ANL-6144
Metallurgy and Ceramics
(TID-4500, 15th Ed.)
AEC Research and
Development Report

ARGONNE NATIONAL LABORATORY
9700 S. Cass Avenue
Argonne, Illinois

METALLOGRAPHY OF ALUMINUM AND SOME
ALUMINUM-1 w/o NICKEL ALLOYS

by

Raymond K. Hart and M. J. Heyduk

Metallurgy Division

Program 10.2.2

June 1960

Operated by The University of Chicago
under
Contract W-31-109-eng-38

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	5
INTRODUCTION.	5
MATERIALS	6
EXPERIMENTAL METHODS	7
a. Surface Preparation	7
b. Cathodic Etching	8
c. Surface Replicas	9
EXAMINATION OF STRUCTURES.	10
a. High-purity Aluminum.	10
b. A300 Alloy	10
c. A255 Alloy	11
d. X8001 Alloy	12
HEAT TREATMENT OF ALUMINUM-NICKEL ALLOYS	12
CONCLUSIONS.	13
ACKNOWLEDGEMENTS	14
REFERENCES	14

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1	Diagram of etching chamber.	15
2	Optical micrograph of an electropolished high-purity aluminum surface.	16
3	Electron micrograph of an electropolished high-purity aluminum surface.	16
4	Electron micrograph of high-purity aluminum electropolished and etched in sodium hydroxide	16
5	Optical micrograph of A300 alloy electropolished and etched in hydrofluoric acid.	17
6	Optical micrograph of A300 alloy electropolished and etched in hydrofluoric acid.	17
7	Electron micrograph of A300 alloy electropolished and etched in hydrofluoric acid.	17
8	Electron micrograph of A300 alloy electropolished and etched in hydrofluoric acid.	17
9	Optical micrograph of A255 alloy mechanically polished and etched in hydrofluoric acid.	18
10	Electron micrograph of A255 alloy mechanically polished and etched in hydrofluoric acid.	18
11	Optical micrograph of A255 alloy electropolished and etched in hydrofluoric acid.	19
12	Optical micrograph of A255 alloy electropolished and etched in hydrofluoric acid.	19
13	Electron micrograph of A255 alloy electropolished and etched in hydrofluoric acid.	19
14	Optical micrograph of A255 alloy electropolished and etched in sodium hydroxide.	20
15	Electron micrograph of A255 alloy electropolished and etched in sodium hydroxide.	20
16	Optical micrograph of X8001 alloy mechanically polished and etched in hydrofluoric acid.	21
17	Electron micrograph of X8001 alloy mechanically polished and etched in hydrofluoric acid.	21

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
18	Optical micrograph of X8001 alloy electropolished and etched in hydrofluoric acid	22
19	Optical micrograph of X8001 alloy electropolished and etched in hydrofluoric acid	22
20	Electron micrograph of X8001 alloy electropolished and etched in hydrofluoric acid	22
21	Optical micrograph of X8001 alloy electropolished and etched in sodium hydroxide.	23
22	Electron micrograph of X8001 alloy electropolished and etched in sodium hydroxide.	23
23	Optical micrograph of A255 alloy electropolished and etched in sulphuric acid	24
24	Electron micrograph of X8001 alloy electropolished and etched in sulphuric acid	24
25	Electron micrograph of A255 alloy electropolished and etched in sulphuric acid	24
26	Optical micrograph of A300 alloy electropolished and cathodically etched	25
27	Electron micrograph of A300 alloy electropolished and cathodically etched	25
28	Optical micrograph of X8001 alloy electropolished and cathodically etched	25
29	Electron micrograph of X8001 alloy electropolished and cathodically etched	25
30	Electron micrograph of X8001 alloy heat-treated 20 hours at 500° C	26
31	Electron micrograph of A255 alloy heat-treated 20 hours at 500° C	26

METALLOGRAPHY OF ALUMINUM AND SOME ALUMINUM-1 w/o NICKEL ALLOYS

by

Raymond K. Hart and M. J. Heyduk

ABSTRACT

The microstructures of both high-purity aluminum and aluminum-1 w/o nickel alloy surfaces have been investigated by optical and electron optical techniques. Suitable surfaces were prepared by mechanical polishing and electropolishing. The prepared surfaces were etched by a variety of reagents so that their effects on the various surfaces could be observed. Several alloy surfaces were also etched by cathodic ion bombardment. The results of surface examinations are described in detail. Particular attention has been given to anomalous furrow structures which were observed in electron micrographs.

INTRODUCTION

Until five years ago, when Draley and Ruther⁽¹⁾ reported that aluminum-rich aluminum-nickel alloys had corrosion characteristics which made them suitable cladding material in high-temperature water-cooled nuclear reactors, they were of little importance and as a consequence much technical data on them are lacking.

The binary aluminum-nickel equilibrium diagram given by Mondolfo⁽²⁾ shows that in the 1 per cent nickel range only one compound is formed. This is NiAl_3 , orthorhombic with lattice parameters $a = 6.5982 \text{ \AA}$, $b = 7.3518 \text{ \AA}$ and $c = 4.8021 \text{ \AA}$. In the case of commercial alloys of the X8001 type, small amounts of iron, copper and silicon in addition to nickel are also present. These additional impurities usually come from the 1100 aluminum stock from which the alloys are made. In aluminum containing small amounts of iron and nickel as impurities there may be three different phases present. In addition to FeAl_3 and NiAl_3 , a ternary phase, FeNiAl_9 , may also be present. Ôsawa⁽³⁾ found FeAl_3 to be orthorhombic with $a = 11.870 \text{ \AA}$, $b = 8.088 \text{ \AA}$ and $c = 15.804 \text{ \AA}$. These results are different from those reported by Bachmetew,⁽⁴⁾ who found FeAl_3 to be orthorhombic with $a = 47.43 \text{ \AA}$, $b = 15.46 \text{ \AA}$ and $c = 8.08 \text{ \AA}$. These differing results for FeAl_3 may be due to complete or partial decomposition into Fe_2Al_5 or Fe_2Al_7 .⁽⁵⁾ The ternary

phase FeNiAl_9 is said to be monoclinic and isomorphous with Co_2Al_9 . However, there appears to be some evidence⁽⁵⁾ to support a formula $\text{FeNi}_2\text{Al}_{13}$ instead of FeNiAl_9 . No reference has been found as to whether or not a structural determination has been done to determine the more appropriate formula.

Since the alloys that are used for cladding are chill cast, it is assumed that both copper and silicon are present, mostly in solid solution.

Etching data for the above compounds are given in a table of etchants by Mondolfo,⁽²⁾ and it was from this table that the etchants used in the current experiments were selected. It has been pointed out by Raynor and Pfeil⁽⁶⁾ that caution should be exercised if the various phases are to be identified by their etching characteristics. In a number of cases these authors found that reproducible etching could be effected by first pretreating the surface for 2 seconds with a 5 per cent solution of potassium hydroxide at 70° C. This pretreatment led to shorter times of immersion in the acid reagent.

In order to obtain a clearer insight into the superior corrosion behavior in high-temperature water of aluminum-1 w/o nickel, as opposed to other aluminum alloys, the following investigation was carried out. It was designed chiefly to determine a suitable method - or methods - to prepare the metal surface for subsequent metallographic examination, the point of main interest being the distribution of second-phase precipitates. Super-purity aluminum was incorporated into these experiments for use as a control and also for completeness. Alloy A300, a description of which will be given later, was also used as a control.

It should be pointed out that from a corrosion standpoint, the second phase should be finely distributed throughout the aluminum matrix. In order to obtain this fine distribution, melts are chill cast and then heavily cold rolled. In this investigation all alloys except the A300 control were examined in the above condition and not in the as-cast condition, which is customary for phase identification.

Materials

The following materials were used in these experiments:

- a) pure aluminum
- b) X8001 alloy
- c) A255 alloy
- d) A300 alloy.

Pure aluminum specimens were obtained from "Swiss" high-purity aluminum stock. This material is nominally 99.999% pure. However, analysis

indicated it is approximately 99.997% pure, the main impurities being iron, 7 ppm; copper, 4 ppm; silicon, 12 ppm; and magnesium, > 2 ppm. Samples were cut from $\frac{1}{8}$ -in. thick cold-rolled sheets which were obtained by rolling down $\frac{3}{4}$ -in. square section castings. The X8001 alloy is a commercial alloy supplied by Alcoa in the form of $\frac{1}{8}$ -in. thick sheet. The following analysis is typical for this alloy:

Nickel	0.89	Silicon	0.11
Copper	0.13	Manganese	0.01
Iron	0.48	Titanium	0.02

Numbers given above are percentages.

The A255 series alloy was made from 99.99% purity aluminum stock to which was added 1.0% nickel and 0.1% titanium. This alloy was made in a graphite crucible and all melting and casting done under vacuum. The molten alloy was held at 900°C for 2 hours and then lowered to 720°C for 10 minutes before pouring into a water-cooled mold. This treatment was designed to remove the volatiles, zinc and magnesium. The 2-in. diameter billets were more than 50% cold worked and then hot rolled into $\frac{1}{8}$ -in. thick sheet.

The A300 control alloy was made from superpurity aluminum stock to which was added 1 w/o "spec pure" nickel. The heat was cast in a water-cooled mold and specimens were prepared directly from this casting.

Experimental Methods

a. Surface Preparation: Both mechanical polishing and electro-polishing techniques were used to prepare the surfaces for metallographic examination. Mechanically polished surfaces were prepared by the following method. Firstly, specimens were abraded with successively finer grades of emery paper to 000 emery, using kerosene as lubricant. Surface abrasion was then continued with diamond paste, starting with 6 grade and continuing through 3 grade to one grade. Hypres oil was used as lubricant. It was found beneficial to abrade the samples through lens tissue which was placed over the diamond paste after the latter had been spread out on "Vellux" tracing paper. Specimens were finally polished on microcloth with MgO as abrasive.

When the surface was to be electropolished, the initial preparation was as described above down to 6 grade diamond. Electropolishing was carried out at 50 to 60 volts in a 1:9 perchloric acid/acetic acid bath operating at 15°C. Polishing was continued for twice the period of time required to remove the scratches left by the 6 grade diamond abrasive.

The polished surfaces were firstly viewed in the as-polished condition and then etched. The following etchants were selected for their ability to show up the various second-phase precipitates:

1. HF solution: 0.5 ml conc HF in 100 ml solution - FeAl_3 , NiAl_3 , FeNiAl_9 .
2. 9% NaOH solution at 70°C for 5 sec - FeAl_3 , NiAl_3 , FeNiAl_9 .
3. H_2SO_4 solution: 20 ml conc. H_2SO_4 in 100 ml solution, at 70°C for 30 sec - FeAl_3 .

The etching characteristics of 0.5% HF are similar to the modified Keller's etch (0.5% HF, 1.5% HCl, 2.5% HNO_3) which is also commonly used for etching aluminum alloys but was not used in this investigation.

b. Cathodic Etching: An introductory investigation of the cathodic etching of some of these alloys has been successfully carried out. No reference can be found to this having been done in the past. A number of experimental rigs were investigated before the one described below was selected as being the most suitable. All the setups investigated contained two glass envelopes, the electrodes being confined to the inner envelope. The problem of design of a suitable cathodic etching apparatus resolved itself into one of finding a suitable geometrical arrangement for the components inside the large outer glass envelope so that the entire discharge was confined to the electrode section within the inner envelope. The design finally selected is shown schematically in Fig. 1, and basically is similar to the unit used by Padden and Cain.⁽⁷⁾ The aluminum cathode assembly was hollow so that specimens could be cooled during etching by circulating a coolant down this hollow stem from a reservoir attached to the top. In the case of aluminum-nickel alloys it was found unnecessary to cool the specimen; rather it was allowed to warm up during etching. The electrode assembly was designed so that the anode-to-cathode distance could be altered by screwing the anode up or down on its threaded shaft.

Prior to etching, the system was evacuated to 2×10^{-5} mm of Hg and then the specimen was "incubated" for one hour in an argon atmosphere at 10^{-2} mm of Hg. Sufficient current was passed to just maintain a discharge between the electrodes, i.e., 8 kv at approximately 3 ma. At the conclusion of an hour the argon pressure within the apparatus was increased to 3.5×10^{-2} mm of Hg and the current increased to approximately 15 ma at 5.6 kv. Almost immediately a bright silvery mirror appeared on the inside wall of the inner envelope. Etching was continued for one minute. At the conclusion of this time the argon supply was turned off and the specimen allowed to cool to room temperature under an argon pressure of 2×10^{-5} mm of Hg.

Welding-grade argon was supplied to the etching apparatus from a tank and this proved to be quite suitable, providing a dynamic system was maintained.

The sputtered film from an A300 alloy was dissolved off the glass envelope in 5 cc of aqua regia, made up from redistilled acids, and subjected to spectroscopic analysis. The analysis report was as follows:

Aluminum	70	Nickel	1.0
Calcium	< 1.5	Lead	< 1.0
Chromium	< 0.15	Tin	< 5.0
Iron	2.5	Titanium	< 0.2
Potassium	0.3	Zinc	< 5.0 - interference
Sodium	1.2	Zirconium	< 0.5

The results are reported in micrograms per ml and the estimated accuracy is a factor of two. This analysis shows that attack on the alloy surface is nonpreferential and that both constituents of the alloy are removed. The iron analysis is considerably higher than that which could be attributed to interference alone. Concurrent analyses showed the iron interference to be < 1. This iron then must have been sputtered from the mild steel components within the inner chamber of the apparatus, these components being the screw thread attachment holding the specimen and the anode support rod.

Since the analysis shows a very low chromium figure, it appears unlikely that any stainless steel was sputtered. Besides, all stainless steel components were far removed from the discharge region.

c. Surface Replicas: Three methods for replicating surfaces were investigated. These were as follows:

1. two-stage shadowed carbon replicas;
2. stripped anodically formed oxide films; and
3. direct-stripped, self-shadowed carbon replicas.

Results from replicas prepared by these three methods were compared, and as a result of this comparison all future replication was standardized on method 1. This method gave consistently good replicas of great clarity and had a resolution limit of less than 100 Å.

Preparation of replicas by this method is as follows. A plastic replicating material is made up of 10% ethyl methacrylate, 90% methyl methacrylate and hardener. The liquid is heated at 90°C for 1 to 3 minutes, the exact time depending on the viscosity required, then poured onto the prepared specimen surface and after smoothing out is allowed to harden. The first impression should always be discarded as it is used to clean the surface. Stripping of the replica from the surface is made easier by immersing the specimen plus replica in distilled water for five minutes at 50°C. No difference could be detected when replicas removed from the surface by this method were compared with other stripped replicas.

Next, the plastic replica is placed face upright on a clean microscope slide, using a drop of distilled water to hold it flat, and positioned in a vacuum evaporation unit. A few angstroms of 80% platinum-20% palladium alloy is then evaporated onto the plastic impression at $\tan^{-1/3}$. Immediately afterwards, and without breaking the vacuum, about 100 Å of carbon is evaporated normal to surface. After this, the composite film is removed from the evaporator and the plastic impression removed with chloroform, the shadowed carbon film being supported on a standard electron microscope grid.

Examination of Structures

High-purity Aluminum

When polycrystalline aluminum is electrolytically polished, it is found that individual grains become visible under the optical microscope (Fig. 2). This can be attributed to varying rates of metal dissolution on differently oriented grains, the rate being dependent on the surface potential of each grain. When examined at high magnification (Fig. 3), surfaces were found to exhibit a fine structure which is commonly called "orange peel."⁽⁸⁾ In addition to the orange-peel structure these surfaces exhibited "coarse" furrows, and these were in general unidirectional. This type of furrow structure has appeared on many of the electron micrographs taken during these experiments, and several additional experiments have been carried out with the aim of elucidating this structure. These experiments will be discussed at a later stage in this report.

Etching electropolished aluminum surfaces in either hydrofluoric acid or sulphuric acid does not alter the resolvable structure seen by electropolishing alone. In the case of surfaces etched in 10% sodium hydroxide, the occurrence of many small nodules was noted (Fig. 4). These nodules averaged some $0.25\ \mu$ in diameter and can possibly be attributed to entrapped hydrogen which is produced in appreciable quantity by the vigorous attack of sodium hydroxide.

A300 Alloy

The second phase of this alloy is etched up mainly in the form of "Chinese script" when viewed at low magnification (Fig. 5). However, the individual stringers are not continuous but appear at higher magnification (Figs. 6, 7, 26 and 27) to consist of lamellae and roughly spherical particles. Smaller second-phase particles ($\sim 0.25\ \mu$) are also visible in the matrix except in the region immediately adjacent the "Chinese script." Substructure in the form of unidirectional furrows also appears in the matrix (Figs. 7 and 8). Since this is a cast material, the direction of furrowing cannot be associated with any direction of work. Additionally in Fig. 8 there is evidence of a different type of furrowing at A in the top

right hand corner. This is immediately adjacent to and running parallel with the "Chinese script" direction. There are several explanations for this particular type of furrowing. One is that the area marked A has been denuded of nickel during the formation of second phase. Another and more likely explanation is that this region is highly strained due to the differing contractions of the matrix and second phase. Cathodically etched A300 alloy (Fig. 27) does not show either type of furrowing. This may be due to the annealing out of this structure by the heat generated by the discharge. The smoothness of the electropolished surface has also given way to a surface which is generally micropitted. Again this is probably due to condition of etch since other surfaces such as shown in Fig. 29 do not show this effect.

A255 Alloy:

A basic similarity exists between the microstructure of this alloy and the A300 alloy. The difference comes from rolling when the "Chinese script" structure of the second phase is broken down and the particulate matter distributed more uniformly throughout the matrix. This breakdown is, however, far from complete, as can be seen in Figs. 9 to 15. Figures 10 and 13 show that the particle size varies over an appreciable range from approximately 0.08 to 1 μ . Evidence of grain boundaries may be seen at A and B in Fig. 10. Fine parallel furrows indicated by C in this figure appear to be associated with subgraining. The direction of these various sets of furrows varies and is not associated with the rolling direction, which is from the bottom to the top of this figure. In addition, there are coarser furrows all of which are running roughly in the rolling direction and appear similar to the furrows observed in electron micrographs from aluminum and the other alloys investigated in this work.

The attack of sodium hydroxide etchant on the alloy surface was more severe than that of hydrofluoric acid, but the topographical detail observed on microscopic examination was the same (Figs. 14 and 15). The furrowing seen in Fig. 15 is mainly of the fine variety as indicated by C in Fig. 10. There is also evidence of coarser furrowing near the lower middle of this figure.

Sulphuric acid attack on A255 alloy is severe but less than that of sodium hydroxide. Both optical and electron micrographs, Figs. 23 and 25, show that etching is largely confined to the aluminum matrix, which becomes covered with triangular-shaped etch pits. Grain boundaries in the aluminum matrix are also etched up, as shown in Fig. 23. The second phase, although well outlined by this etchant, does not show the fine structural detail which is brought out by the other etchants.

X8001 Alloy

Microscopic examination of this alloy showed that the shape and distribution of the particulate material are both different to that of the A255 alloy. Surfaces which were polished and etched in 0.5% hydrofluoric acid are shown in Figs. 16 to 20. The particulate matter appeared in a random distribution throughout the matrix and varied in size from under 1 micron to over 10 microns. Many of the particles were longer than they were wide. Replicas taken from the etched surfaces showed the particles to have coarse "orange peel" type of structure. In Fig. 20, both grain boundaries and fine furrowing can be seen in the aluminum matrix. Small etch pits of both square and triangular shapes are also present. These etch figures appear to be concentrated above precipitates which are lying very close to the surface.

Surfaces of this alloy when etched in either sodium hydroxide or sulphuric acid were similar to that shown in Fig. 21. However, electron microscopic examination showed that the attack of these two etchants was entirely different. With sodium hydroxide the whole surface is roughened and the precipitates appear nodular as shown in Fig. 22. A few small nodules can also be seen in the matrix.

The particulate matter in X8001 appears to be attacked very little with sulphuric acid etchant. Electron optically (Fig. 24) the surface was found to contain a high concentration of square-shaped etch pits, many of which could not be directly associated with second-phase precipitates. This suggests that the main reaction occurs between sulphuric acid and the aluminum matrix.

Cathodically etched surfaces of this alloy were found to display considerable structural detail of the second-phase precipitates and also of the matrix. At high optical magnification (see Fig. 28) the shapes of individual particles were clearly outlined, as well as grain boundaries within the matrix. Figure 29 again shows the coarse "orange peel" structure of the second phase. The structure marked A in this figure is similar to that previously described as fine furrowing. The structure B seems to be related to underlying particles which have almost broken through the surface.

Heat Treatment of Aluminum-Nickel Alloys

Specimens of both X8001 and A255 alloy were stripped of their corrosion products after immersion in water at 250°C and 350°C for periods of time ranging from a few hours to a month. In the remaining metal no effects of this heat treatment were observed. This is in agreement with the findings of Coriou *et al.*⁽⁹⁾ Other specimens of these two alloys were heated in air at 500°C for 20 hours and then air cooled to room temperature. The surfaces were then prepared and viewed electron optically (see Figs. 30 and 31).

For interpretation of these two figures it should be noted that the carbon films were shadowed and not the plastic impressions. These micrographs show that coalescence had taken place in both alloys. The randomly distributed spherical particles varied in size from approximately 0.1 micron to 2.5 microns.

CONCLUSIONS

The surface structure of the investigated alloys was equally well portrayed on both mechanically and electropolished specimens. However, because of the difficulty attached to the mechanical polishing of such soft materials and consequently because of the time involved, the electropolishing technique is to be preferred. To obtain the best results with electropolishing, surfaces should be firstly abraded to at least 6 grade diamond.

The particulate material in these alloys is etched up with a high degree of fidelity by the use of either hydrofluoric acid or sodium hydroxide etchants. It has also been shown that cathodic etching is suitable for aluminum-nickel alloys providing certain precautions are taken. Sulphuric acid was found unsuitable as an etchant for these alloys due to the formation of etch pits.

In A300 alloy the second phase forms a "Chinese script" pattern which consists of lamellae and spherical particles. Additionally, there are small, approximately 0.25-micron, particles distributed throughout the matrix. However, the area adjacent the "Chinese script" is denuded of this fine material.

The A255 alloy is basically similar to the A300 alloy. The second-phase "Chinese script" is elongated in the rolling direction and somewhat broken up. Particles varied from 0.08 micron to over 1 micron. The matrix was shown to be polycrystalline and there is evidence of subgraining.

Within the X8001 alloy there is a more random distribution of second phase, the size of which varied from 1 to 10 microns. The difference in the distribution of second phase in A255 and X8001-type alloys is probably due to casting and fabrication differences. The commercial X8001 alloy is continuously cast and then worked to a much greater degree than the laboratory-prepared A255 alloy.

The fine furrow structure seen on a number of electron micrographs can be attributed to two classes of substructure. The first class, which is indicated, for instance, by C in Fig. 10, is associated with the granular structure and orientation of the matrix. The second class is shown in Fig. 29 at B. This structure appears to be associated with nearly exposed second-phase particles and is usually outlined by the shape of the submerged particles. Also, a marked similarity is immediately evident when this structure is compared with the topographical structure of exposed second phase.

The cause of the coarse furrows is not so easy to discern. It has been suggested that these furrows are in fact microcracks in the surface. In order to investigate this assumption, the following experiment was carried out. Four test specimens were cut from A255 alloy sheet, two with their long axis parallel to the rolling direction, and the other two at right angles to the rolling direction. Two rectangular-shaped block specimens were also cut from this stock. The test pieces were stressed at 2.8 kg/mm^2 for 24 hours while the temperature was maintained at 150°C . Block specimens were kept at 150°C for 24 hours, but not stressed.

From an examination of electron micrographs taken before and after these experiments no obvious topographical changes to the surface could be detected. If these furrows are, in fact, cracks, then they remain immobile under the conditions of these experiments and consequently their exact nature is not immediately obvious.

Heat treatment at temperatures up to 350°C appears to have very little effect on these alloys. However, at 500°C there is a coalescence of the second phase, with the formation of randomly distributed spherical particles of 0.1 to 2.5-micron diameter.

ACKNOWLEDGEMENTS

The authors wish to thank J. E. Draley and W. E. Ruther for supplying the metals used in these experiments and for the helpful discussions while this work was in progress. Thanks are also due to S. Matras for detailing the mechanical polishing technique for these aluminum alloys as well as for supplying the mechanically polished A255 alloy.

REFERENCES

1. Draley, J. E. and Ruther, W. E., ANL-5430 (1955).
2. Mondolfo, L. F., Metallography of Aluminum Alloys, John Wiley and Sons, New York (1943).
3. Osawa, A., Sci. Rep. Tohoku. Imp. Univ., 22, 803 (1933).
4. Bachmetew, R., Z. Krist., 88, 575 (1934).
5. Bradley, A. J. and Taylor, A., J. Inst. Metals 66, 53 (1940).
6. Raynor, G. V. and Pfeil, P. C. L., J. Inst. Metals, 73, 397 (1947).
7. Padden, T. R. and Cain, F. M., Metal Progr. 108, 66 (1954).
8. Brown, A. F., Nature 163, 961 (1949).
9. Coriou, H., Fournier, R., Grall, L., Herenguel, J., Hure, J. and Lelong, P. Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva (1958), Vol. 5, p. 128.

FIG. 1 DIAGRAM OF ETCHING CHAMBER

110205

X150

Fig. 2. Optical micrograph of an electro-polished high-purity aluminum surface.

100487

X16000

Fig. 3. Electron micrograph of an electro-polished high-purity aluminum surface.

100271

X6000

Fig. 4. Electron micrograph of high-purity aluminum electropolished and etched in sodium hydroxide.

110098

X150

Fig. 5. Optical micrograph of A300 alloy electro-polished and etched in hydrofluoric acid.

110251

X1000

Fig. 6. Optical micrograph of A300 alloy electro-polished and etched in hydrofluoric acid.

100479

X6000

Fig. 7. Electron micrograph of A300 alloy electro-polished and etched in hydrofluoric acid.

100480

X16,000

Fig. 8. Electron micrograph of A300 alloy electro-polished and etched in hydrofluoric acid.

110248

X1000

Fig. 9. Optical micrograph of A255 alloy mechanically polished and etched in hydrofluoric acid.

100358

X6000

Fig. 10. Electron micrograph of A255 alloy mechanically polished and etched in hydrofluoric acid.

110149

X150

Fig. 11. Optical micrograph of A255 alloy electro-polished and etched in hydrofluoric acid

110252

X1000

Fig. 12. Optical micrograph of A255 alloy electro-polished and etched in hydrofluoric acid

100453

X6000

Fig. 13. Electron micrograph of A255 alloy electropolished and etched in hydrofluoric acid

110211

X150

Fig. 14. Optical micrograph of A255 alloy electro-polished and etched in sodium hydroxide

100461

X6000

Fig. 15. Electron micrograph of A255 alloy electro-polished and etched in sodium hydroxide

110255

X1000

Fig. 16. Optical micrograph of X8001 alloy mechanically polished and etched in hydrofluoric acid

100467

X6000

Fig. 17. Electron micrograph of X8001 alloy mechanically polished and etched in hydrofluoric acid

110148

X150

Fig. 18. Optical micrograph of X8001 alloy electro-polished and etched in hydrofluoric acid

110067

X1000

Fig. 19. Optical micrograph of X8001 alloy electro-polished and etched in hydrofluoric acid

100276

X6000

Fig. 20. Electron micrograph of X8001 alloy electro-polished and etched in hydrofluoric acid

110121

X150

Fig. 21. Optical micrograph of X8001 alloy electropolished and etched in sodium hydroxide

100470

X6000

Fig. 22. Electron micrograph of X8001 alloy electropolished and etched in sodium hydroxide

110253

X150

Fig. 23. Optical micrograph of A255 alloy electropolished and etched in sulphuric acid

100267

X6000

Fig. 24 Electron micrograph of X8001 alloy electropolished and etched in sulphuric acid

100265

X6000

Fig. 25. Electron micrograph of A255 alloy electropolished and etched in sulphuric acid

110246 X1000
Fig. 26. Optical micrograph of A300 alloy electro-polished and cathodically etched.

100452 X6000
Fig. 27. Electron micrograph of A300 alloy electro-polished and cathodically etched.

110256 X1000
Fig. 28. Optical micrograph of X8001 alloy electro-polished and cathodically etched.

100483 X6000
Fig. 29. Electron micrograph of X8001 alloy electro-polished and cathodically etched.

D1153

X16,000

Fig. 30. Electron micrograph of X8001 alloy
heat-treated 20 hours at 500°C.

D1147

X16,000

Fig. 31. Electron micrograph of A255 alloy
heat-treated 20 hours at 500°C.