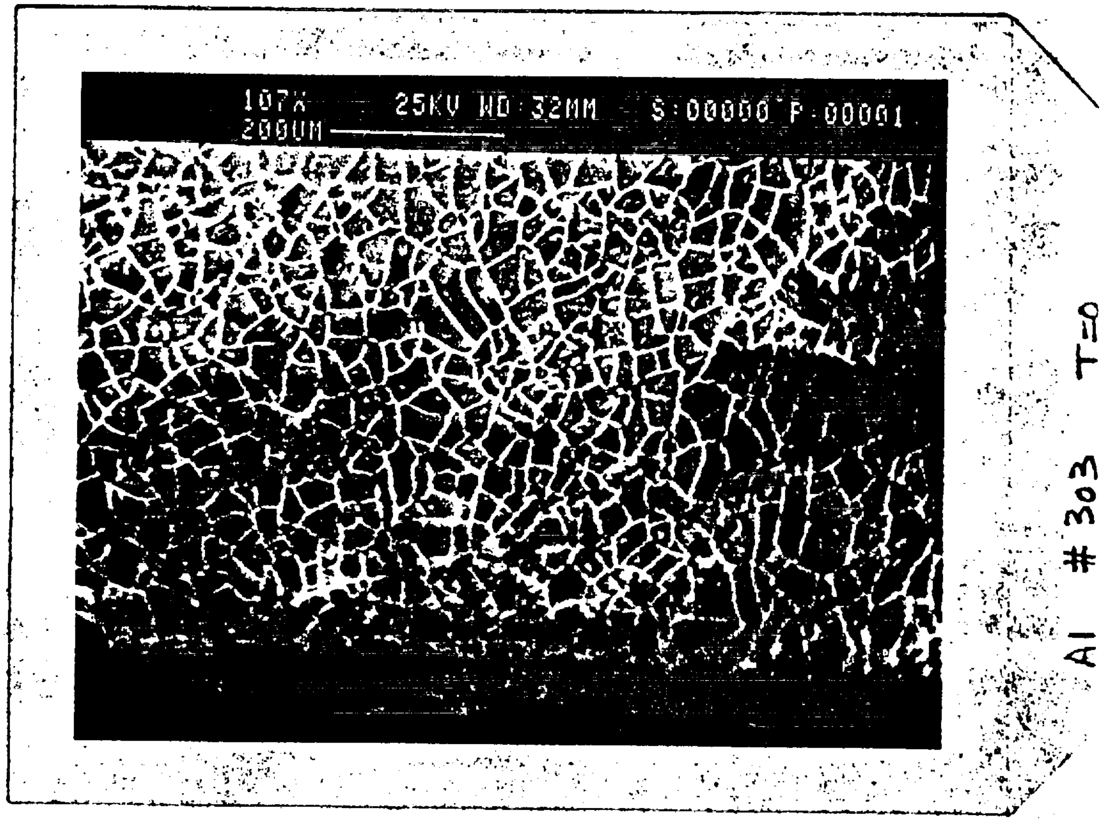


Anodized
Aluminum
T = 120 minutes

Figure 3-20

SEM/Anodized Aluminum/2 cycles/Branson

**Anodized
Aluminum
T = 0 minutes**



**Anodized
Aluminum
T = 20 minutes**

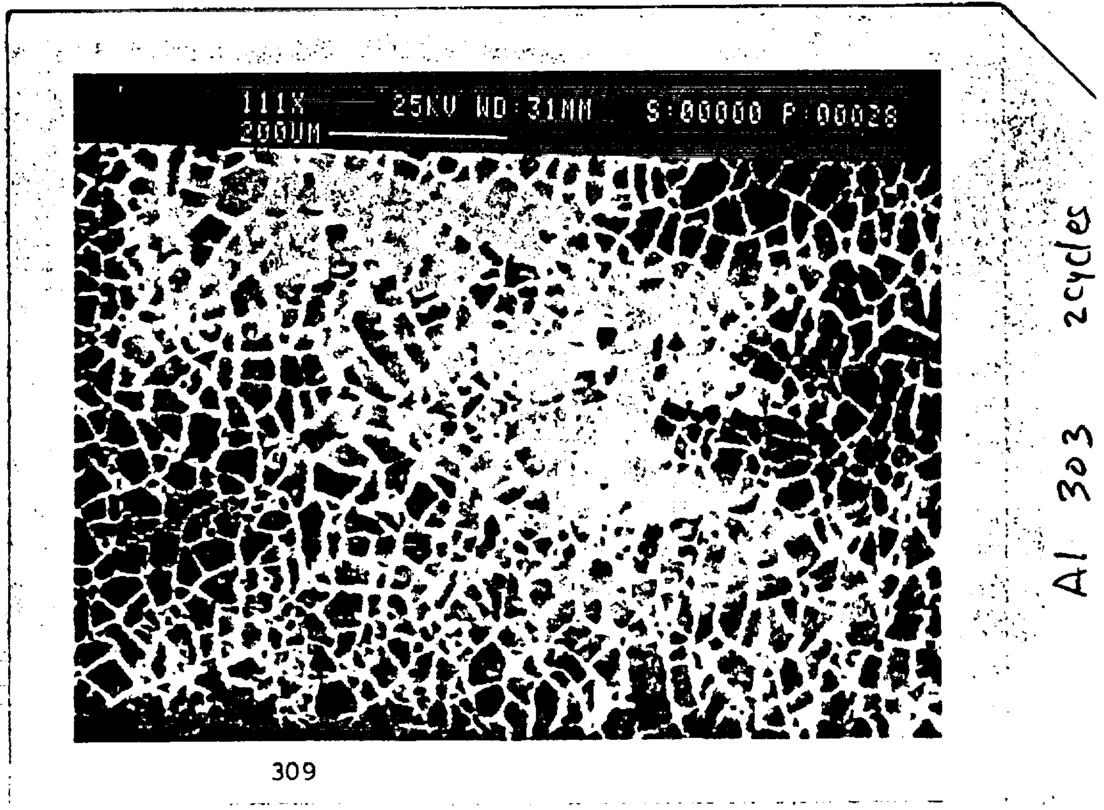
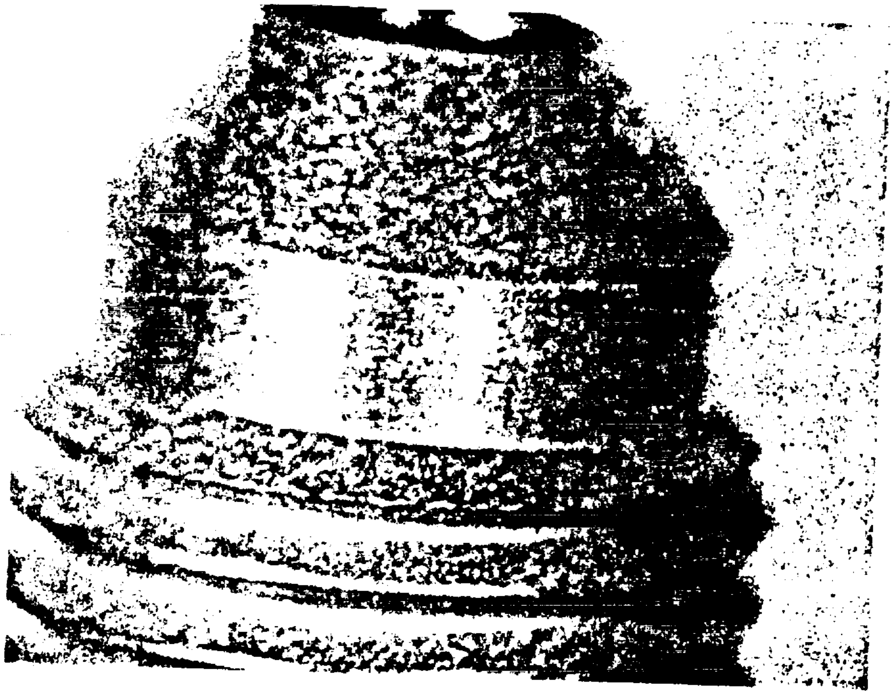


Figure 3-21

Optical Photograph/Anodized Aluminum/2- and 12 cycles/Branson

Anodized
Aluminum
T = 20 minutes



Anodized
Aluminum
T = 120 minutes

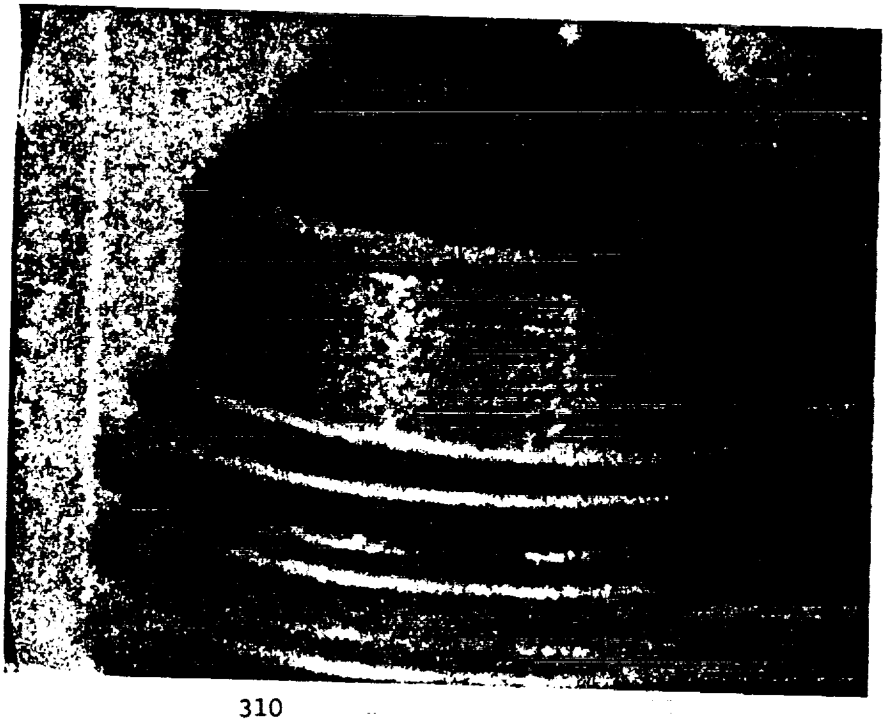
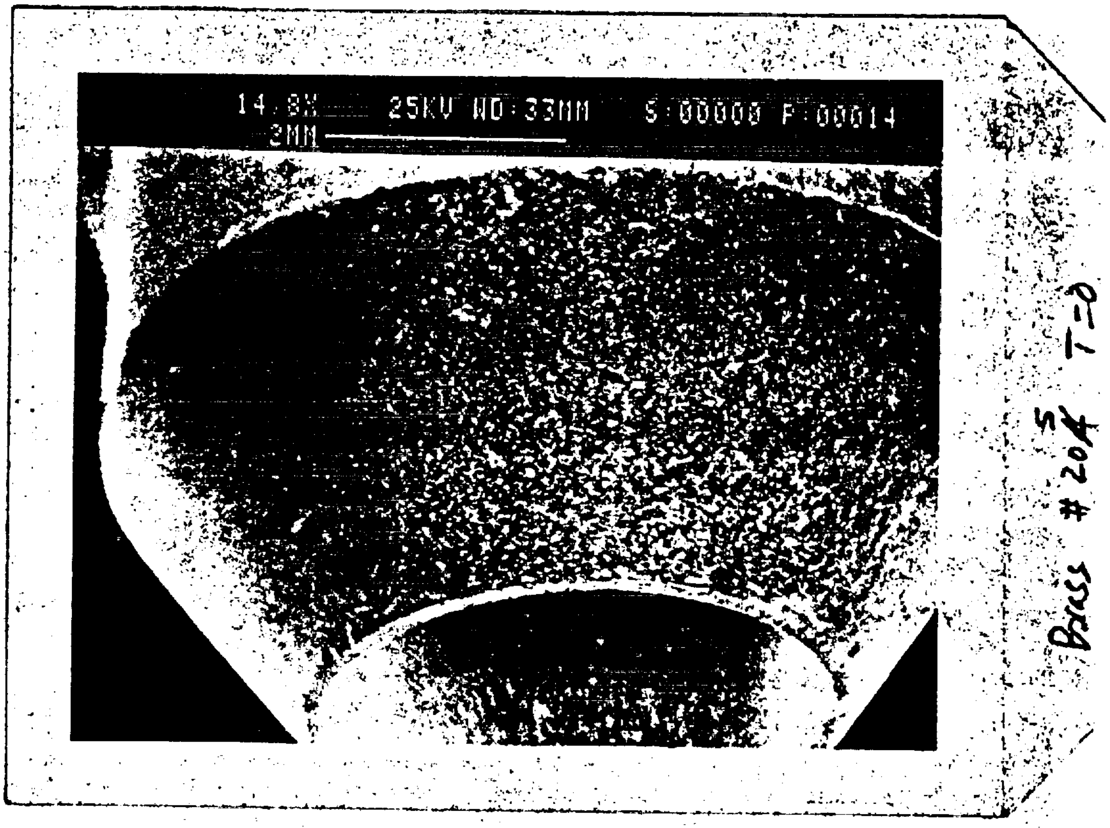


Figure 3-22a

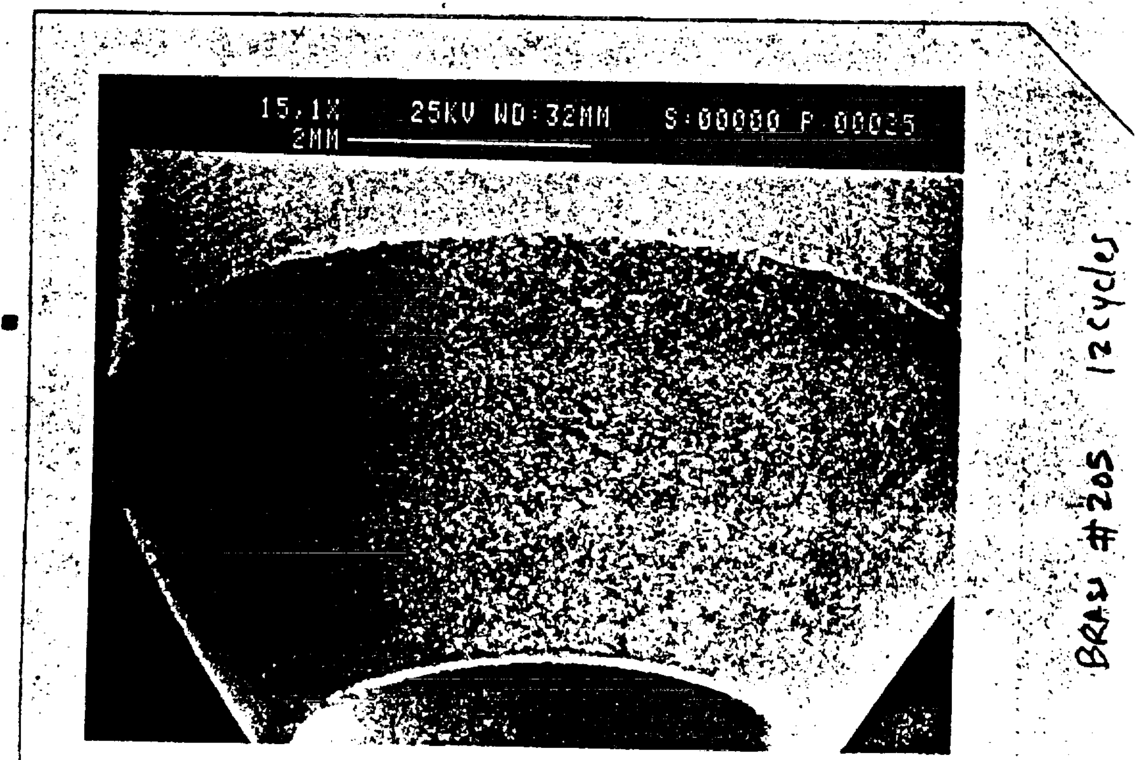
SEM/Brass/12 cycles/Branson

Brass
T = 0 minutes



Brass #20~~4~~5 T=0

Brass
T = 120 minutes



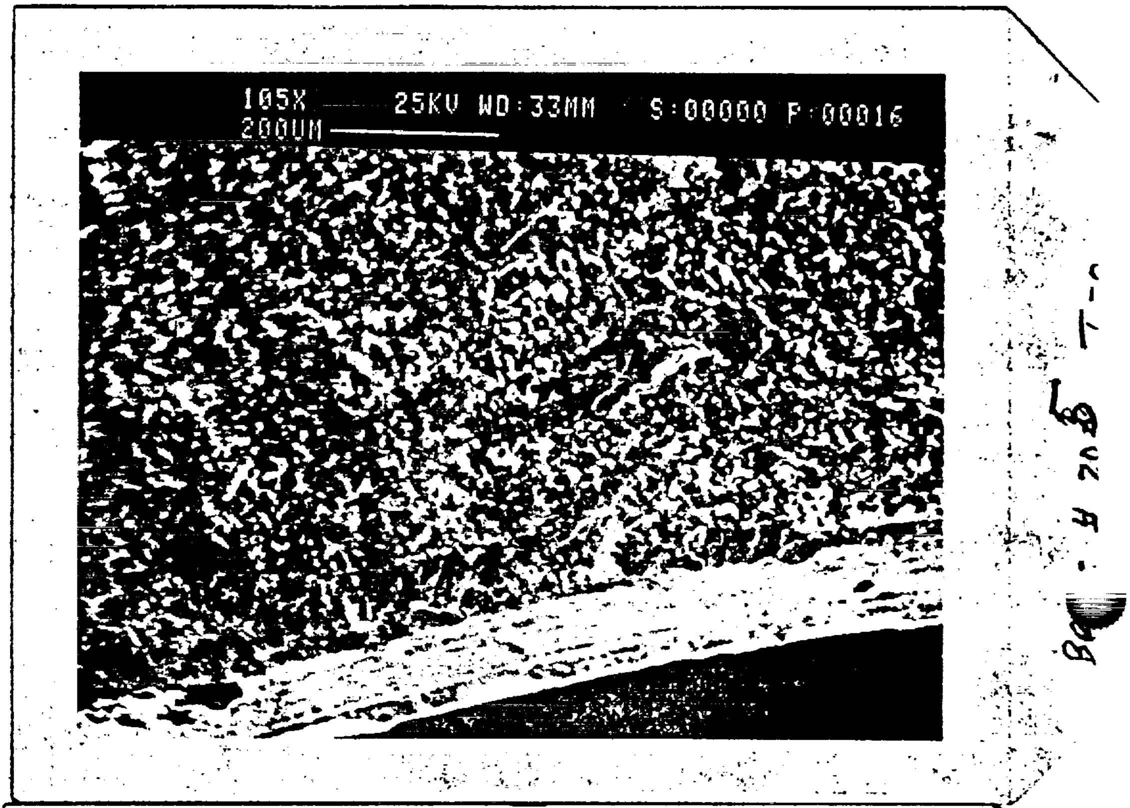
BRASS #205 12cycles

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-22b

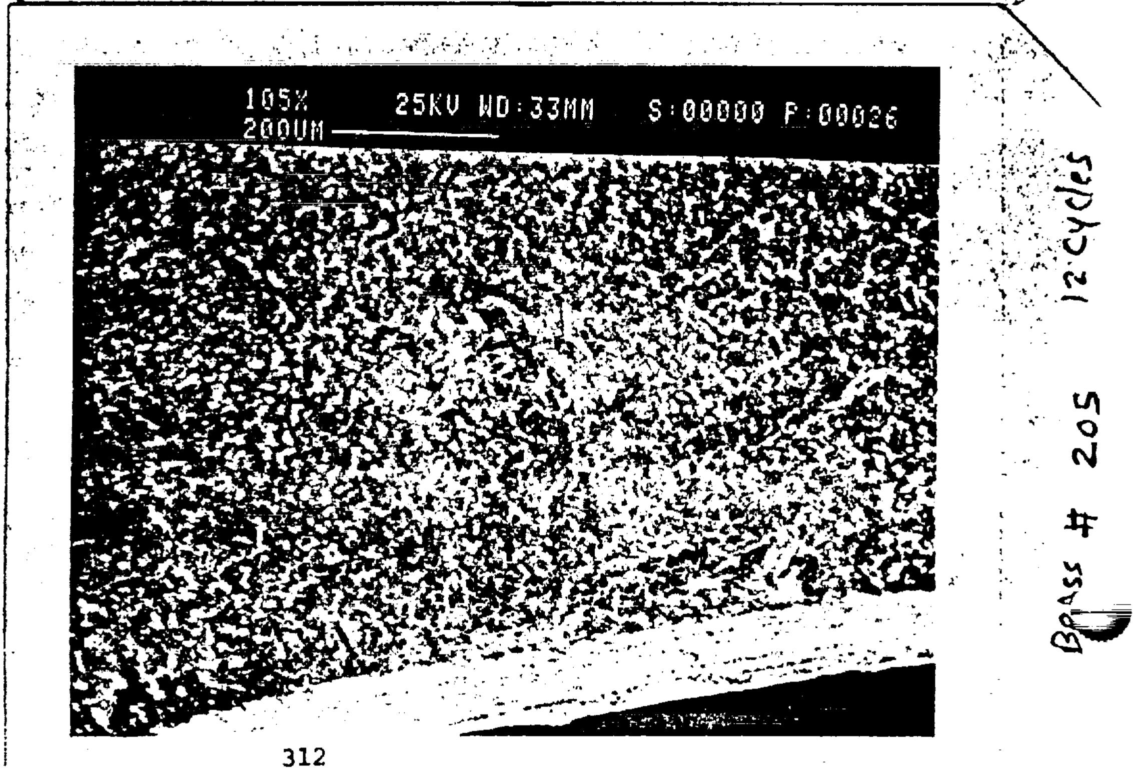
SEM/Brass/12 cycles/Branson

Brass
T = 0 minutes



B. # 205 T-0

Brass
T = 120 minutes



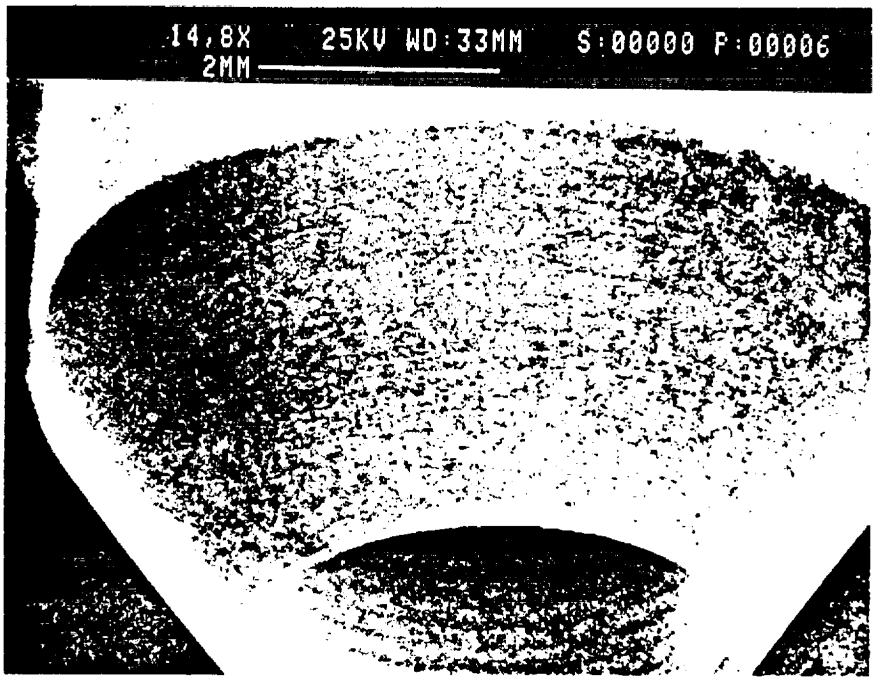
Brass # 205 12 cycles

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-23a

SEM/Stainless Steel/12 cycles/Branson

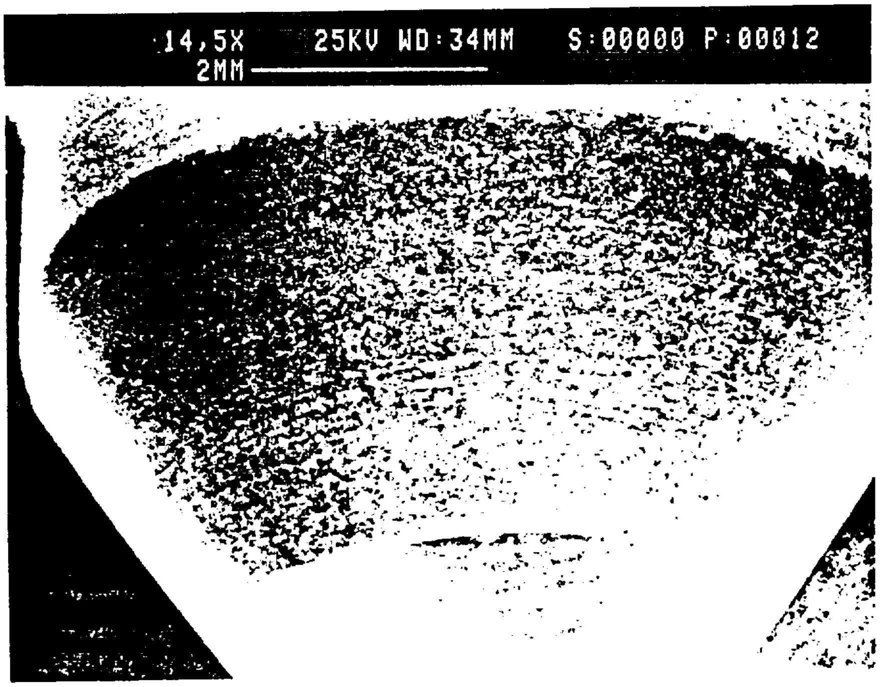
Stainless Steel
T = 0 minutes



RS

S.S #256 T=0

Stainless Steel
T = 120 minutes



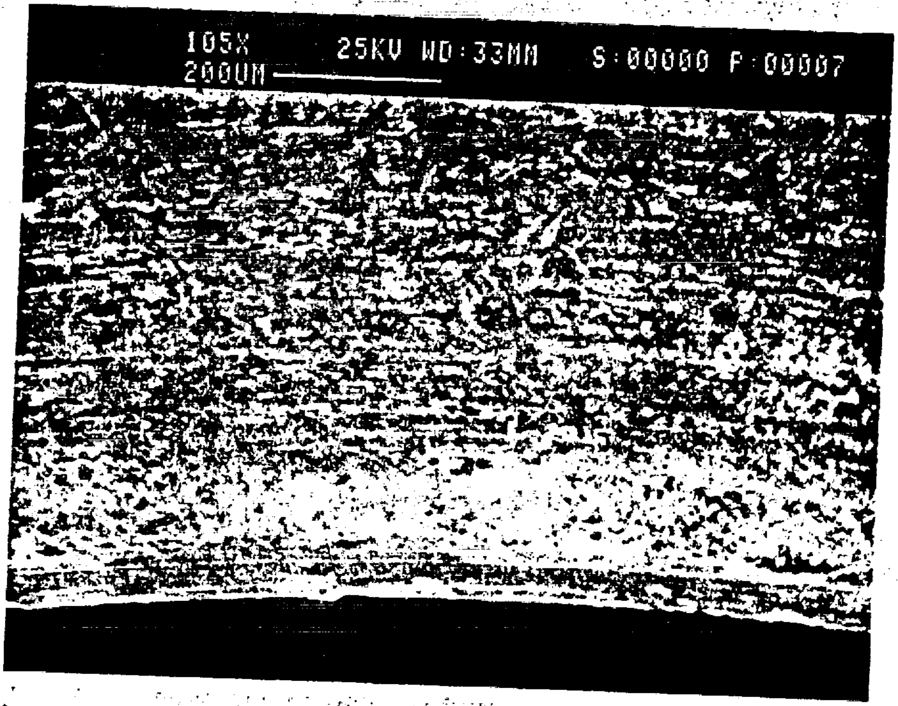
12 cycles
SS #256 after Sebray Cl_2CF_2

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-23b

SEM/Stainless Steel/12 cycles/Branson

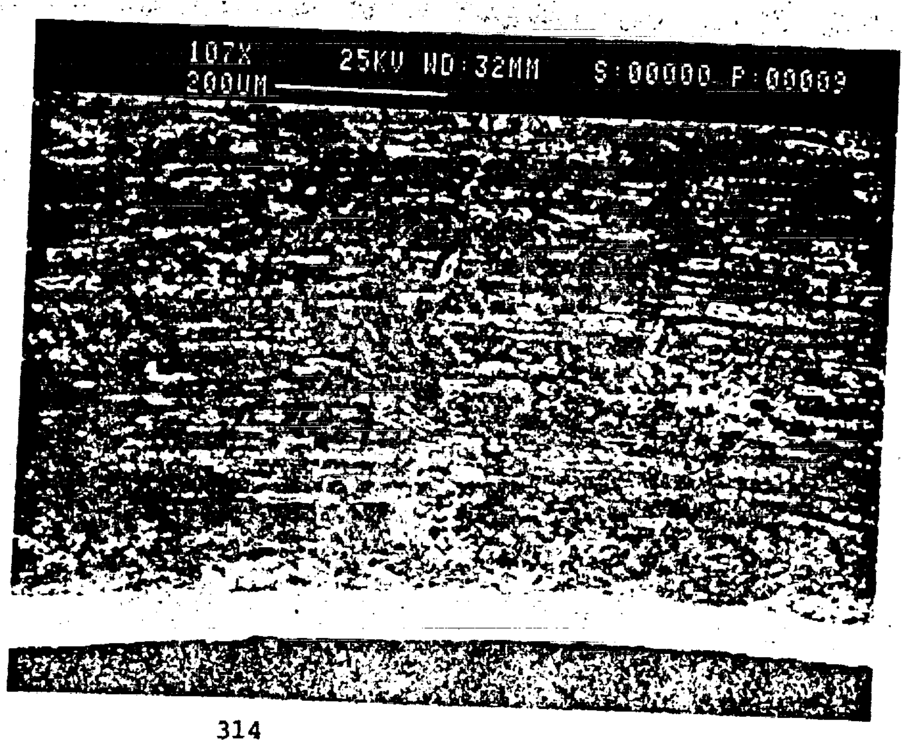
Stainless Steel
T = 0 minutes



R5

S.[illegible] T-[illegible]

Stainless Steel
T = 120 minutes

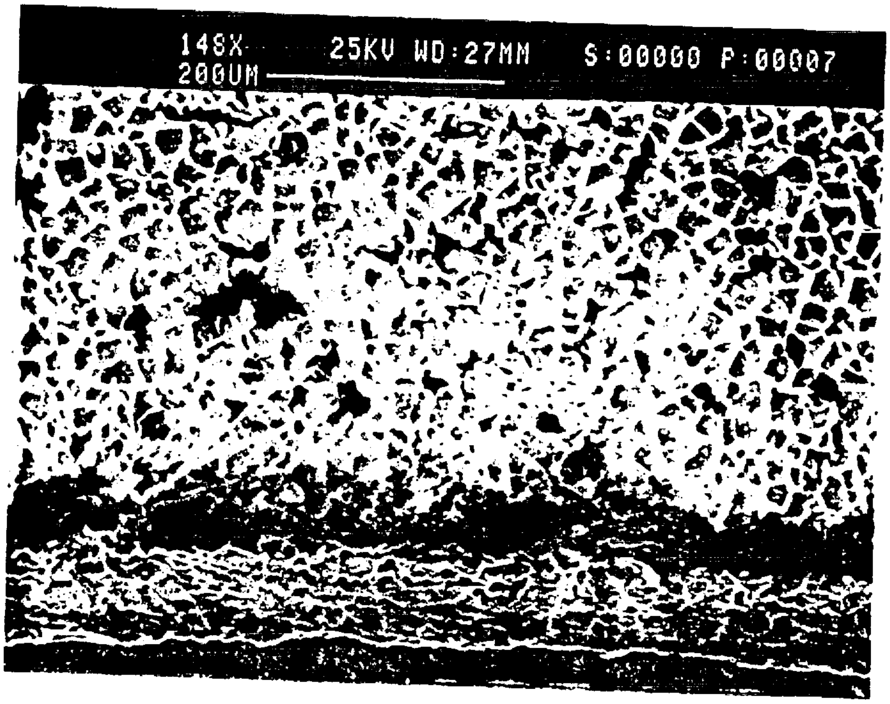


SS #256 12 cycles

Figure 3-24

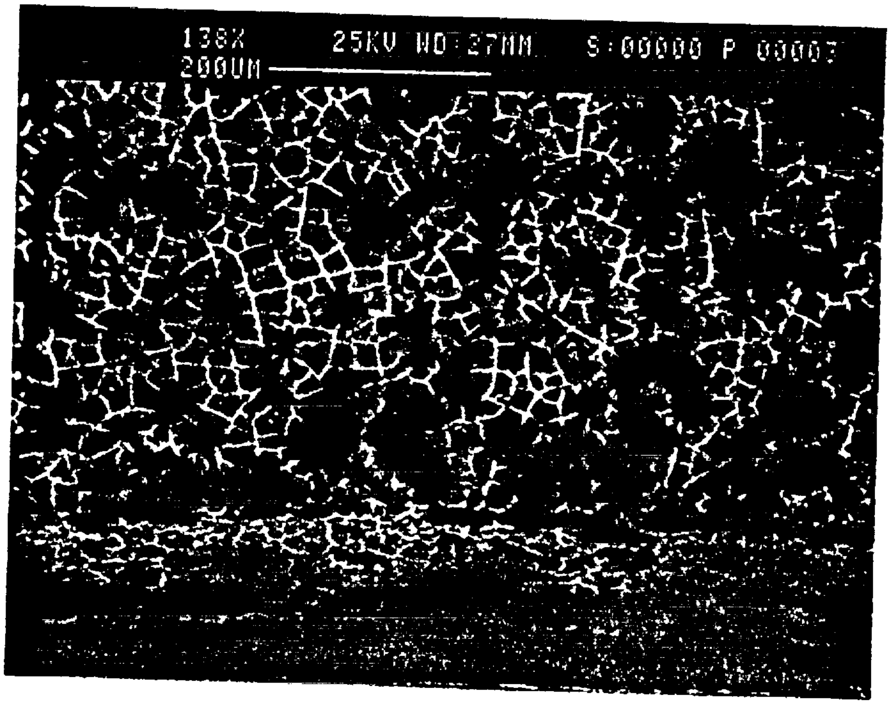
SEM/Anodized Aluminum/Blue Wave

Anodized
Aluminum
T = 20 minutes



Al = 5sec 2 cycles

Anodized
Aluminum
T = 40 minutes



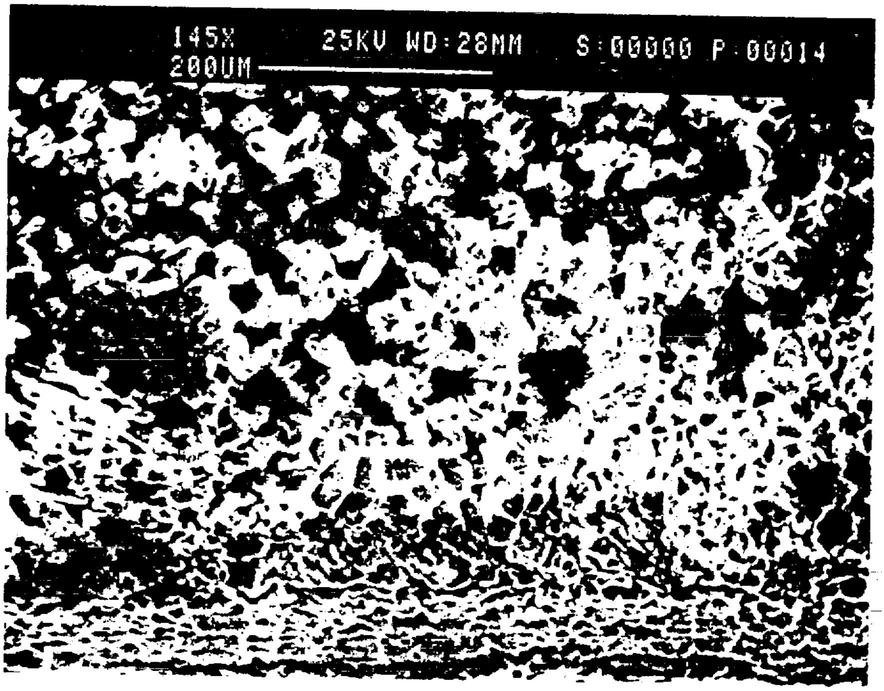
Al 4sec 4 cycles

ORIGINAL PAGE IS
OF POOR QUALITY

Figure 3-25

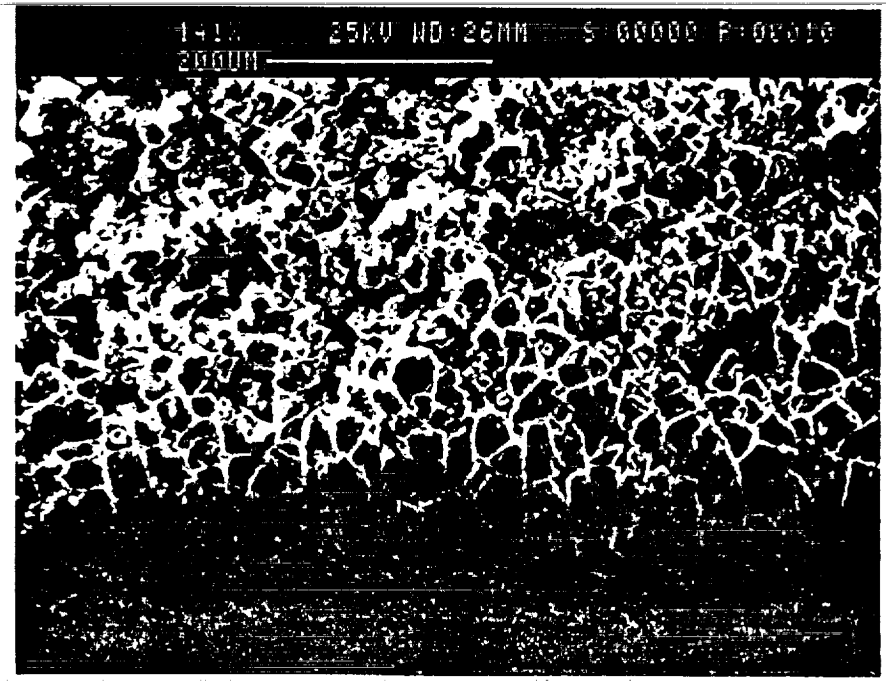
SEM/Anodized Aluminum/Blue Wave

Anodized
Aluminum
T = 80 minutes



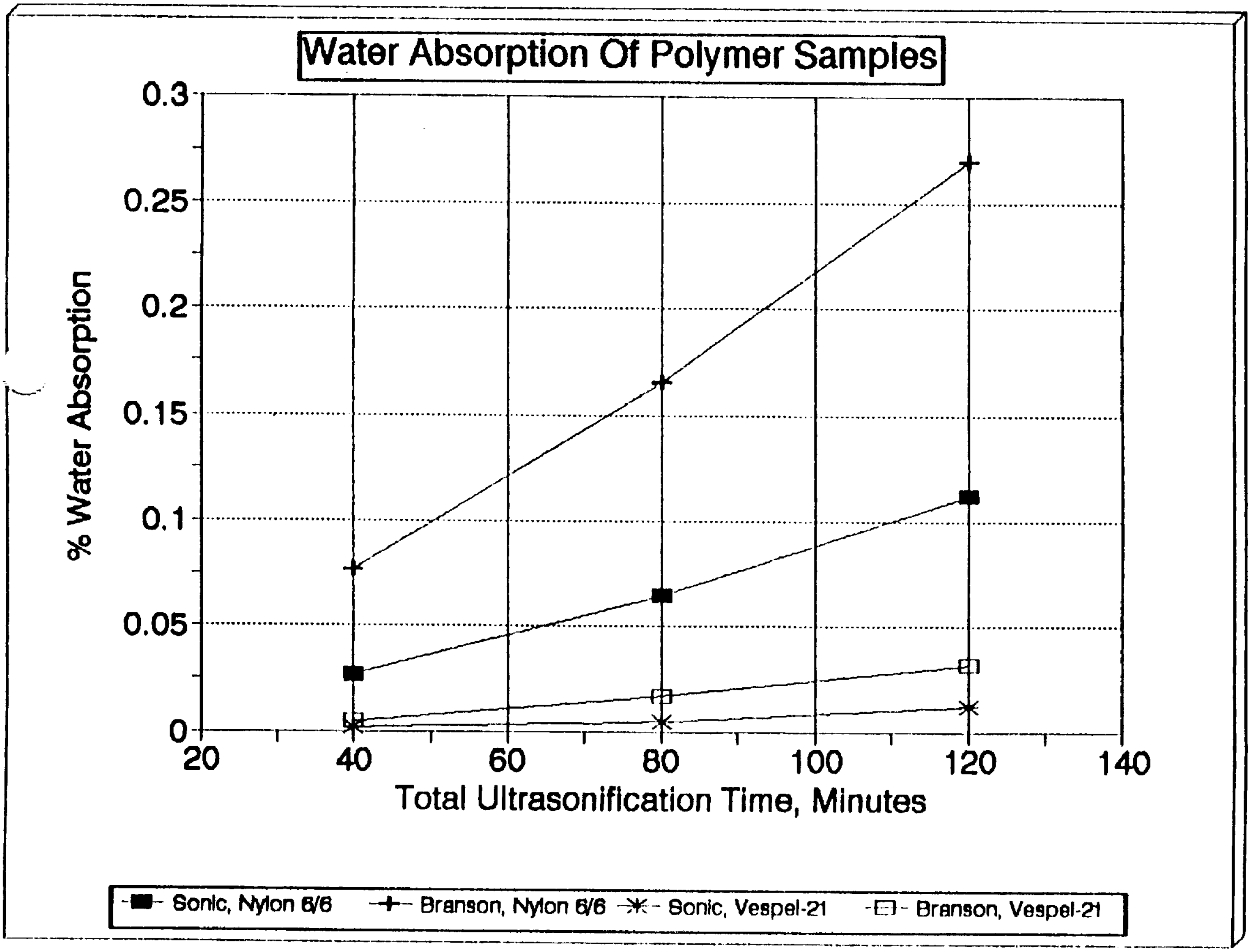
3 Series Al #10

Anodized
Aluminum
T = 120 minutes



Series #150

Figure 26


Water Absorption Of Polymer Samples
% Water Absorption
0.3
0.25
0.2
0.15
0.1
0.05
0
20
40
60
80
100
120
140
Total Ultrasonification Time, Minutes
■ Sonic, Nylon 6/6
+ Branson, Nylon 6/6
✳ Sonic, Vespel-21
□ Branson, Vespel-21