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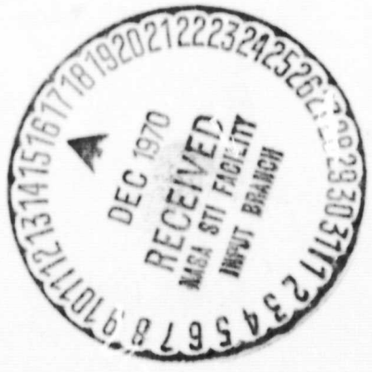

ETCHING OF METALLOGRAPHIC SPECIMENS BY CAVITATION

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INTRODUCTION

Cavitation da nage to materials occurs in many engineering applications where bubbles, formed by transient low pressures in moving liquids collapse rapidly on or near solid surfaces. A recent excellent review of the field of cavitation erosion, as well as erosion by solid and liquid impingement can be found in reference 1.

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Of the various possible methods of evaluating materials for resistance to cavitation damage in the laboratory, the ultrasonic vibration technique has become the most widely accepted. In 1968, the ASTM G-2 Committee on Cavitation by Erosion or Impingement coordinated a series of round-robin tests[2] in which several different types of ultrasonic vibration devices were used and in which eleven laboratories, including the Lewis Research Center, participated[3]. Recently, the author used a vibratory technique to

determine the cavitation damage in distilled water to several unalloyed metals and to one nickel-base superalloy[4]. This paper describes those portions of the investigation of ref. 4 which relate to the metallographic aspects of that investigation.

Some of the unalloyed metals studied were too soft to withstand the forces generated within the specimen when it was ultrasonically vibrated. Therefore an alternate technique was used in which the specimens were held stationary beneath an ultrasonically vibrated head, and cavitation damage was caused to the specimen by the cavitating cloud formed between the vibrating head and the stationary specimen. Similar types of arrangements have been used by other investigators[5].

During the course of this investigation, various aspects of the stationary specimen method of ultrasonic vibratory testing were studied. For example, the degree to which cavitation damage was affected by the distance between the stationary specimen and the vibrating head was determined. Also, the effect on cavitation damage of particles trapped within the cavitating cloud was determined. Metallographic studies were made which in-

volved obtaining macrographs, photomicrographs, and electron microscope replica photographs of the tested materials at various stages of cavitation testing. The materials studied were the unalloyed metals, zinc, nickel 270 (Ni-270), iron, and tantalum, as well as the nickel-base alloy, Udimet 700. All test conditions were the same as those specified for the ASTM round robin[3].

MATERIALS, APPARATUS AND PROCEDURE

Materials

The materials investigated for cavitation damage in water were the unalloyed metals, zinc, as-received NI-270, annealed Ni-270, iron, and tantalum, and Udimet 700, a nickel-base superalloy. The chemical compositions and conditions of heat treatment of the materials, are listed in table I. All heat treatments were performed after machining of the specimens unless otherwise stated. Physical properties of the metals are listed in table II.

Cavitation Damage Apparatus

A schematic diagram of the apparatus used is shown in figure 1, and a

more detailed schematic diagram of the specimen and holder assembly is shown in figure 2. A magnetostrictive transducer was used to vibrate a rod with its free end immersed in distilled water. This end of the vibrating rod, called the vibrating head, was detachable and made from L-605, a moderately cavitation-damage-resistant material. The head was replaced three times during the entire program, although very little damage was noted to the L-605. The test specimen, shown in the figure, is mounted directly below the vibrating head. Cavitation bubbles induced in the water by vibration collapsed on the face of the stationary specimen where they caused damage.

As shown in figure 1 a magnetic pickup was used to monitor the vibration amplitude. A feedback signal from the magnetic pickup was used to control the transducer input frequency to match the natural resonant frequency of the transducer assembly which was approximately 25 000 hertz. Level and translational adjustments and a contact circuit were used to position vibrating head and specimen surfaces and in obtaining parallel meas-

ured gaps between the specimen and the vibrating head. Water temperatures were maintained constant by a water circulator capable of either heating or cooling the distilled water test fluid.

Specimens

Several different types of test specimens were used in these cavitation damage experiments. They are shown in figure 3. The threaded specimens shown in figures 3(a) and (b) were designed for earlier experiments in which the specimens were vibrated; however, the soft zinc specimens could not withstand forces generated in the threads by vibration. Therefore, all specimens were tested in the stationary position explained in the previous section. Positioning the specimens this way permitted greater freedom in specimen size and design. One specimen each of zinc and Udimet 700 shown in figures 3(c) and (d) was not threaded and was larger in diameter than others to study the effect of specimen diameter on the damage pattern. The surfaces of all specimens were polished metallographically before test except for as-received nickel 270. These specimens were ground to a 600 paper finish.

Test Conditions

The test conditions for cavitation damage were made to conform with those previously specified by the ASTM for the round-robin tests in which eleven laboratories participated[2]. The primary exceptions to the round-robin conditions were that specimens were held stationary and face up under a vibrating head.

All tests were made in distilled water at $75\pm1^{\circ}$ F (24° C). Local atmospheric pressure was 29.17 ± 0.25 inches of mercury (1×10^{5} N/m^{2}).

The total displacement (double amplitude) of the vibrating head was 0.00175 ± 0.00005 inch (4.45×10^{-2} mm). The suggested amplitude for the round-robin tests was 0.002 inch (5.1×10^{-2} mm). The amplitude of 0.00175 inch (4.45×10^{-2} mm) was used in our tests of both vibrated and stationary specimens because of limitations of the equipment at the high frequency used. The nominal frequency of vibration was 25 000 hertz.

The distance between the specimen and vibrating head was held at 0.015 inch (0.038 cm) for all tests except those which were made to determine the effect of separation distance on cavitation damage.

Test Procedure

Specimens were cleaned in distilled water and alcohol and air dried; then they were photographed and weighed. After the test bath was brought to the desired temperature, specimens were securely placed in the specimen holder, and the specimen and holder were placed into the test bath. The specimen assembly was then adjusted to place the specimen surface directly below the L-605 head and to assure parallel surfaces between the specimen and head. The specimen assembly was then raised until contact was made with the L-605 head as indicated by a contact circuit light. Then the specimen was backed away to the desired separation distance for the test. Distance was measured to the nearest 0.001 inch (0.025 mm) by a dial on the positioning table. Power was then supplied to the magnetostrictive vibrator and the specimens were subjected to cavitation for varying intervals. After each period of operation, the specimens were removed from the bath, cleaned, weighed, and photographed.

RESULTS AND DISCUSSION

Determination of Optimum Separation Distance Between Specimen and Vibrating Head

The cavitation damage to as-received Ni-270 at various separation distances between the specimen and vibrating head is plotted in figure 4. Volume loss is plotted against separation distance for several different cavitation exposure times. The maximum damage for tests of 480 minutes duration was observed at approximately 0.015 inch (0.038 cm). Therefore, this value was chosen as a "standard" gap to be used for tests of all other materials. For test times shorter than 480 minutes, a maximum in damage occurred at a separation of 0.020 inch (0.041 cm).

Comparisons of Materials

Cavitation damage results for all materials are shown in figure 5. Because the curves for zinc and Udimet 700 are separated by three orders of magnitude, it was necessary to plot the volume loss on a logarithmic scale to include both materials on the same plot. Zinc lost approximately

233 cubic millimeters during 105 minutes of test, while Udimet 700 lost only 2 cubic millimeters in 1140 minutes. Annealed Ni-270 was less resistant to cavitation damage than Ni-270 in the as-received condition. This probably resulted from the lower hardness of the annealed nickel when compared to the as-received nickel. After about 400 minutes, the iron specimen showed volume loss results which fell between those of the as-received and annealed nickel specimens. Tantalum was the most resistant of the unalloyed metals that were tested.

Metallography

Comparisons of tested specimens. - Macrographs were taken of all the specimens tested. These are included in figures 6 to 8. Figure 6 shows the damaged surfaces of the as-received Ni-270 specimens. These were exposed to cavitation damage in water at 75° F (24° C) for times up to 480 minutes and for separation distances from 0.005 to 0.025 inch (0.013 to 0.064 cm). In figure 6 the texture of the damaged surface is fine at 0.005 inch (0.013 cm) and becomes successively more coarse at increasing distances up to 0.025 inch (0.064 cm), the maximum considered. These ob-

servations were confirmed by actual surface roughness measurements.

The damaged surfaces of the materials compared at a separation distance of 0.015 inch (0.038 cm) are shown in figure 7. Two zinc specimens of different diameters (0.562 and 1.25 in. or 1.43 and 3.18 cm) were tested. From figures 7(a) and (b) the pattern of damage to both specimens was approximately the same. Nickel (fig. 7(c)) showed a more uniform damage pattern than the other materials. During the early stages of testing, iron (fig. 7(d)) and tantalum (fig. 7(e)) showed several large pits (probably weaker areas or inclusions in the specimens). These tended to widen with increasing test time. Udimet 700 (fig. 7(f)) showed only an etch effect even after 1140 minutes of testing.

Effect of abrasive particles on damage. - An additional experiment was conducted to answer the question: "Do particles which break off from the specimens during tests continue to damage the surface by means of an 'ultrasonic drilling' effect?" The results of this study are shown in figure 8. Two specimens of 316 stainless steel were polished metallographically to a flat mirror finish. One of these is shown in the upper left

of figure 8. The other polished specimen was covered with 100 mesh particles of aluminum oxide and carborundum immediately before exposure to cavitation. This specimen is shown in the lower left of figure 8. Both specimens were subjected to 8 minutes of cavitation exposure. The photograph on the upper right shows that the specimen without particles experienced damage in the form of randomly distributed pits. The photograph on the lower right, of the specimen run with particles, shows a more pronounced damaged area in the center of the specimen as well as random pits; however, no abrasive particles were left on the specimen after the run was stopped. Because no central damage pattern appeared in any of the material evaluation tests, it was concluded that particles resulting from cavitation damage were expelled shortly after being dislodged from the specimens, and thus had no effect on the damage. In addition, the particles dislodged from specimens, having the same hardness as the specimens themselves, would have even a smaller tendency to cause damage than the abrasive particles used in this experiment.

Effect of cavitation damage on metal surface. - Photomicrographs of the damaged surface of the unalloyed metals during early stages of cavitation damage are shown in figure 9. Each material was chemically etched after polishing to obtain the micrograph of the untested material in the upper left of each figure. It was repolished before commencing the cavitation exposure.

In the early stages of damage, zinc, which is hexagonal close packed (hcp), showed parallel striations in one direction in each grain (fig. 9(a)). These may be traces of the basal (0001) plane revealed by mechanical etching. Similar striations were observed by previous investigators[6] after chemically etching a single crystal of zinc cleaved on a twinning plane (1012). After extended cavitation damage, individual grains of zinc were removed and cleaved faces were observed.

The face-centered cubic (fcc) nickel specimens were relatively fine grained and had a number of annealing twins. This soft material was rapidly damaged, and after only 0.5 minute (fig. 9(b)) linear features which were probably due to the presence of annealing twins were barely discernible.

At longer times the surface exhibited the "hills and valleys" pattern reported to be characteristic of soft nickel[6].

On a macro scale the body-centered cubic (bcc) iron specimens were similar in appearance to the fcc nickel specimens (figs. 7(d) and (c), respectively). However, on a micro scale, grain boundaries of the iron specimens (fig. 9(c)) were evident up to 30 minutes exposure. The pits and inclusions evident in the as-polished specimen tended to widen as cavitation damage progressed.

Tantalum (bcc structure) on both a macro and micro scale exhibited its grain structure in the early stages of damage. Boundaries were still evident in the tantalum specimen even after 90 minutes cavitation (fig. 9(d)). Although no inclusions or voids were evident in the polished and etched specimen, cavitation formed pits similar to those observed in the iron specimen. At longer test times the pits widened and joined with small linear damage features in the matrix.

Cavitation appears to be effective in revealing microstructural features of a complex alloy such as Udimet 700. Figure 10 is a replica

electron micrograph of such a microstructure showing features typical of a gamma prime strengthened superalloy. It was mechanically etched by 120 minutes exposure to cavitation.

In view of the previous findings, that is, preferential damage of unalloyed metals and mechanical etching of a nickel-base alloy, it is suggested that cavitation may be useful as a technique for the selective etching of materials for metallographic examination. The weaker phases would be removed, leaving the tougher, harder, impact resistant phases. This method would also allow the investigator to easily recover material from the distilled water (or any other fluid desired) for further analysis without the disadvantages associated with the use of reactive chemicals.

SUMMARY OF RESULTS

Specimens of several unalloyed metals (zinc, nickel, iron, and tantalum) and Udimet 700 were subjected to cavitation damage in water using a vibratory apparatus under conditions which were with one exception those established for earlier ASTM round-robin tests. In the present investigation the specimens were stationary instead of being vi-

brated and were placed beneath an ultrasonically vibrated head. The following results were obtained:

1. Preferential damage to metallurgical features in unalloyed metals and Udimet 700 in distilled water indicates that cavitation may be useful as a technique for mechanically etching materials for metallographic examination in situations where reactive chemicals would be undesirable.

2. When abrasive particles of aluminum oxide and carbides were intentionally placed between the vibrating head and a stationary specimen, increased damage was observed at the center of the specimen; however, no preferred damage such as this was observed when no abrasives were added. This result suggests that any metal particles dislodged during normal cavitation testing are probably ejected by the cavitating cloud and do not contribute further to specimen damage.

3. For as-received Ni-270, the one material tested at various separation distances between the specimen and vibrating head, a distance of 0.015 inch (0.038 cm) gave both the most constant damage rate over

the longest period of time and the highest damage for the total length of the test.

4. The relative ranking of materials, in order of decreasing volume loss after approximately 400 minutes of testing was zinc, annealed nickel 270 (Ni-270), iron, as-received Ni-270, tantalum, and Udimet 700. The volume loss of zinc and Udimet 700 were displaced by three orders of magnitude.

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2. Chao, C.; Hammitt, F. G.; King, C. L.; and Rogers, D. O.: ASTM Round Robin Test with Vibratory Cavitation and Liquid Impact Facilities of 6061-T 6511 Aluminum Alloy, 316 Stainless Steel, Commercially Pure Nickel. Rep. MMPP-344-3-T, 01357-4-T, Univ. Michigan, Nov. 1968.

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5. Brager, D.; Cheesewright, R.; Hammitt, F. G.; and Kemppainen, D. J.: Cavitation Erosion of a Stationary Specimer in Close Proximity to an Oscillating Surface. Rep. TR-08153-4-T, Univ. Michigan, May 1967. (Available from DDC as AD-816260.)

6. Plesset, M. S.; and Ellis, A. T.: On the Mechanism of Cavitation Damage. Trans. ASME, vol. 77, no. 7, Oct. 1955, pp. 1055-1064.

TABLE I. - CHEMICAL ANALYSIS AND HEAT TREATMENT CONDITIONS OF TEST METALS

Material	Heat treatment condition	Analysis, percent
Zinc[a]	Annealed spontaneously during extrusion process	99.997 Zn, 0.001 Pb, 0.0005 Cd, 0.0015 Fe
Ni-270[a]	Annealed in air at 900° F (482° C) for 1 hr; air cooled (A.C.)	99.981 Ni, 0.01 C, 0.001[b] of each of Si, Mn, Fe, S, Cu, Cr, Ti, Mg, Co
Ni-270[c]	As received - no annealing treatment after machining	99.98 Ni, 0.005 C
Iron[a]	Vacuum annealed at 1750° F (954° C) for 3 hr; furnace cooled	99.842 Fe, 0.025 C, 0.054 Mn, 0.006 P, 0.011 S, 0.062 Cu
Tantalum[a]	Vacuum annealed at 2350° F (1288° C) for 1 hr	99.845 Ta, 0.01 W[b], 0.01 Fe[b], 0.001 C, 0.01 Si[b], 0.005 Ni[b], 0.10 Cb, 0.01 Ti[b]
Udimet-700[a]	2135° F (1168° C) for 4 hr; A.C. 1975° F (1079° C) for 4 hr; A.C. 1550° F (843° C) for 12 hr; A.C. [d]1550° F (843° C) for 12 hr; A.C. 1400° F (760° C) for 16 hr; A.C.	42.33 Ni, 4.32 Mo, 15.55 Co, 14.47 Cr, 4.28 Al, 3.18 Ti, 0.104 C, 0.002 S, 0.02 Mn[b], 0.04 Si, 0.012 B, 0.02 Zr[b], 0.31 Fe, 0.02 Cu[b], 0.004 P

[a]Data furnished by Dr. O. G. Engel, General Electric Co., Cincinnati, Ohio, (analysis by suppliers).
[b]Maximum.
[c]Ref. 3 (nominal composition).
[d]Specimens machined at this stage.

TABLE II. - PHYSICAL PROPERTIES OF TEST MATERIALS

Material	Ultimate tensile strength		Yield strength (0.2 percent offset)		Elongation, percent	Reduction in area, percent	Density, g/cm^3	Hardness	Grain size ASTM standard
	psi	N/m^2	psi	N/m^2					
Zinc[a]	15 500	1.07×10^8	6 600	0.46×10^8	9	7.10	7.133	Brinell 38	2
Annealed Ni-270[a]	51 400	3.54	9 100	.62	70	86.40	8.902	RF 62 (Brinell 56)	6
As-received Ni-270[b]	48 800	3.36	8 000	.55	61	91.50	8.940	RB 25 (Brinell 64)	0 to 2
Iron[a]	43 300	2.99	21 400	1.48	52	73.18	7.874	RF 75.4 (Brinell 70)	4 to 5
Tantalum[a]	34 800	2.40	22 700	1.57	69	84.80	16.600	94 DPH (Brinell 89)	1
Udimet 700[a]	201 000	13.86	130 000	8.96	19	18.51	7.920	RA 68.3 (Brinell 332)	1

[a]Data furnished by Dr. O. G. Engel, General Electric Co., Cincinnati, Ohio.
[b]Ref. 3.

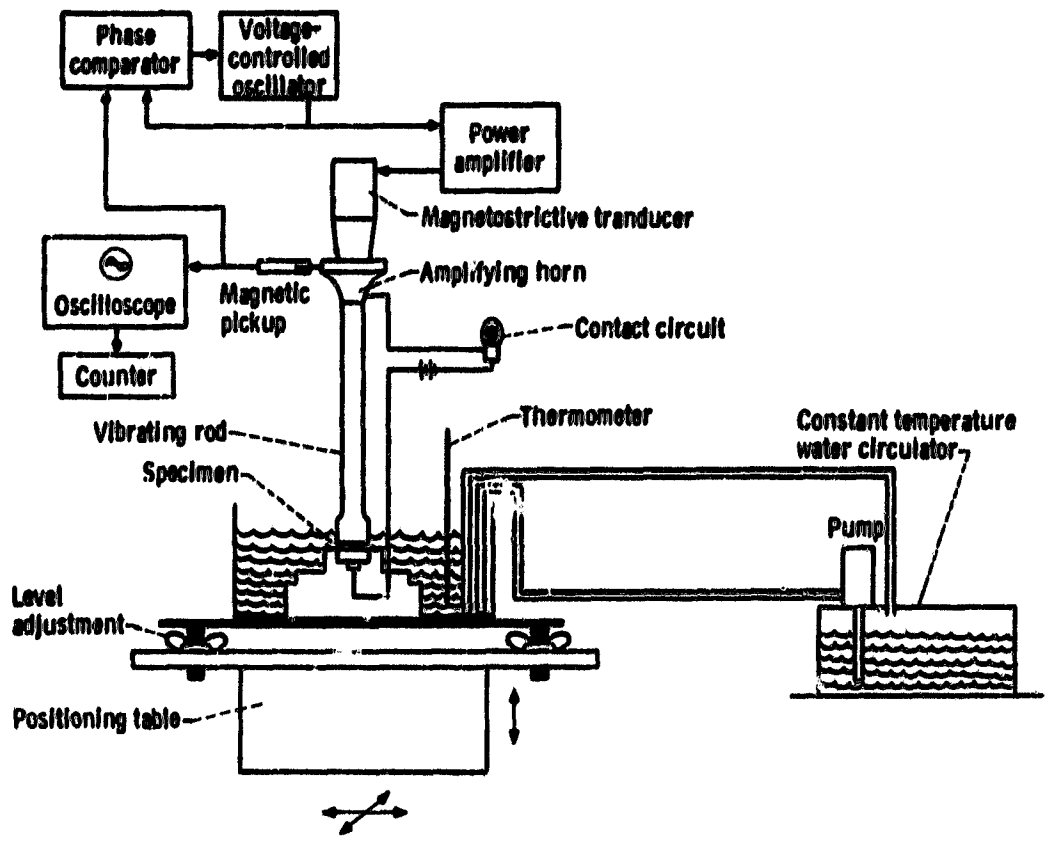


Figure 1. - Schematic diagram of cavitation test apparatus with stationary specimen.

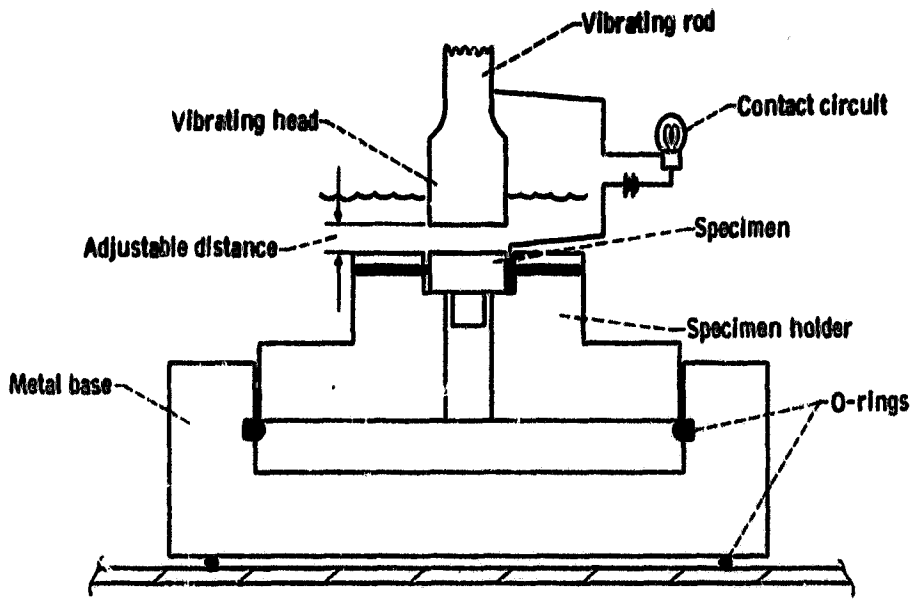
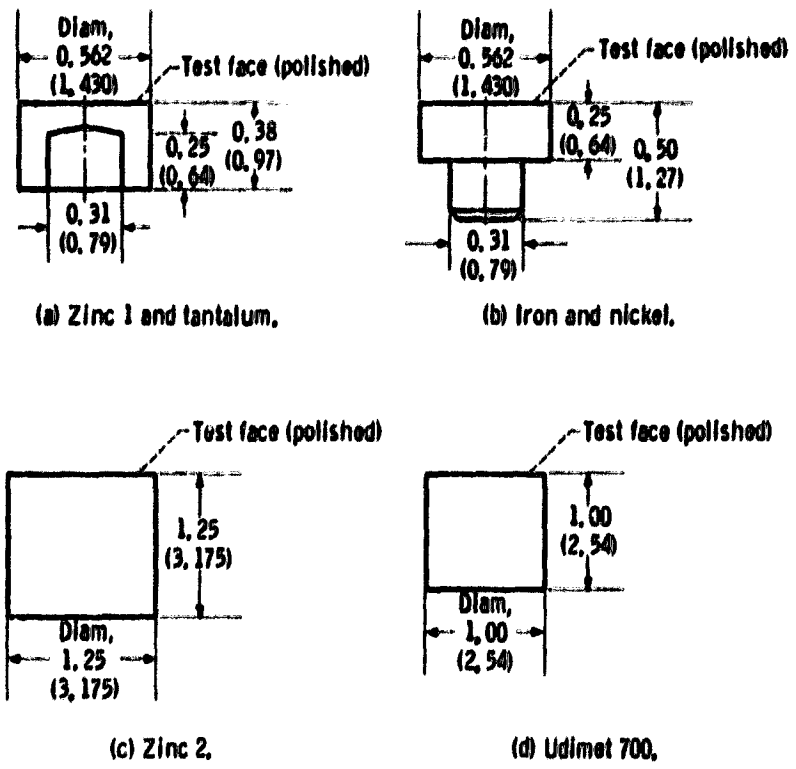


Figure 2. - Cavitation specimen holder assembly.



(a) Zinc 1 and tantalum.

(b) Iron and nickel.

(c) Zinc 2.

(d) Udimet 700.

Figure 3. - Vertical cross sections of four different types of cavitation test specimens used. (All dimensions are in inches (cm).)

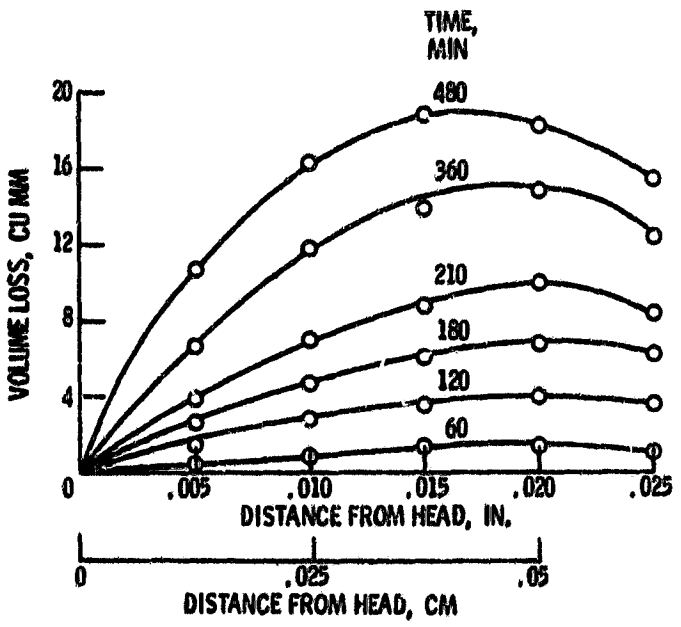


Figure 4. - Effect of separation distance on cavitation damage (volume loss) to as-received NI-270 in water at 75° F (24° C) at various test times.

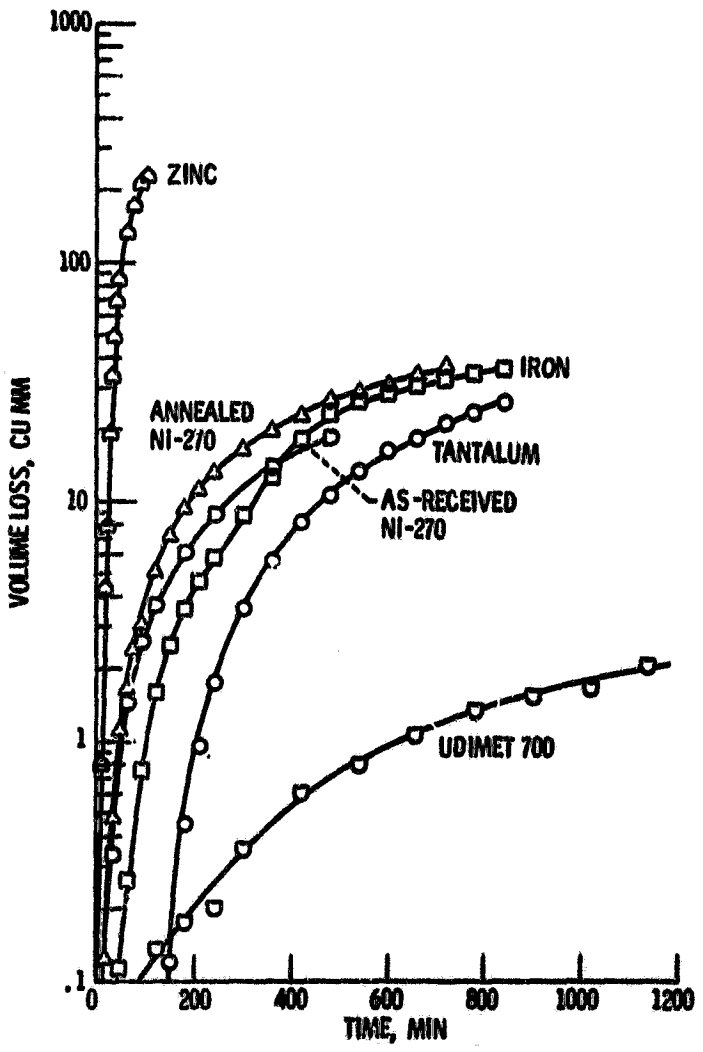
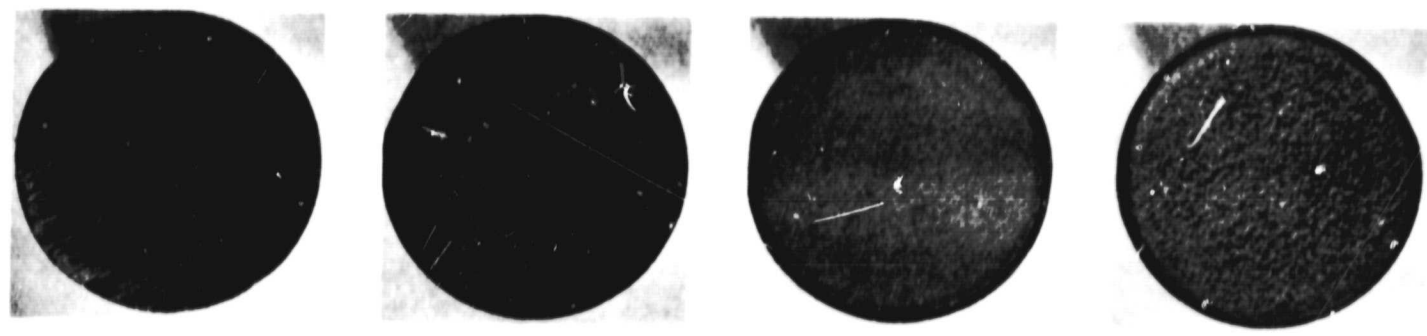
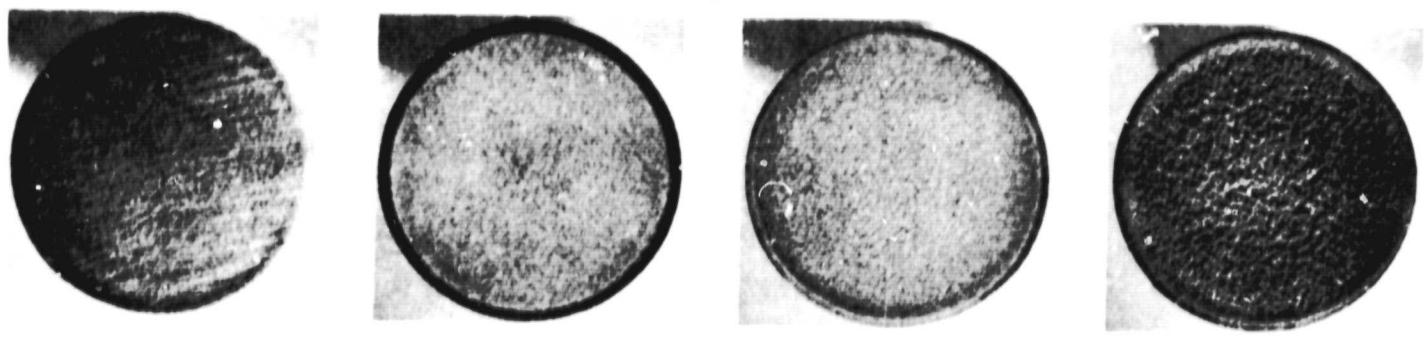


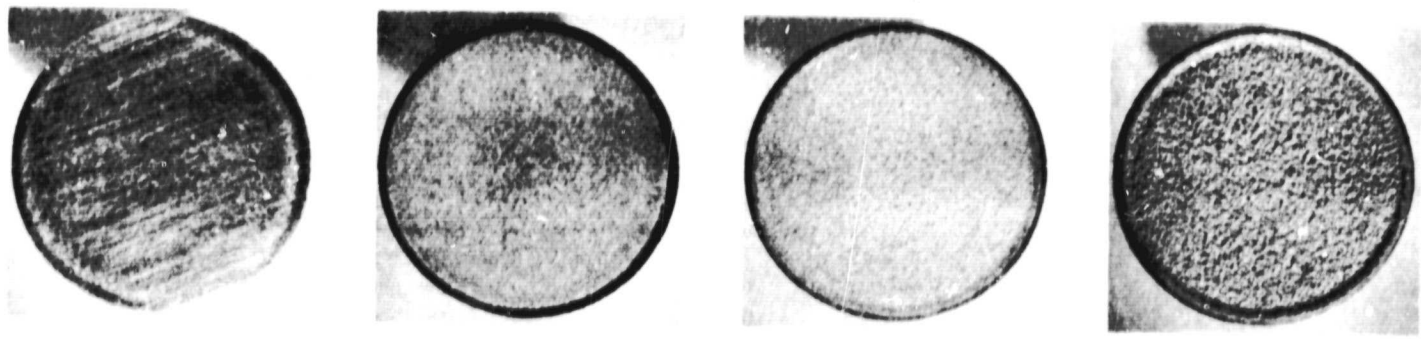
Figure 5. - Cavitation damage (volume loss) of metals in water at 75° F (24° C).



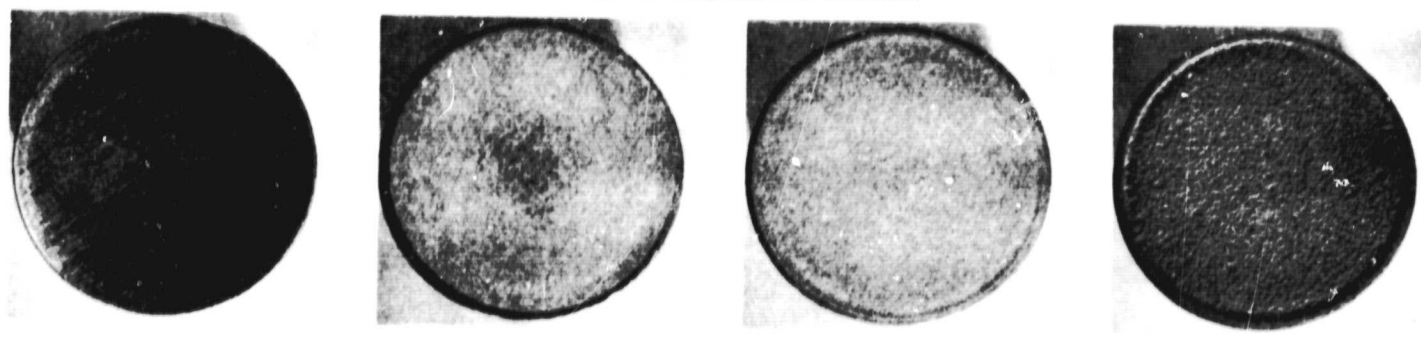
(a) Distance from head, 0.025 inch (0.064 cm).



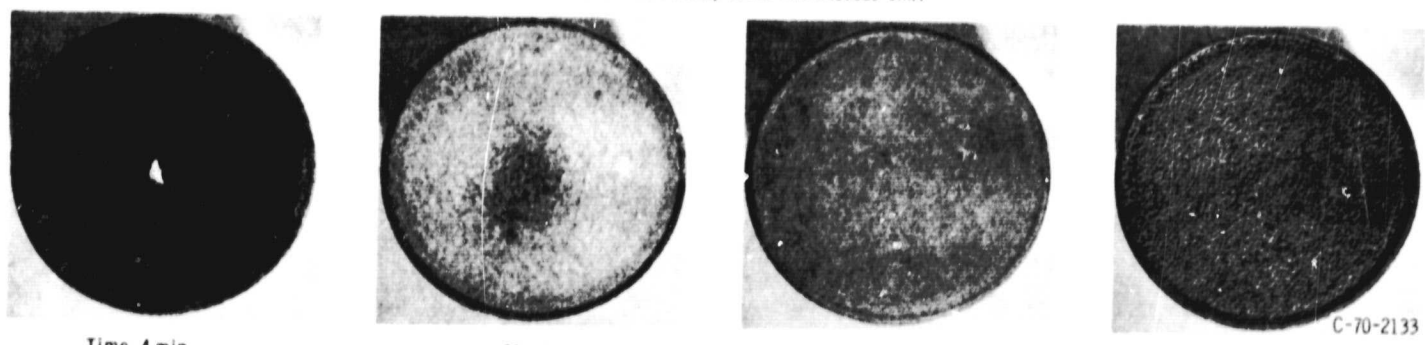
(b) Distance from head, 0.020 inch (0.051 cm).



(c) Distance from head, 0.015 inch (0.038 cm).



(d) Distance from head, 0.010 inch (0.025 cm).



Time: 4 min

30 min

120 min

480 min

(e) Distance from head, 0.005 inch (0.013 cm).

Figure 6. - Damaged surfaces of as-received Ni-270 specimens after exposure in water at 75° F (24° C) for various times and distances from head.

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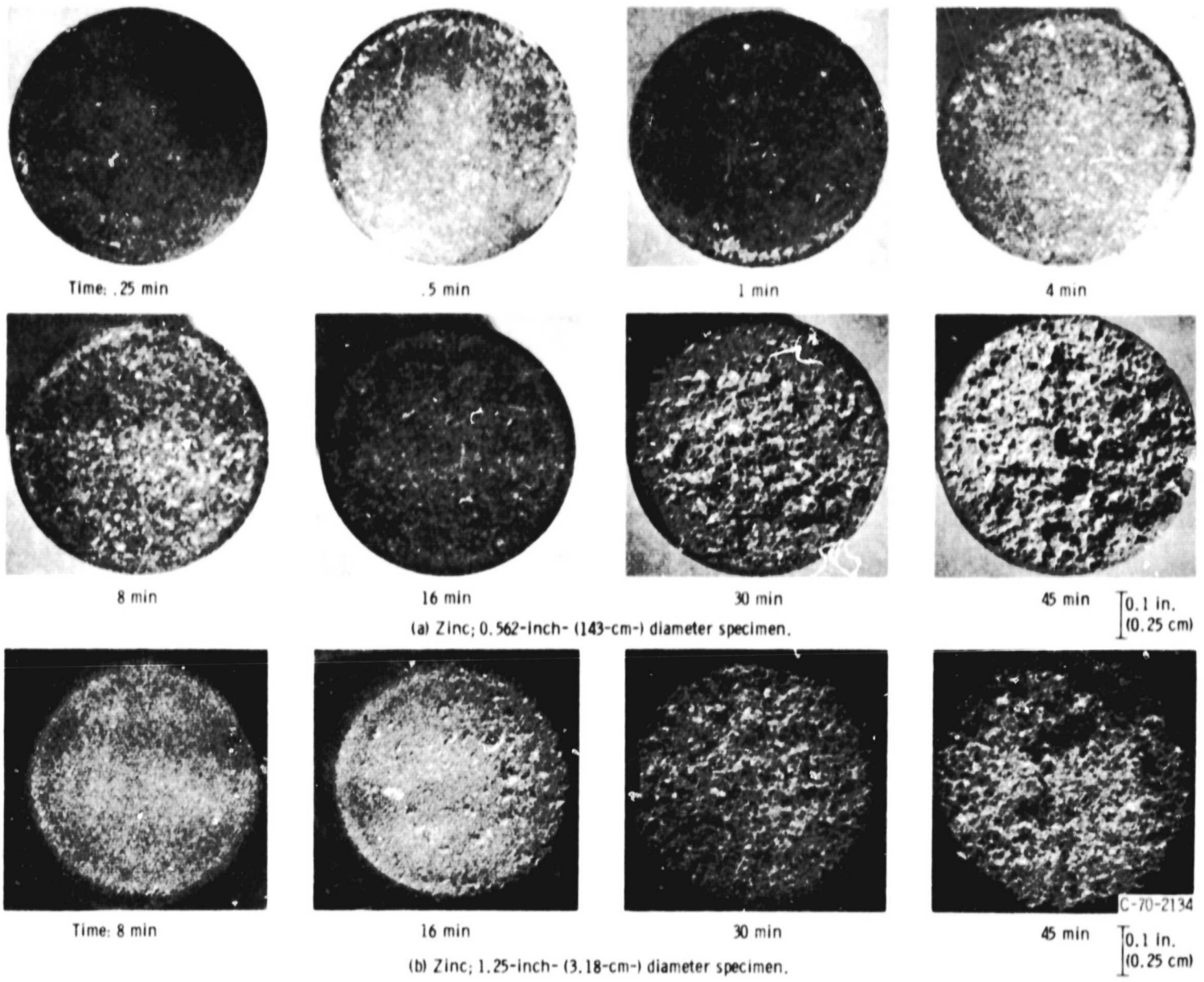

(a) Zinc; 0.562-inch- (143-cm-) diameter specimen.

(b) Zinc; 1.25-inch- (3.18-cm-) diameter specimen.

Figure 7. - Damaged surfaces of specimens after exposure to cavitation in 75° F (24° C) water for various times at separation distance of 0.015 inch (0.038 cm).

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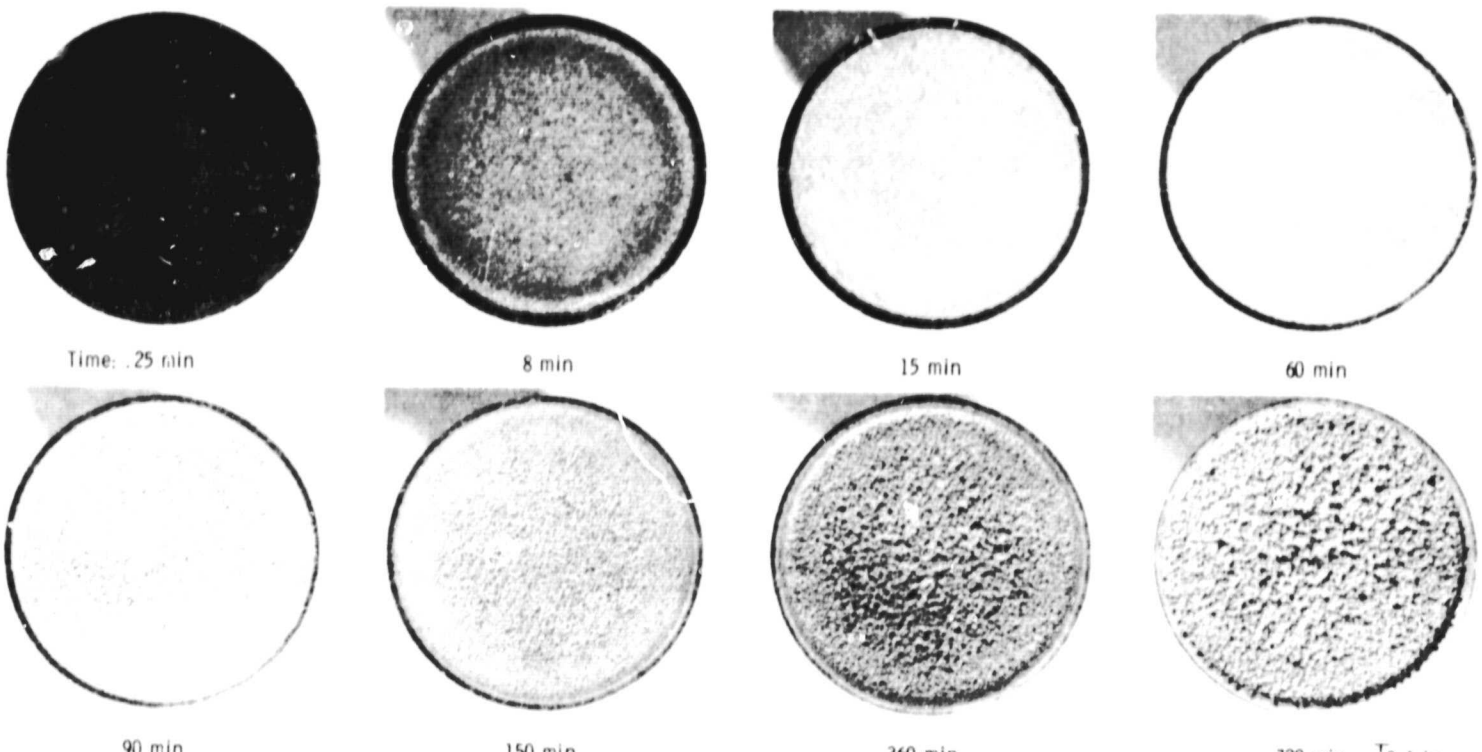
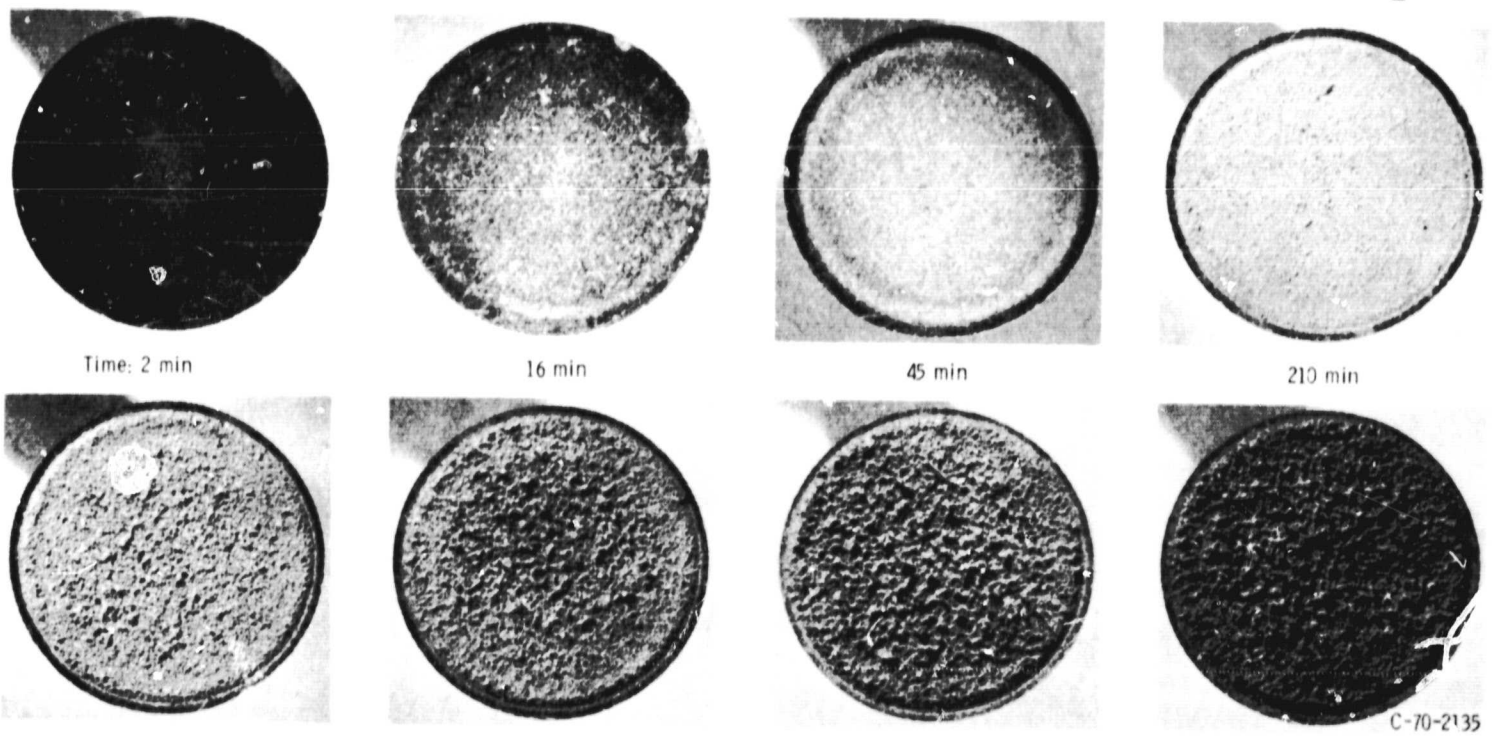
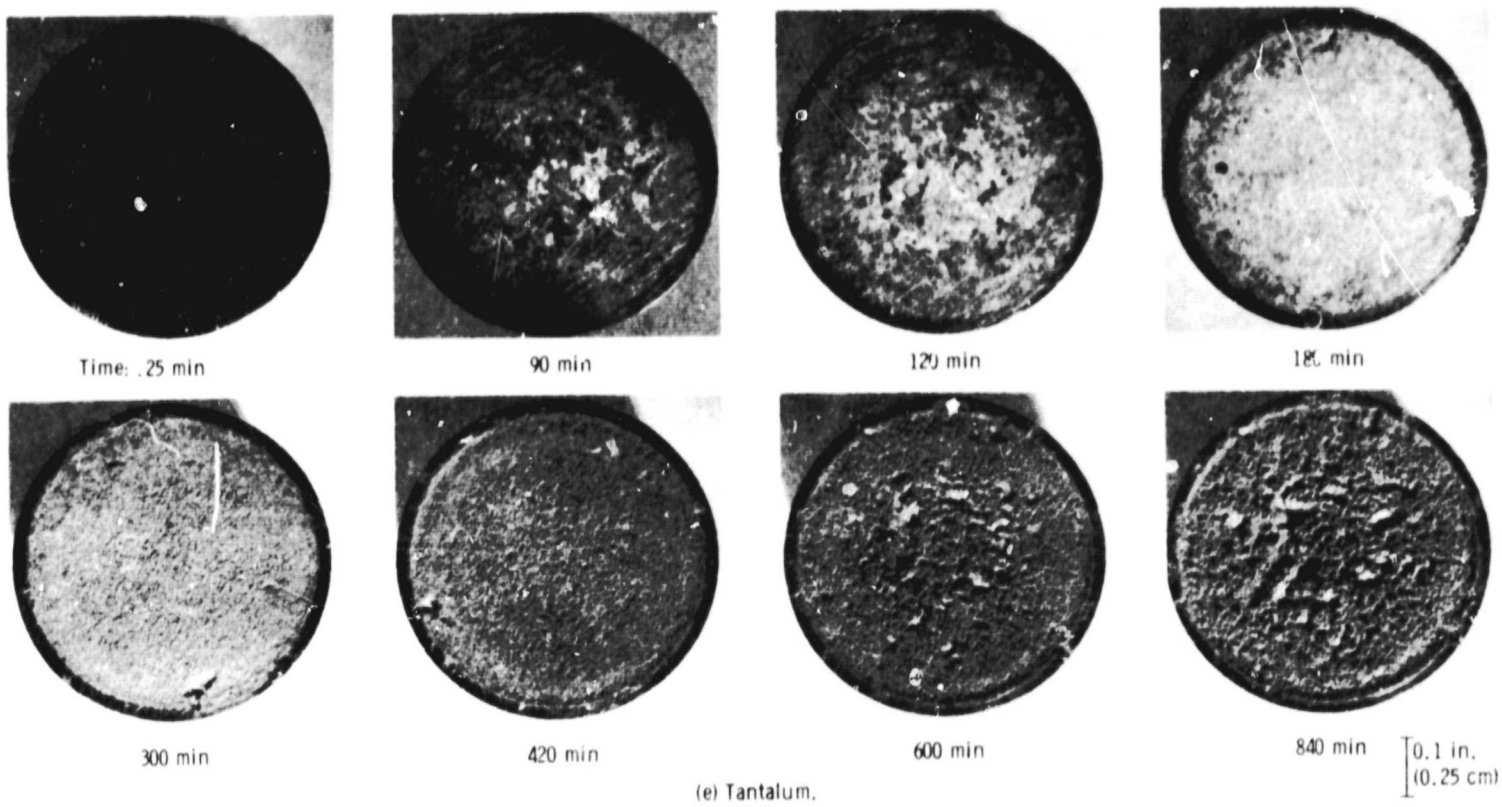


(c) Nickel (annealed).

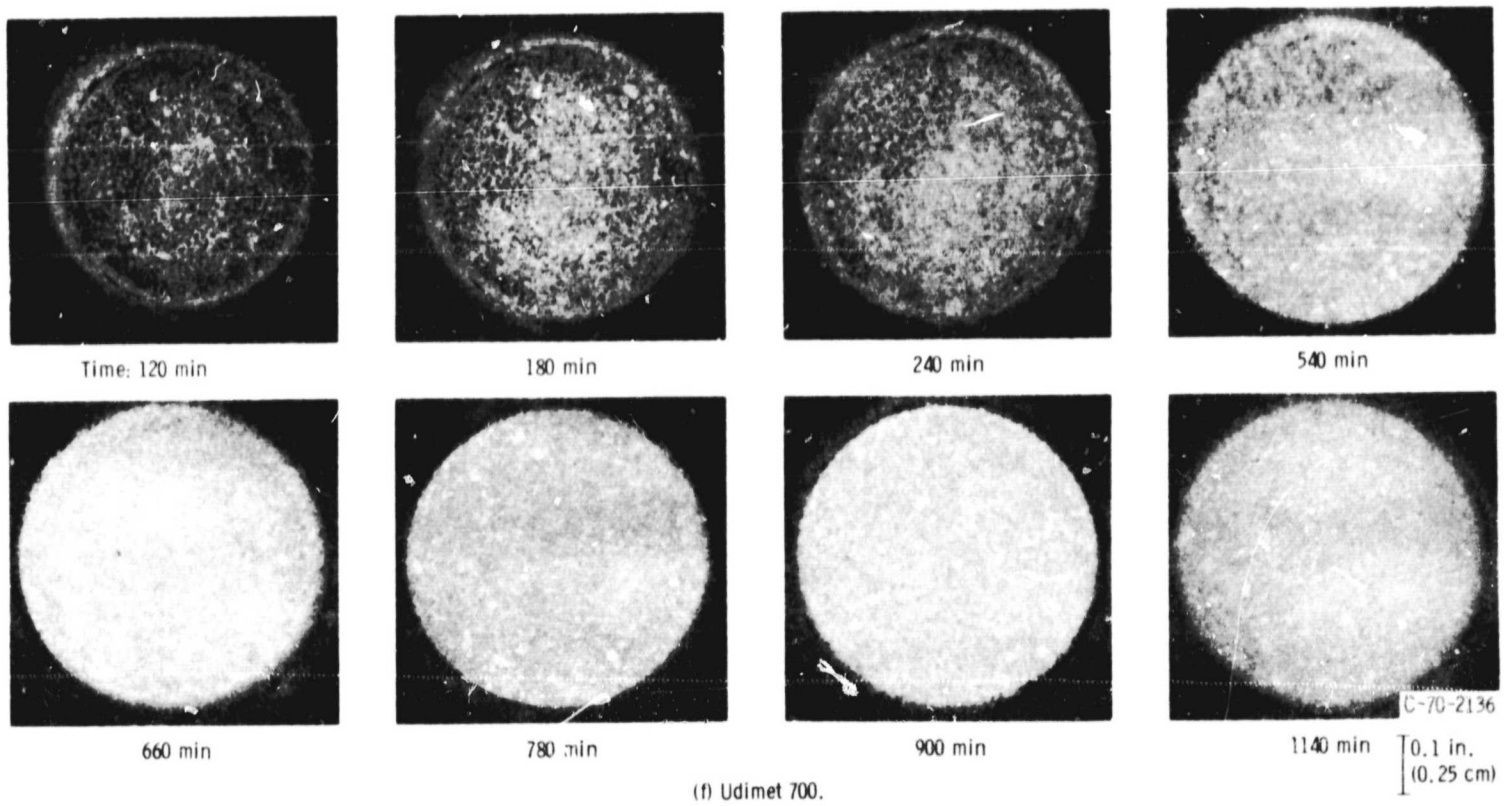


(d) Iron.

Figure 7. - Continued.



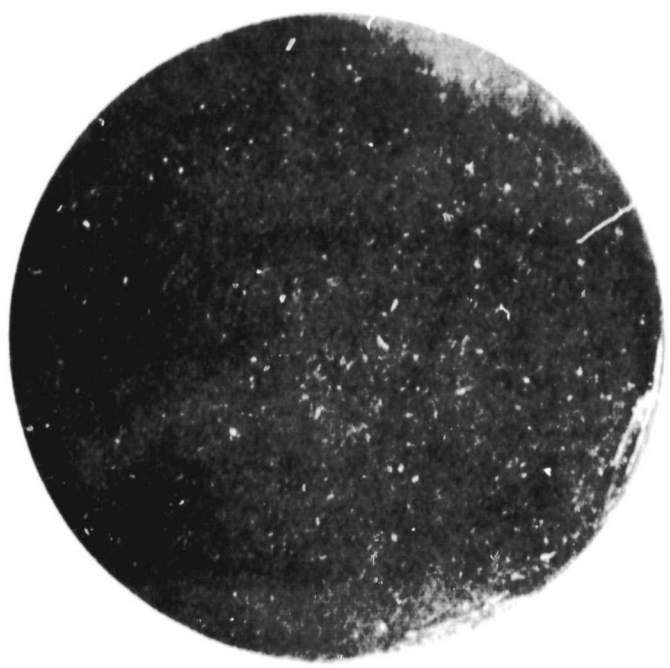
(e) Tantalum.



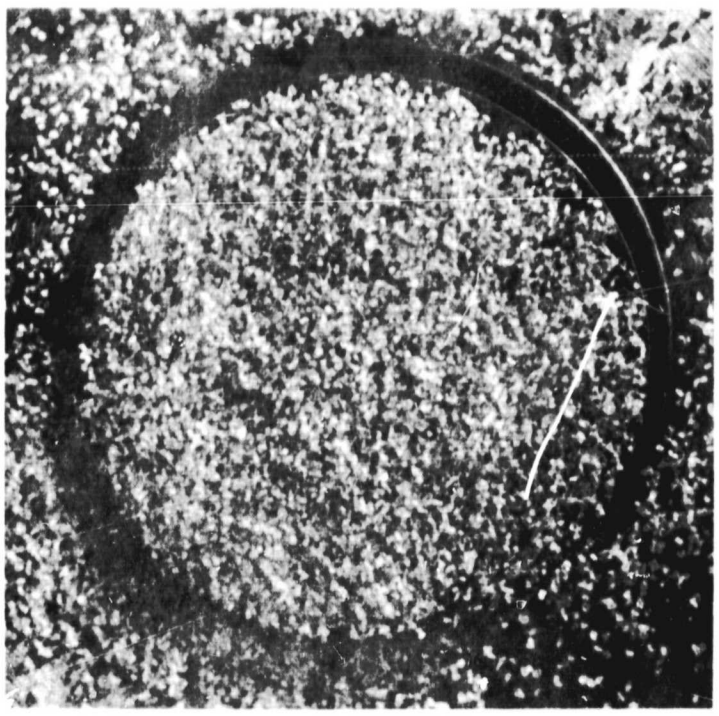
(f) Udimet 700.

Figure 7. - Concluded.

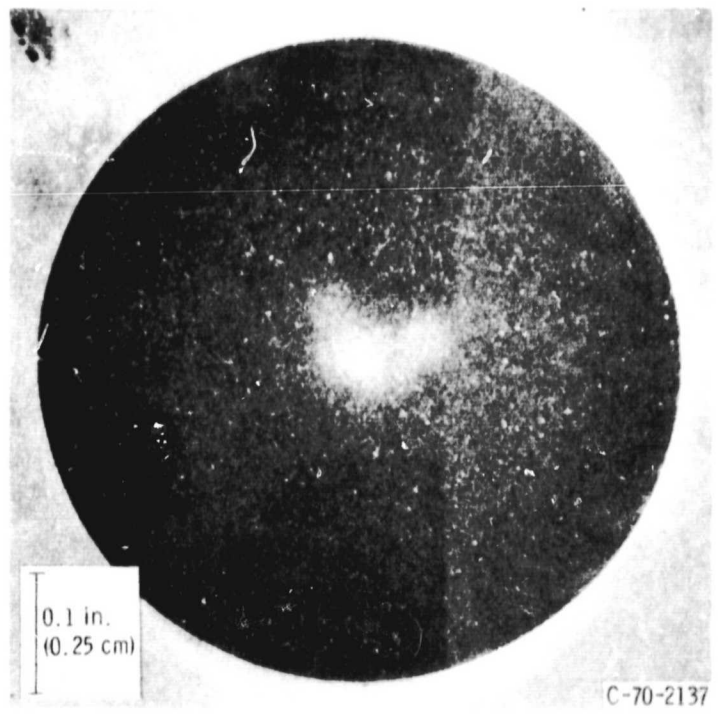
(a) Untested polished specimen without particles.



(b) 8 Minutes normal exposure.



(c) Untested polished specimen covered with particles.



(d) 8 Minutes exposure showing damage caused by particles.

Figure 8. - Effect of 100 mesh abrasive particles on cavitation damage to AISI type 316 stainless steel specimen in water at 75° F (24° C).

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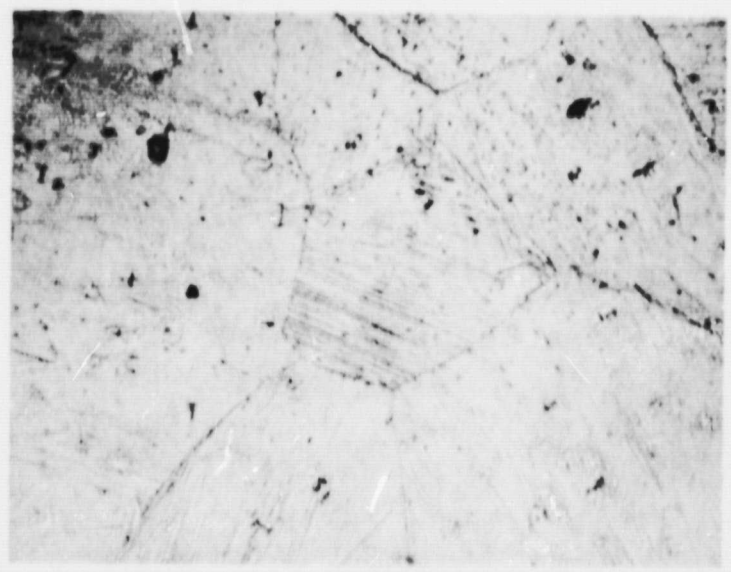
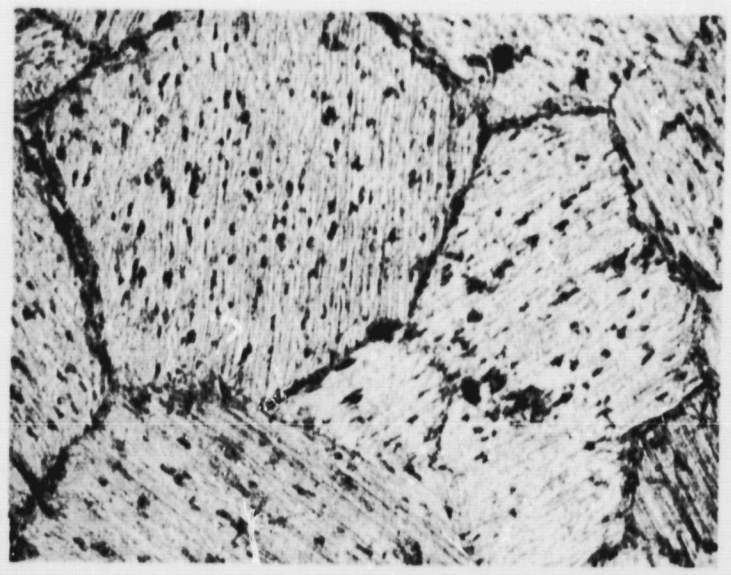
Time: 0 min (polished - etched)

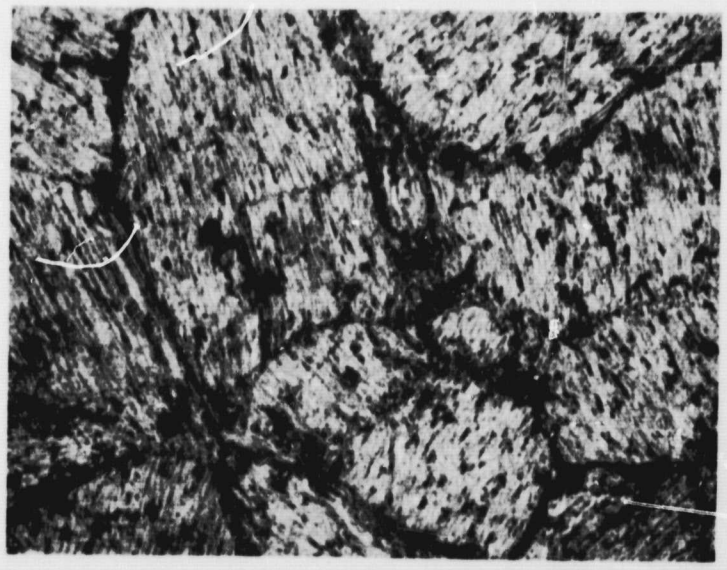


0.5 min

1 min



4 min



8 min

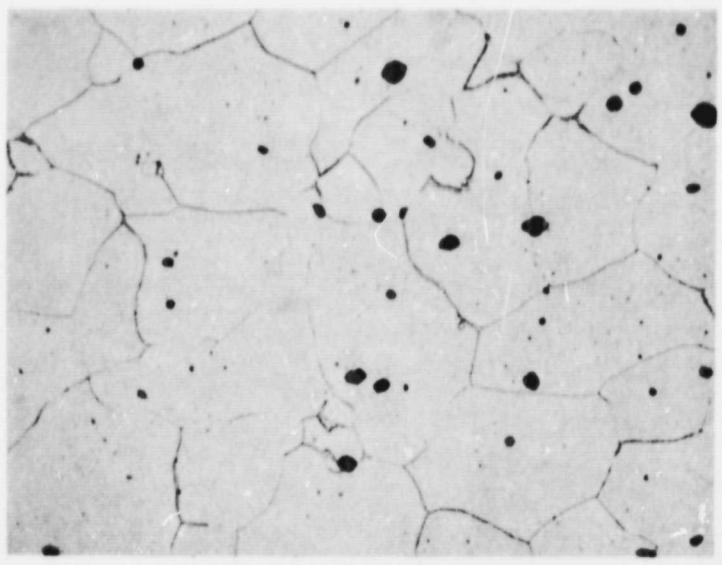
C-70-2138

16 min

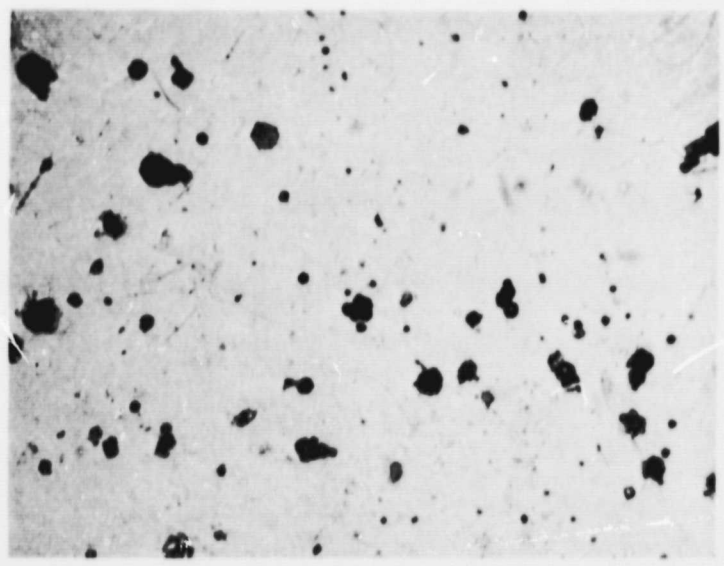
0.001 in.
(0.0025 cm)

(a) Zinc.

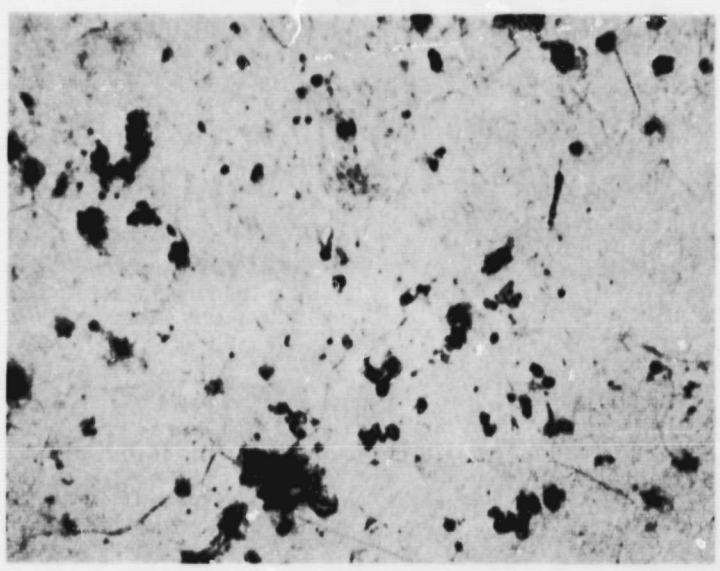
Figure 9. - Photomicrographs of damaged surfaces of specimens exposed to cavitation for various times at 75° F (24° C). (Tested specimens were repolished after 0-min photographs were made.)



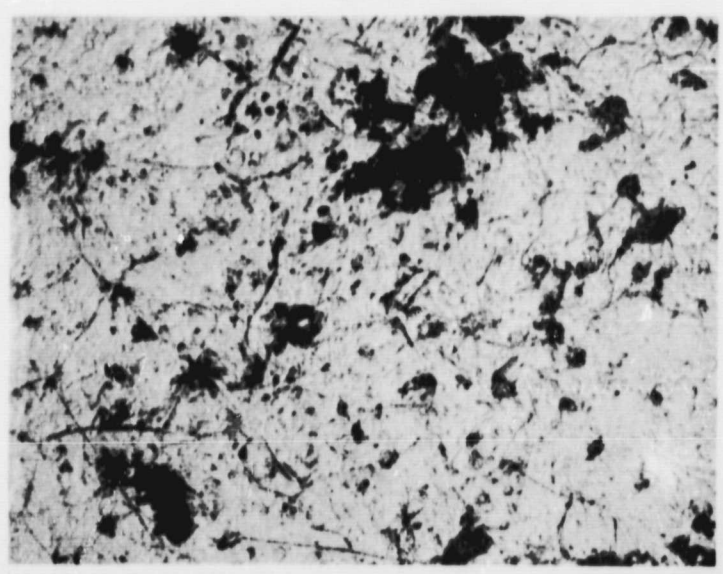
Time: 0 min (polished - etched)



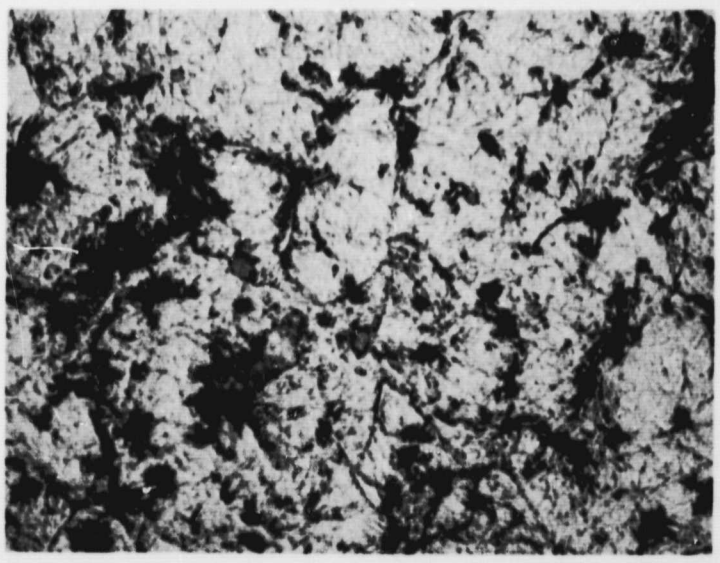
2 min



8 min



16 min



30 min

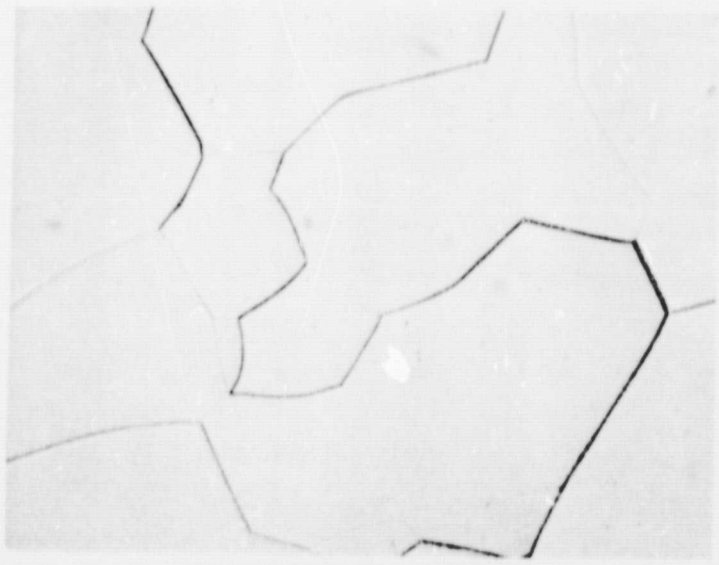
60 min

0.001 in.
(0.0025 cm)

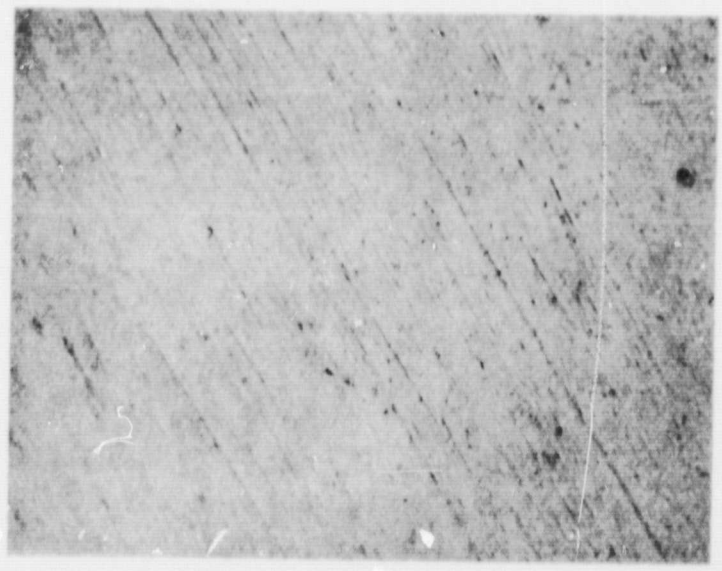
(c) Iron.

Figure 9. - Continued.

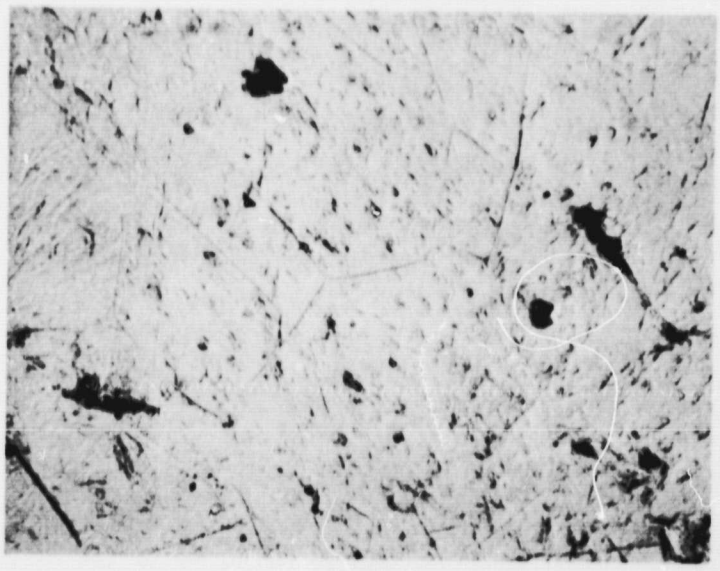
E-5688



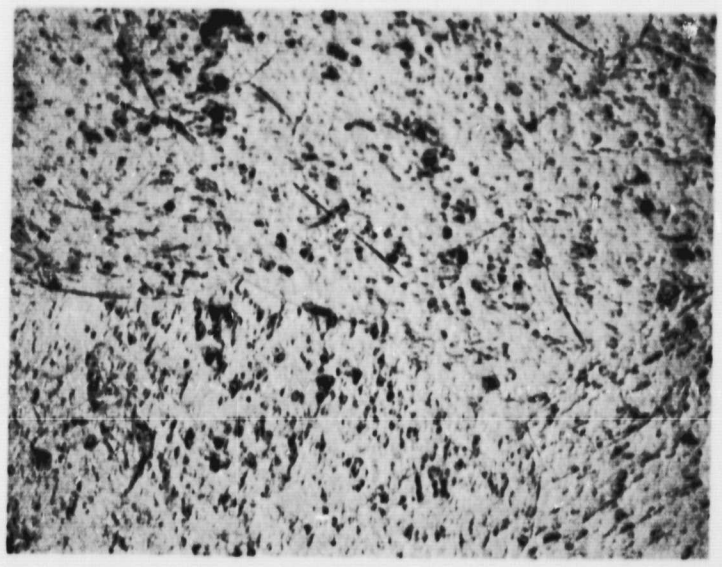
Time: 0 min (polished - etched)



0.25 min



30 min



45 min

60 min

90 min

0.001 in.
(0.0025 cm)

(d) Tantalum.

Figure 9. - Concluded.

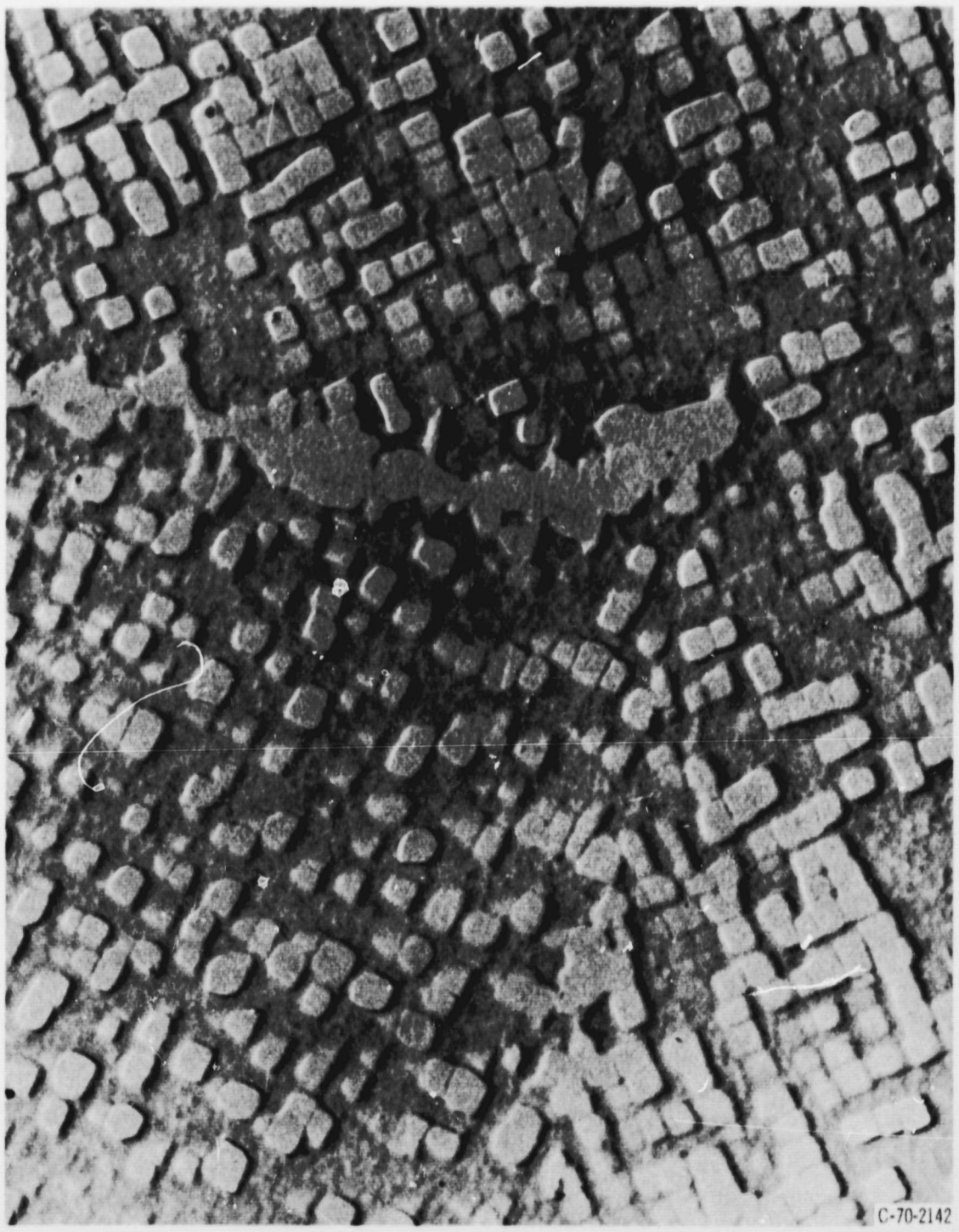


Figure 10. – Electron microscope replica of surface of Udimet 700 subjected to cavitation in water at 75° F (24° C) for 120 minutes. X17 500. (Reduced 30 percent in printing.)