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ARR Aug. 1942

(ARR-AUG.1942)

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

WARTIME REPORT

ORIGINALLY ISSUED

August 1942 as
Advance Restricted Report

PROCEDURE USED AT ALUMINUM RESEARCH LABORATORIES FOR DETERMINING TYPE OF ATTACK IN SOME ALUMINUM ALLOYS

By C. J. Walton and F. Keller
Aluminum Company of America

NACA

WASHINGTON

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ADVANCE RESTRICTED REPORT

PROCEDURE USED AT ALUMINUM RESEARCH LABORATORIES FOR DETERMINING TYPE OF ATTACK IN SOME ALUMINUM ALLOYS

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SUMMARY

Representative samples of 17S-T and 24S-T aluminum alloys were subjected to accelerated corrosion tests in which the corrosion period was established at 6 hours for samples that had been etched to produce a uniform surface. The results show the types of corrosion attack, which are described in standard terminology, and may form a basis for estimating the resistance of the material to corrosion by a comparison of the relative depth and frequency of the corrosive attacks.

INTRODUCTION

The resistance to corrosion and type of corrosion attack for some aluminum alloys, especially 17S-T and 24S-T materials, are checked ordinarily by conducting accelerated corrosion tests on representative samples. The methods of testing and the terminology employed in reporting the results of such tests have not heretofore been entirely consistent because (1) the same corrosion period was not always employed, (2) surface conditions of the samples varied, and (3) the nomenclature for describing corrosive attack was not standardized. The procedure that is recommended in the present paper takes into account these factors. The corrosion period has been established at 6 hours for samples which have received a treatment to produce a uniform surface. Results obtained with this procedure and reported in accordance with the standard terminology should be comparable.

Acknowledgment is due G. W. Wilcox for his skillful photomicrographic work in connection with the development of this procedure.

TESTING PROCEDURE

Samples selected for testing should be etched to produce a uniform surface condition, immersed in the standard sodium chloride-hydrogen peroxide solution for 6 hours and subsequently examined microscopically. Details of the various steps in the procedure are as follows:

Method of sampling. - For sheet material, samples about 1 by 5 inches are suitable; for rolled and extruded sections, pieces several inches long should be cut. The ends of the rolled and extruded specimens should be coated with a mixture of beeswax and rosin (approximately 50-50) in order to avoid possible protection of the surface by the more anodic central portion of the specimen that is exposed at the ends.

Surface treatment. - Prior to the corrosion test, each specimen should be immersed for 1 minute in an etching solution at approximately 95° C. The solution is made by adding to distilled water 50 milliliters of concentrated nitric acid (70 percent) and 5 milliliters of hydrofluoric acid (48 percent) per liter of solution. After this treatment, the specimen should be rinsed in distilled water, immersed for 1 minute in concentrated nitric acid (70 percent) at room temperature to remove any metallic copper that may have plated out on the specimen, rinsed in distilled water, and allowed to dry.

The hydrofluoric acid in the etching solution is gradually used up by the specimen and by most containers; Heresite-coated aluminum beakers should therefore be used to prevent reaction between the hydrofluoric acid and the container. If pyrex beakers are used, the action of the hydrofluoric acid on the beaker can be minimized by carefully adding the acid after the water is up to the desired temperature. When the solution does not contain enough hydrofluoric acid, it will produce a matte surface. It is advisable, therefore, always to use a fresh solution. A spent solution, however, can be rejuvenated by the addition of about 1 milliliter of hydrofluoric acid per liter.

If the samples contain an anodic or a chemical coating, such a coating must be removed before the surface treatment is used. The removal of this coating can be accomplished by abrading the surface with No. 120 Aloxite cloth or by immersing the sample in aqueous solution containing 2 percent chromic acid and 5 percent phosphoric acid. This solution is used at a temperature of 85° C. The period of immersion required depends on the thickness of the coating but should not be unnecessarily prolonged.

Corrosive mediums. - The solution that is used for the corrosion test is made up by adding to distilled water 57 grams of U.S.P. grade sodium chloride and 10 milliliters of 30 percent hydrogen peroxide (the grades of hydrogen peroxide that have been found satisfactory are Merck's Superoxyl and Baker's c.p. grade) per liter of solution. If only type of attack is desired, 100 milliliters of 3 percent hydrogen peroxide (the 3 percent hydrogen peroxide usually contains stabilizers, some of which are known to accelerate corrosion to various degrees) per liter can be substituted for the 30 percent hydrogen peroxide.

Method of exposure. - The specimens should be totally immersed in a freshly prepared corrosive solution. If quantitative results for depth of attack are desired, a standard quantity of solution should be used per unit area of surface. Thirty milliliters of solution per square inch is the quantity recommended. Ordinarily, when only type of attack is required, the minimum amount of solution should be 250 milliliters.

If more than one specimen is exposed in the same container, the specimens should be electrically insulated from each other, preferably suspended from glass rods with porcelain separators or suspended from glass hooks.

The type of attack is substantially independent of the temperature of the solution but the extent of corrosion is not. More consistent results with regard to depth of attack will be obtained if the corrosion tests are conducted at a constant temperature; for example, at 30° C. It is permissible, however, to conduct tests at room temperature provided that a record is kept of temperatures at which the tests are made.

Exposure time. - A period of 24 hours had previously been employed but tests have shown that, because of the consumption of the hydrogen peroxide, the corrosion rate decreases rather rapidly after the first 6 to 8 hours of exposure. A period of 1 hour conserves time and is sufficient if only the type of attack is to be determined but, if both the type and the extent of corrosion are to be obtained, a 6-hour period is recommended.

In the case of thin samples (0.020 in. or less) from slowly quenched material, a 6-hour exposure might be too drastic. In such cases, a 1-hour exposure is advisable.

Microscopic examination. - Representative cross sections cut from the corroded samples should be prepared for microscopic examination in accordance with recommended practices (references 1, 2, and 3). These examinations should be made at magnifications of 100 to 500 diameters. In the case of sheet, rod, and tubing, at least three sections should be examined. For rolled or extruded sections of complicated shapes, at least one section should be examined from each thickness of material represented in the shape.

In general, after an initial examination, it is advisable to etch the specimens to develop the microstructure in order that the relation of corrosive attack to microstructural characteristics is established. The etchant that is recommended for duralumin-type alloys contains 1.0 milliliter concentrated hydrofluoric acid, 1.5 milliliters concentrated hydrochloric acid, 2.5 milliliters concentrated nitric acid, and 95.0 milliliters of distilled water. The etching is accomplished by immersing the specimen, polished surface up, for a period of 10 to 20 seconds in a freshly prepared solution at room temperature. The specimen is then washed free of the acid in a stream of warm (not hot) running water and blown dry.

TERMINOLOGY

The terminology for expressing the various types of corrosive attack is given in table I. This table also refers to the photomicrographs that were taken to illustrate the different types of attack that might be encountered in studying corroded wrought aluminum alloys.

The terminology selected is an outgrowth of much work on material subjected to both accelerated tests and natural environments and should suffice to describe most of the types of attack ordinarily encountered in wrought aluminum alloys.

Two types of corrosive attack that are sometimes observed in forged or extruded materials are included in this terminology - namely, interdendritic and interfragmentary. Interdendritic attack is encountered in wrought material in which the original dendritic structure had not been thoroughly broken up by the working operations. Under these circumstances, the corrosive attack may follow the arms of the dendrites. Interfragmentary attack is encountered in wrought material in which the fragmentation of the grains produced by the working operations had not been eliminated by recrystallization during subsequent thermal treatments.

DISCUSSION OF RESULTS

These methods have been developed in order to obtain an approximate idea of the resistance to corrosion of different samples without resorting to the mechanical testing of corroded samples. If the methods described are closely followed, the comparative resistance to corrosion of different lots of material may be estimated by comparing the relative depth and the frequency of the corrosive attack. The fact that some samples may show areas of slight intergranular attack does not mean necessarily that the resistance to corrosion will be adversely affected.

The recommended procedure for determining the type of corrosion attack in duralumin-type alloys will give only a rough measure of the resistance of the material to corrosion. This procedure is not intended to supplant the accelerated alternate-immersion test (reference 4) and the standard salt-spray test (reference 5). A reliable evaluation of the resistance of duralumin-type alloys to corrosion requires the mechanical testing of corroded samples and the determination of changes in mechanical properties in comparison with uncorroded samples of the same material.

Aluminum Research Laboratories,
Aluminum Company of America,
New Kensington, Pa.

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TABLE I

TERMINOLOGY FOR EXPRESSING RESULTS OF A MICROSCOPIC EXAMINATION OF CORRODED SPECIMENS

Type of attack	Symbol	Figure showing typical attack	Remarks
Pitting	P	1,2	The attack progresses irrespective of grains and grain boundaries.
Undermining pitting	UP	3	The attack spreads out to a marked extent beneath the surface of the metal. On certain cross sections, therefore, the channel leading from the surface to the body of the pit might not be visible. This type of attack is frequently associated with simple pitting.
Pitting with slight intergranular	P + SI	4	Specimens with this type of attack show only very shallow intergranular attack, usually in the form of short stringers. There should be no well-developed intergranular network.
Separate areas of pitting and definite intergranular, with predominating type named first	P + I) I + P)		Although many specimens exhibiting these types of attack have been studied, separate areas of distinct pitting and intergranular attack are seldom sufficiently close to each other to be shown in one photomicrograph.
Definite intergranular	I	5,6, 7,8	Occasional areas of definite pitting attack are disregarded if the intergranular attack is by far the predominant type.
Exfoliation	Exf.	9	This type of attack refers to the condition in which there has developed definite scaling or lifting of surface layers of metal. Exfoliation can result from different types of attack - intergranular, undermining pitting, or attack along certain planes, especially in cold-worked material. The corrosion products have a greater volume than the metal from which they are formed, and therefore lift the overlying layer.

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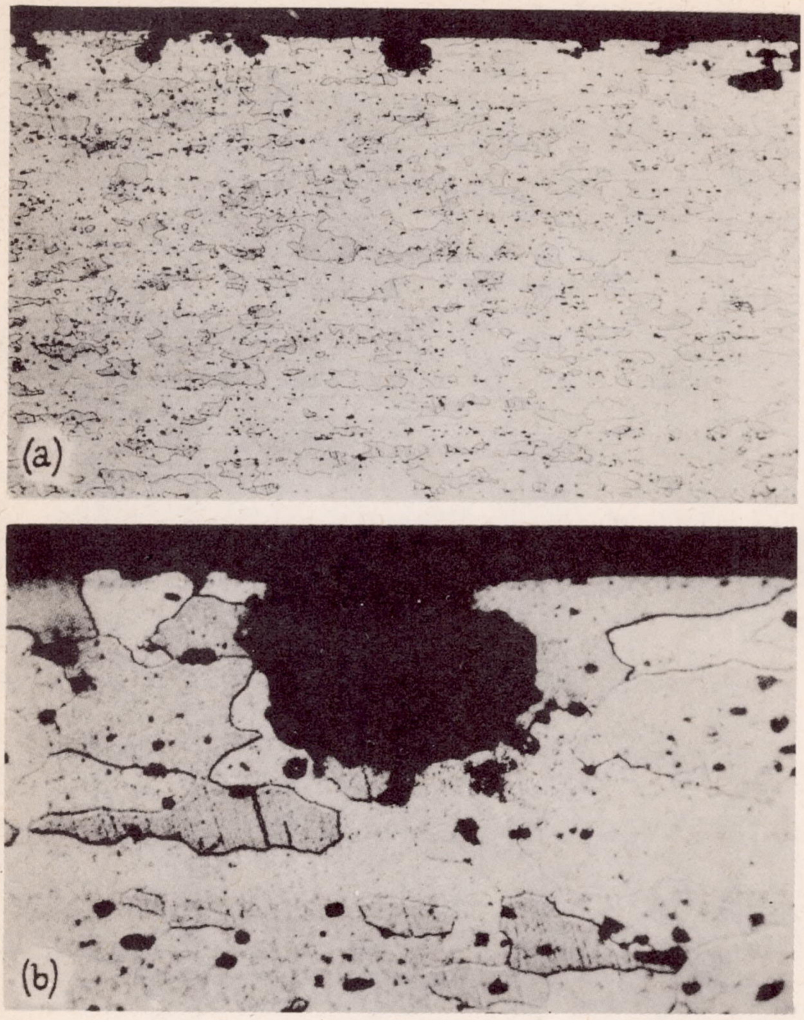
TABLE I (Continued)

Type of attack	Symbol	Figure showing typical attack	Remarks
Interdendritic	ID	10	This type results from attack that progresses around the dendrite arms. Certain portions of large forged or extruded sections still exhibit a definite dendritic structure, and are sometimes susceptible to interdendritic attack (fig. 10). In other cases, such zones might be susceptible to only an intergranular (I) type of attack (fig. 8).
Interfragmentary	IF	11	Interfragmentary attack refers to the type of attack obtained when corrosion extends along fragment boundaries in unrecrystallized areas usually found in certain regions of large forged or extruded sections.

Note.- Asterisks are to be employed with symbols to indicate any unusual degree of attack that requires a more detailed description in a footnote.



(a) Magnification, 100 diameters.
(b) Magnification, 500 diameters.

Figure 1a,b.- Pitting attack in 24S-T aluminum-alloy sheet, specimen 43870-6.

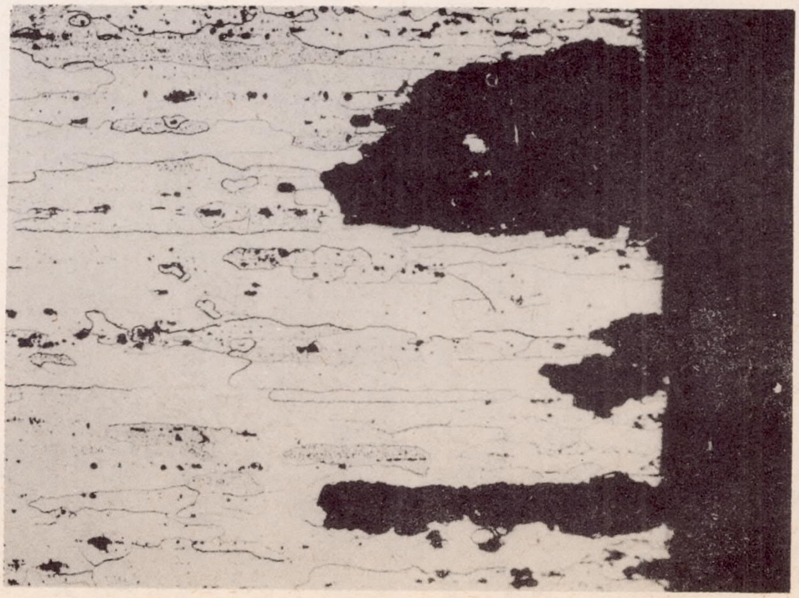


Figure 2.- Pitting type of edge attack in 24S-T aluminum-alloy tubing, specimen 45974. Magnification, 100 diameters.

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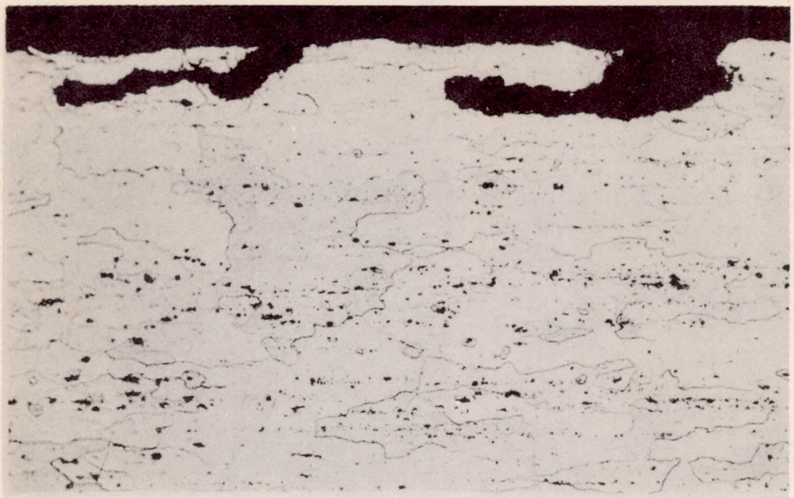
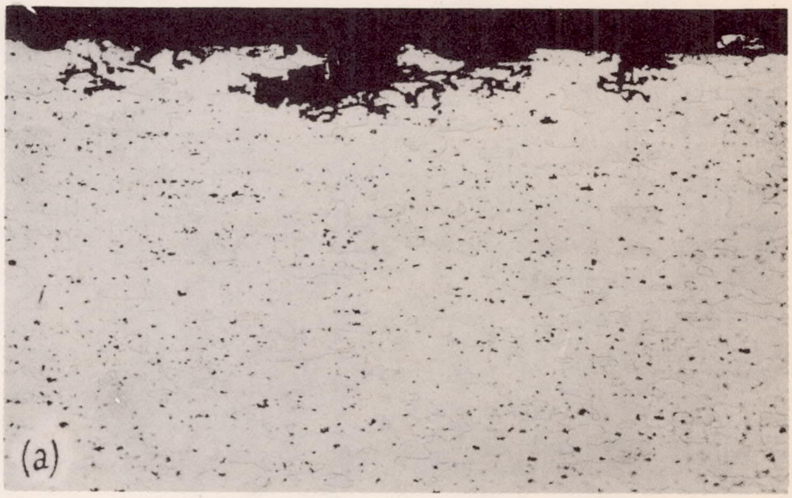
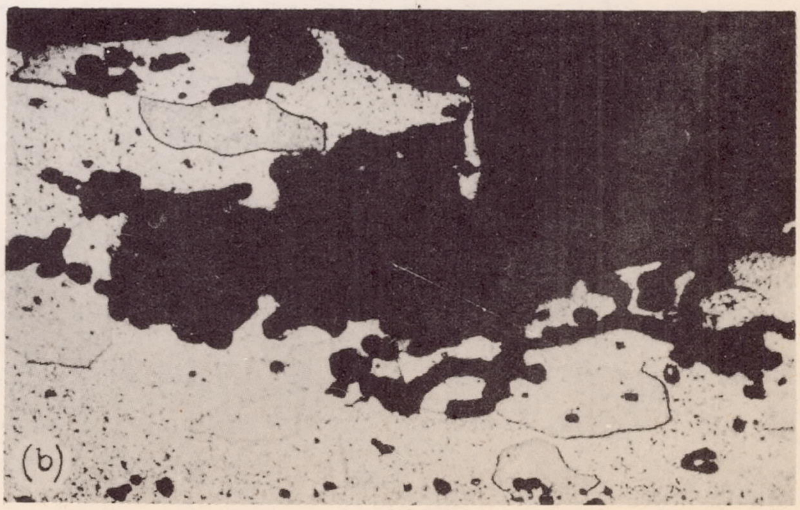


Figure 3.- Undermining pitting attack in 24S-T aluminum-alloy tubing, specimen 45974. Magnification, 100 diameters.





(a) Magnification, 100 diameters.
(b) Magnification, 500 diameters.

Figure 4a,b.- Pitting with slight intergranular attack in 24S-T aluminum-alloy sheet, specimen 43872-5.

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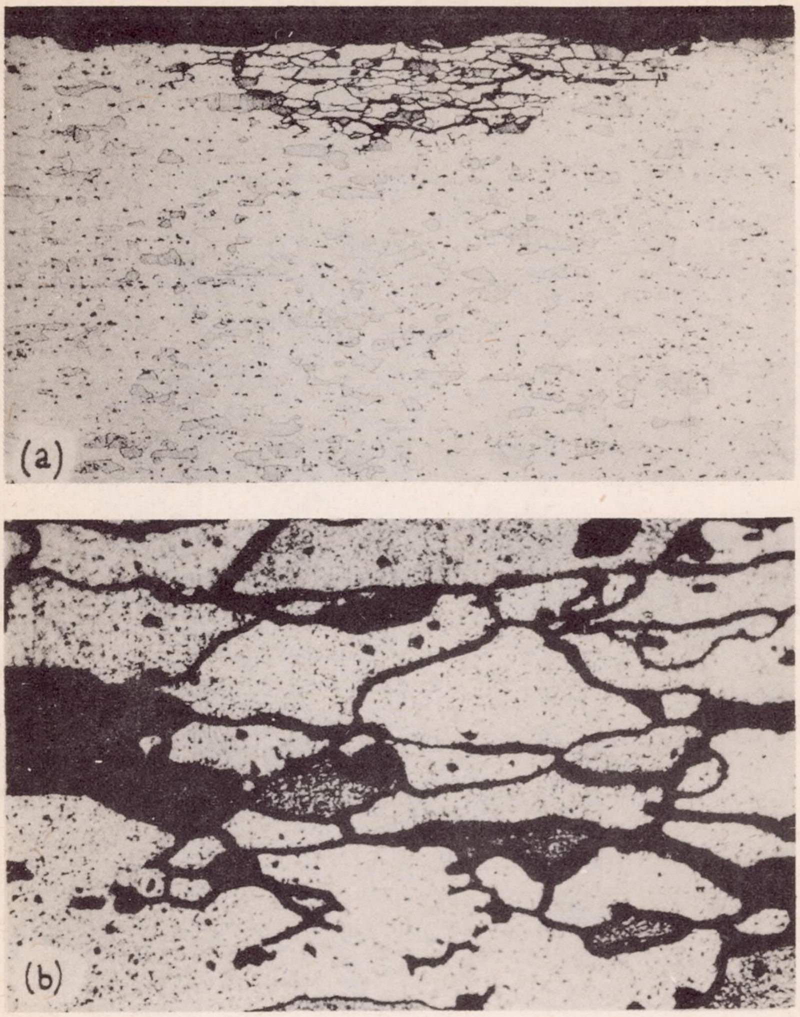

(a) Magnification, 100 diameters.
(b) Magnification, 500 diameters.

Figure 5a,b.- Intergranular attack in 24S-T aluminum-alloy sheet, specimen 43879-5.

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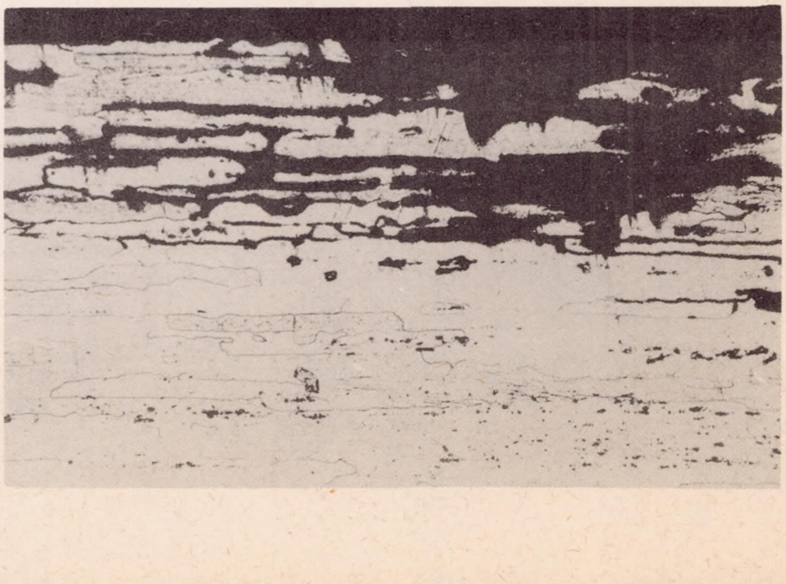
Figure 6.- Intergranular attack, which may in certain environments cause exfoliation, in 24S-T aluminum-alloy tubing, specimen 45973. Magnification, 100 diameters.



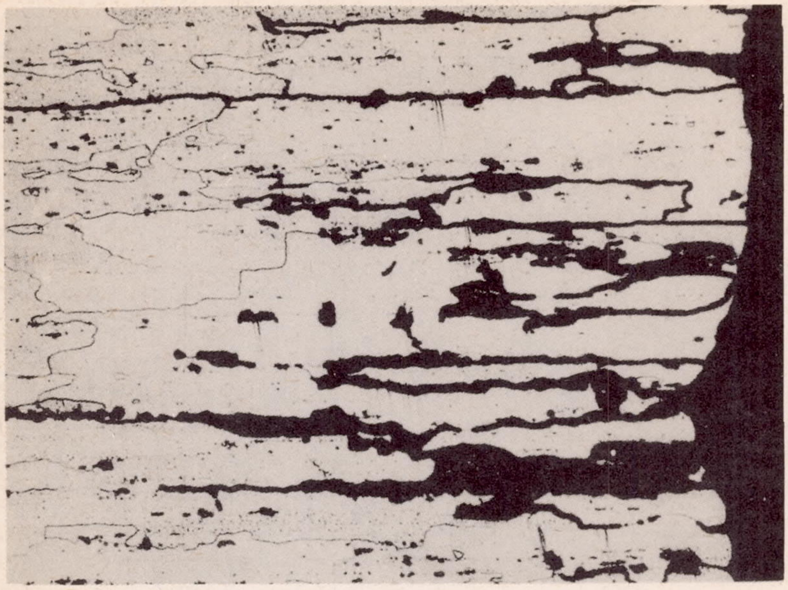
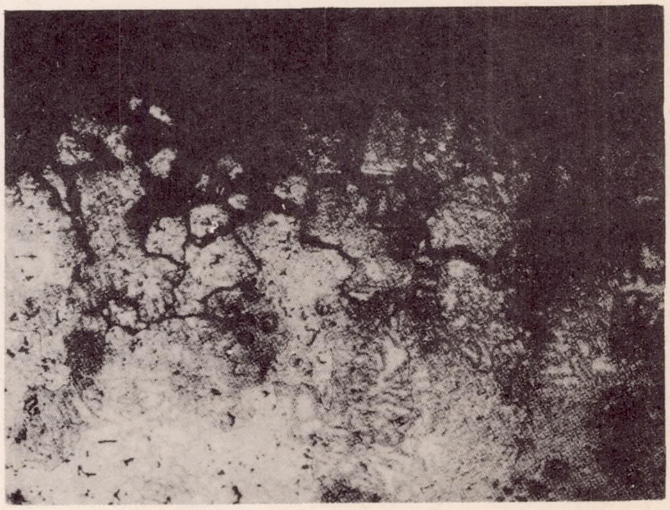


Figure 7.- Intergranular type of edge attack in 24S-T aluminum-alloy tubing, specimen 45973. Magnification, 100 diameters.

Figure 8.- Intergranular attack in a zone still exhibiting a dendritic structure in extruded 24S-T aluminum alloy, specimen 36990-4. Magnification, 100 diameters.



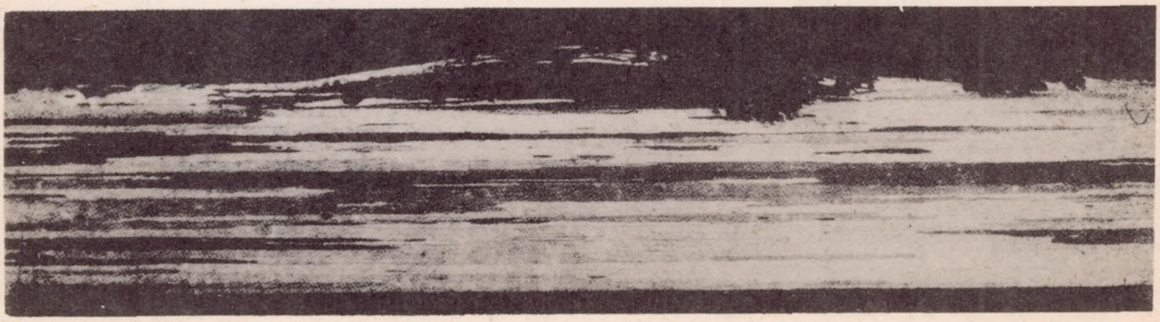


Figure 9.- Exfoliation attack in extruded 24S-T aluminum alloy, specimen 32027. Magnification, 10 diameters.

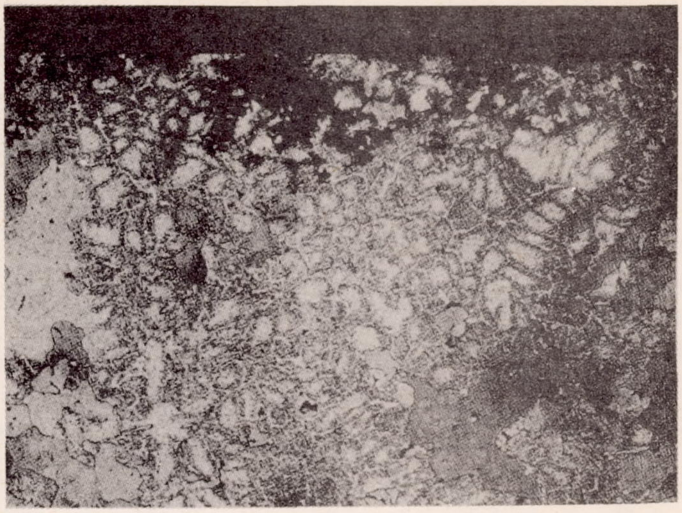


Figure 10.- Interdendritic attack in extruded 24S-T aluminum alloy, specimen 36989-1. Magnification, 100 diameters.

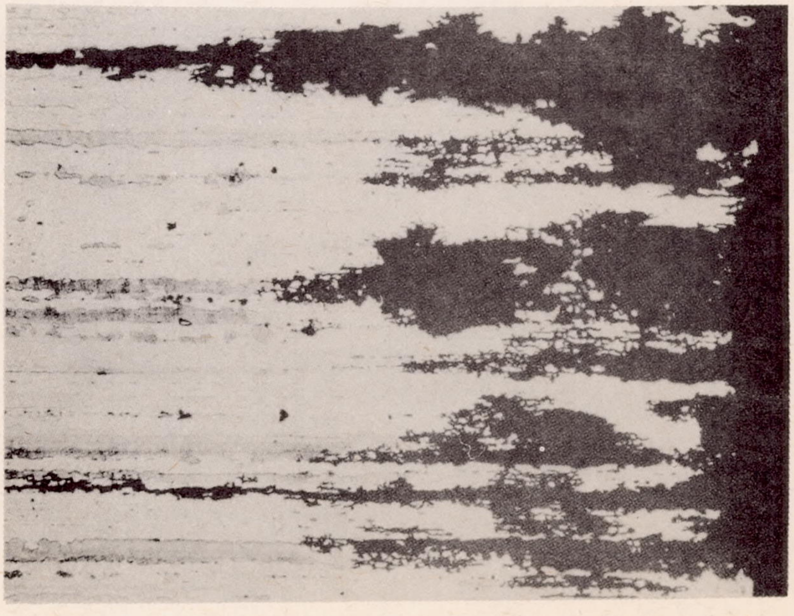


Figure 11.- Interfragmentary attack in extruded 24S-T aluminum alloy, specimen 26076. Magnification, 100 diameters.